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International Standard ISO 2322

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber, styrene-butadiene (SBR, YSBR) — Emulsion and solution-polymerized types — Test recipes and evaluation of vulcanization characteristics

Caoutchoucs butadiène-styrène (SBR, YSBR) — Types polymérisés en émulsion et en solution — Formules d'essai et évaluation des caractéristiques de vulcanisation

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2322 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 2322-1981), of which it constitutes a technical revision (see the Introduction).

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Rubber, styrene-butadiene (SBR, YSBR) — Emulsion and solution-polymerized types — Test recipes and evaluation of vulcanization characteristics

0 Introduction

In this third edition of ISO 2322 the scope has been widened to include solution polymerized rubbers and rubbers with high styrene content. A second test formula has been introduced for evaluation of the high styrene rubbers.

1 Scope and field of application

This International Standard specifies standard materials, equipment and processing methods for evaluating vulcanization characteristics of emulsion and solution-polymerized styrene-butadiene rubbers (SBR, YSBR), including oil-extended rubbers.

It applies to the rubbers listed in table 1.

Table 1 — Types of raw styrene-butadiene rubbers

Rubber (oil-extended or non oil-extended)	Styrene	
	Form	Content % (*m*/*m*)
Series A		
1) Emulsion SBR	random	< 50
2) Solution SBR	random	< 50
3) Solution SBR		total < 50
	of which in block	< 30
Series B		
1) Emulsion SBR	random	> 50
2) Solution SBR	random	> 50
3) Solution SBR		total < 50
	of which in block	> 30
4) Solution YSBR	total block	—

2 References

ISO 37, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 1796, *Rubber, raw — Sample preparation.*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

3 Test recipes

3.1 Standard test formulae

The standard test formulae are given in table 2.

The materials shall be NBS[1] standard reference materials as indicated in table 2, or shall be in accordance with equivalent national standards.

Table 2 — Test formulae

Material	NBS standard reference material number	Parts by mass	
		Series A	Series B
Non-pigmented styrene-butadiene rubber (SBR) (including oil in oil-extended SBR)	—	100,00	—
Type 1500 SBR*	386	—	65,00
Series B SBR	—	—	35,00
Sulfur	371	1,75	1,75
Stearic acid	372	1,00	1,00
Oil furnace black (HAF)**	378	50,00	35,00
Zinc oxide	370	3,00	3,00
TBBS***	384	1,00	1,00
		156,75	141,75

* Europrene 1500 EST developed by ENICHEM may be used in place of NBS 386 but this may give slightly different results.

** The current Industry Reference Black may be used in place of NBS 378, but this may give slightly different results.

*** *N-tert*-butyl-2-benzothiazole-sulfenamide. This shall be supplied in powder form having an initial ether- or ethanol-insoluble matter content of less than 0,3 %. The material shall be stored at room temperature in a closed container and the ether- or ethanol-insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

1) National Bureau of Standards of the USA.

3.2 Procedure

3.2.1 Equipment and procedure

Equipment and procedure for the preparation, mixing and vulcanization shall be in accordance with ISO 2393.

3.2.2 Mill mixing procedure

The standard laboratory mill batch mass, in grams, shall be based on four times the formulae mass. The surface temperature of the rolls shall be maintained at 50 ± 5 °C.

NOTE — All mill openings should be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

	Series A		Series B	
	Duration (min)	Cumulative time (min)	Duration (min)	Cumulative time (min)
3.2.2.1 Homogenize series B rubbers with the mill opening set at 1,1 mm at a temperature of 100 °C	—	—	1	—
3.2.2.2 Band the rubber with the mill opening set at 1,1 mm and make 3/4 cuts every 30 s from alternate sides	7	7	—	—
After banding SBR 1500, add the homogenized rubber (as in 3.2.2.1) and make 3/4 cuts from both sides every 30 s	—	—	8	9
3.2.2.3 Add the sulfur slowly and evenly across the rubber	2	9	2	11
3.2.2.4 Add the stearic acid. Make one 3/4 cut from each side	2	11	2	13
3.2.2.5 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	12	23	12	25
3.2.2.6 Add the zinc oxide and the TBBS with the mill opening still at 1,8 mm	3	26	3	28
3.2.2.7 Make three 3/4 cuts from each side	3	29	3	31
3.2.2.8 Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise through the rolls six times	2	31	2	33

3.2.2.9 Sheet the batch to an approximate thickness of 6 mm and check weigh the batch. The mass of the mixed batch shall not differ from the total mass of all the materials by more than ± 1,0 %. Remove sufficient sample for oscillating disc curemeter testing.

3.2.2.10 Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

3.2.2.11 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing at a standard laboratory temperature as defined in ISO 471.

4 Evaluation of vulcanization characteristics

4.1 Evaluation according to stress-strain properties

Vulcanize sheets at 145 °C for three periods selected from a cure series of 15, 25, 35, 50 and 75 min.

Alternatively vulcanize sheets at 150 °C for three periods selected from a cure series of 10, 15, 20, 25, 30, 35 and 50 min. These conditions will give results different from the ones obtained by the recommended standard vulcanization conditions.

NOTE — *The three periods of cure selected should cover the undercure, optimum cure and overcure of the rubber under test.*

Condition the vulcanized test slabs for 16 to 72 h at a standard laboratory temperature as defined in ISO 471.

Measure the stress-strain properties in accordance with ISO 37.

4.2 Evaluation according to oscillating disc curemeter test

Measure the following standard test parameters :

M_L, M_H, $t_{s\,1}$, t'_c (50) and t'_c (90)

in accordance with ISO 3417, using the following test conditions :

oscillation frequency :	1,7 Hz (100 cycles per minute)
amplitude of oscillation :	1° arc
selectivity :	to be chosen to give at least 75 % full scale deflection at M_H NOTE — With some polymers, 75 % may not be attainable.
die temperature :	160 °C
pre-heat time :	none

5 Precision

To be added later.