Indian Standard

SPECIFICATION FOR COLD POLYMERIZED RAW STYRENE-BUTADIENE RUBBER

(First Revision)

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Indian Standard

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(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 13 August 1985, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1969. In this revision additional requirement for relative density has been included. In the absence of an acceptable test method for the estimation of gel content, it is envisaged to include the same in due course of time when an accurate method is made available. For compounding test recipe based on high abrasion furnace (HAF) carbon black has been prescribed with consequential changes in the requirements of compounded rubber.

0.3 This standard prescribes two types of non-oil-extended raw styrene-butadiene rubbers (SBR) manufactured by cold polymerization using emulsion technique normally below 10°C. Type 1 prescribed in this standard corresponds to the commercially known SBR 1500; this number is allotted by the International Institute of Synthetic Rubber Producers' Inc, USA. Type 2 prescribed in this standard corresponds to non-oil-extended SBR manufactured by cold polymerization, commercially known as SBR 1502 which has also been allotted by the same Institute.

0.3.1 For type 1, the following staining type stabilizers are generally used:

a) An acetone-diphenylamine reaction product; and

b) A mixture of alkylated diphenylamines.

NOTE — Styrenated phenol at 1·0 to 1·8 percent by mass may also be used.

0.3.2 For Type 2, the following non-staining type stabilizers are generally used:

a) Styrenated phenols; and
b) An alkylated aryl phosphite.

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 prescribes the requirements and the methods of sampling and test for non-oil-extended, non-pigmented, raw styrene-butadiene rubbers (SBR), obtained by copolymerization of styrene and butadiene at low temperatures.

2. TYPES

2.1 This standard covers the following two types of copolymers of styrene and butadiene.

Type 1 — Non-oil-extended, non-pigmented, staining, emulsified with rosin acid soap, coagulated with sodium chloride and sulphuric acid.

Type 2 — Non-oil-extended, non-pigmented, non-staining emulsified with fatty acid rosin soap, coagulated with sodium chloride and sulphuric acid.

3. REQUIREMENTS

3.1 Description — The material shall be free from any foreign matter and comply with the requirements given in Table 1.

3.2 The physical requirements of the material, when compounded, according to the method prescribed in Appendix B, cured for the time specified in col 3 of Table 2 and tested according to col 8 of Table 2 shall be as given in col 4 to 7 of Table 2.

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

TABLE 1 REQUIREMENTS FOR COLD POLYMERIZED RAW STYRENE-BUTADIENE RUBBER

(*Clause* 3.1)

Sl No.	Characteristic	Requirements For				Method of Test	
		Type 1		Type 2			
		Min	*Max*	*Min*	*Max*		
(1)	(2)	(3)	(4)	(5)	(6)		(7)
i)	Volatile matter, percent by mass	—	0·75	—	0·75	SBR : 1 or SBR : 2	of IS : 4518 (Part 1)-1967*
ii)	Total ash, percent by mass	—	1·5	—	1·5	SBR : 3	
iii)	Organic acid, percent by mass	5·0	7·2	4·8	7·0	SBR : 4	
iv)	Soap, percent by mass	—	0·5	—	0·50	SBR : 5	
v)	Staining antioxidant, percent by mass	1·0	1·8	—	—	SBR : 6	
vi)	Non-staining antioxidant, percent by mass	—	—	1·0	1·8	SBR : 6	
vii)	Bound styrene, percent by mass	21·5	25·5	21·5	25·5	SBR : 7	
viii)	Mooney viscosity ML_{1+4} at 100°C	46	58	46	58	SBR : 8	
ix)	Solvent extract, percent by mass	6	10	6	10	SBR : 9	of IS : 4518 (Part 2) - 1971†
x)	*Relative density*	0·93	0·95	0·93	0·95	Appendix A	

*Methods of test for styrene-butadiene rubbers (SBR) : Part 1 Determination of volatile matter, total ash, organic acid, soap, antioxidants bound styrene and mooney viscosity.

†Methods of test for styrene-butadiene rubbers (SBR): Part 2 Determination of solvent extract and oil content.

TABLE 2 PHYSICAL REQUIREMENTS FOR COMPOUNDED RUBBER

(*Clause* 3.2)

Sl No.	Characteristic	Cure Time (Minutes)	Requirements For				Method of Test
			Type 1		Type 2		
			Min	*Max*	*Min*	*Max*	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Compounded viscosity ML_{1+4} at 100°C	—	—	80	—	80	SBR : 8 of IS : 4518 (Part 1)-1967*
ii)	Tensile strength MPa (approx kgf/cm²)	35	20·0 (200)	—	21·0 (210)	—	IS : 3400 (Part 1)-1977†
iii)	Percent elongation at break	35	350	—	320	—	
iv)	Modulus at 300 percent	25	5·0 (50)	11·0 (110)	7·0 (70)	17·0 (170)	
	Elongation, cure temperature 145°C,	35	10·0 (100)	15·0 (150)	13·0 (130)	20·0 (200)	
	MPa (approx kgf/cm²)	50	13·0 (130)	18·0 (180)	15·0 (150)	22·0 (220)	

*Methods of test for styrene-butadiene rubbers (SBR): Part 1 Determination of volatile matter, total ash, organic acid, soap, antioxidants, bound styrene and mooney viscocity.

†Methods of test for vulcanized rubbers: Part 1 Tensile stress-strain properties (*first revision*).

4. PACKING AND MARKING

4.1 Packing — The material shall be wrapped in polyethylene sheets and then in paper bags.

NOTE — Low density polyethylene sheets which are easily dispersible in rubber and of the following description are generally found suitable:

Thickness, mm	0·030 to 0·040
Relative density	0·92
Melting point	109°C

4.1.1 The material may also be dusted with talc and packed in paper bags so as to have 33 bales per tonne weighing approximately 30 kg per bag.

4.2 Marking—The material shall be marked with the type of rubber, preceded by the letters low temperature polymerized, net mass, batch number, year of manufacture and trade-mark, if any.

4.2.1 The material may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING AND CRITERIA FOR CONFORMITY

5.1 The scale of sampling and criteria for conformity of a consignment of the rubber to this standard shall be as prescribed in IS : 5599-1970*.

*Methods for sampling of raw rubber.

APPENDIX A
(*Clause* 3.1)

DETERMINATION OF RELATIVE DENSITY IN RUBBER

A-1. RELATIVE DENSITY

A-1.1 Determine the relative density using the pycnometer with alcohol in place of water to eliminate the errors due to air bubbles. Take measurements at a temperature between 24·5 and 25·5°C, unless the coefficient of expansion of the rubber product is known, in that case make the determination at any convenient temperature and correct to 25°C.

A-1.2 Calculation

$$\text{Relative density at } 25°C = 0{\cdot}997\ 1 \times \frac{M_1}{M_1 - (M_2 - M_3)} \times D$$

where

M_1 = mass of specimen;

M_2 = mass of pycnometer filled with specimen and alcohol;

M_3 = mass of pycnometer filled with alcohol; and

D = density of alcohol at 25°C.

APPENDIX B
(*Clause* 3.2)

COMPOUNDING OF RUBBER FOR PHYSICAL TESTING

B-1. MIXING MILL

B-1.1 The laboratory mixing mill has two parallel and cylindrical hardened steel rolls 152·5 ± 2·5 mm in out side diameter. The rolls are fitted with adjustable guides to allow a maximum working width of 265 ± 15 mm. The mill has provisions for maintaining the temperatures of the roll surfaces at 50 ± 5°C during the mixing of the rubber. The two rolls rotate at different speeds. The speed of the slow roll is 24 ± 2 rev/min and the friction ratio is 1 : 1 : 4.

B-1.1.1 If mills having ratios of fast to slow roll speeds lower than 1·4 are used, modifications in the mixing conditions given under procedure may be required to obtain results comparable to those obtained with the standard mill.

B-1.1.2 The mill is designed to permit adjustment of the distance between the rolls from 0·2 mm or less, to 3·0 mm or more.

B-2. PREPARATION OF MIX FOR VULCANIZATION

B-2.1 Using a sample of styrene-butadiene copolymer selected in accordance with IS : 5599-1970* make a mix of the following composition, the batch mass being four times the formula mass expressed in grams.

Material	*Parts by Mass* (g)
Raw styrene-butadiene rubber	100·00
Sulphur (conforming to IS : 8851-1978†)	1·75
Stearic acid (conforming to type 4 or type 5 of IS : 1675-1971‡)	1·00
High abrasion furnace (HAF) carbon black (conforming to IS : 7497-1974 §)	50·00
Zinc oxide (conforming to IS : 3399-1973 ‖)	3·00
N-tertiary butyl-2-benzothiazole sulphenamide (TBBS)	1·00

B-3. METHOD OF MIXING

B-3.1 The duration in minutes for mixing is given against each of the following stages :

	Duration (min)
a) Band the rubber with the mill opening set at 1·1 mm and make 3/4 cuts every 30 seconds from alternate sides, roller temperature being 50 ± 5°C.	7
b) Add the sulphur slowly and evenly across the rubber.	2
c) Add the stearic acid. Make one 3/4 cut from each side.	2

*Methods for sampling of raw rubber.

†Specification for sulphur for rubber industry.

‡Specification for stearic acid, technical (*first revision*).

§Specification for high abrasion furnace (HAF) carbon black.

‖Specification for zinc oxide for rubber industry (*first revision*).

	Duration (min)
d) Add the carbon black evenly across the mill at a uniform rate. When about half the black has been incorporated, open the mill to 1·4 mm and make one 3/4 cut from each side. Then add the remainder of the carbon black, including the black that has dropped into the mill pan. When all the black has been incorporated, open the mill to 1·8 mm and make one 3/4 cut from each side.	12
e) Add the zinc oxide and TBBS with the mill opening at 1·8 mm.	3
f) Make three 3/4 cuts from each side.	3
g) Cut the batch from the mill. Set the mill opening to 0·8 mm and pass the rolled batch end-wise through the rolls six times.	2
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B-3.2 Sheet the batch to an approximate thickness of 6 mm and check the mass. Remove sufficient sample for mooney viscosity testing.

B-3.3 Sheet the batch to approximately 2·2 mm for preparing test slabs or to the appropriate thickness for preparing ring specimens.

B-3.4 Condition the batch for 2 to 24 hours after mixing and prior to vulcanizing.

B-4. TEST SLAB MOULD

B-4.1 The mould used shall have a depth between 1·90 and 2·00 mm for dumb-bell shaped test pieces and shall be capable of moulding square test slabs of side length 150 mm from which specimens may be cut with a die as given in Fig. 1 of IS : 3400 (Part 1) - 1977*.

*Methods of test for vulcanized rubbers: Part 1 Tensile stress-strain properties (*first revision*).

B-5. PLATEN PRESS VULCANIZATION

B-5.1 Bring the mould to vulcanization temperature of 145°C within ±0·5°C in the closed press, and hold at this temperature for at least 20 min before the unvulcanized pieces are inserted. Verify the temperature of the mould by means of a thermocouple or other suitable temperature measuring device inserted in one of the overflow grooves and in intimate contact with the mould.

B-5.2 Open the press, insert the unvulcanized pieces in the mould and close the press in the minimum time possible. When the mould is removed from the press to insert the pieces, precautions should be taken to prevent excessive cooling of the mould by contact with cool metal surfaces or by exposure to air draughts.

B-5.3 The time of vulcanization shall be considered to be the period between the instant the pressure is applied fully and the instant the pressure is released. Hold the mould under a mininum pressure of 3·5 MPa on the cavity areas during vulcanization.

As soon as the press is opened, remove the vulcanized sheet from the mould and cool in water (room temperature or lower) for 10 to 15 min. Then wipe dry the sheets cooled in water and reserve for test. In both of the preceding operations, take care to prevent undue stretching or deformation.

B-5.4 Store at the standard temperature of 27 ± 2°C and relative humidity 65 ± 5.

B-5.5 For all test purposes the minimum time between vulcanization and testing shall be 16 hours.

B-5.6 Maximum time between vulcanization and testing shall be 4 weeks and for evaluation intended to be comparable, the tests, as far as possible, shall be carried out after the same time interval.

(*Continued from page* 2)

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