INTERNATIONAL STANDARD

ISO 2322

Fourth edition
1996-11-01

Styrene-butadiene rubber (SBR) — Emulsion- and solution-polymerized types — Evaluation procedures

Caoutchouc butadiène-styrène (SBR) — Types polymérisés en émulsion et en solution — Méthode d'évaluation

Reference number
ISO 2322:1996(E)

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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2322 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry.*

This fourth edition cancels and replaces the third edition (ISO 2322:1985) which has been technically revised.

Annexes A, B and C of this International Standard are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

Styrene-butadiene rubber (SBR) — Emulsion- and solution-polymerized types — Evaluation procedures

1 Scope

This International Standard specifies

— physical and chemical tests on raw rubbers;

— standard materials, standard test formulations, equipment and processing methods for evaluating the vulcanization characteristics of emulsion- and solution-polymerized styrene-butadiene rubbers (SBR), including oil-extended rubbers.

It applies to those rubbers listed in table 1 which are normally used in vulcanized form.

Table 1 — Types of raw styrene-butadiene rubber

Rubber (oil-extended or non-oil-extended)	Styrene		
	Type of copolymer	Total content % (*m/m*)	Block content % (*m/m*)
Series A			
1) Emulsion SBR	random	< 50	0
2) Solution SBR	random	< 50	0
3) Solution SBR	partial block	< 50	< 30
Series B			
1) Emulsion SBR	random	> 50	0
2) Solution SBR	random	> 50	0
3) Solution SBR	partial block	< 50	> 30

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties.*

ISO 247:1990, *Rubber — Determination of ash.*

ISO 248:1991, *Rubbers, raw — Determination of volatile-matter content.*

ISO 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity.*

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing.*

ISO 1795:1992, *Rubber, raw, natural and synthetic — Sampling and further preparative procedures.*

ISO 2393:1994, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 6502:—[1)], *Rubber — Introduction and guide to the use of curemeters.*

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards.*

ISO 11235:—[2)], *Rubber compounding ingredients — Sulfenamide-type accelerators — Methods of test.*

ASTM D 412-92, *Test methods for vulcanized rubber and thermoplastic rubbers and thermoplastic elastomers — Tension.*

ASTM D 1646-95a, *Test methods for rubber — Viscosity, stress relaxation, and pre-vulcanization characteristics (Mooney viscometer).*

ASTM D 2084-93, *Test method for rubber property — Vulcanization characteristics using oscillating disk cure meter.*

ASTM D 3185-88(1994), *Test methods for rubber — Evaluation of SBR (styrene-butadiene rubber) including mixtures with oil.*

3 Sampling and further preparative procedures

3.1 Take a laboratory sample of approximately 1,5 kg by the method described in ISO 1795.

3.2 Prepare test portions in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1 on a test portion prepared in accordance with the preferred method of ISO 1795 (unmilled test portion).

Record the result as ML(1 + 4) at 100 °C.

NOTES

1 If ML(1 + 4) at 100 °C exceeds 100 MU, the small rotor may be used and the result reported as MS(1 + 4) at 100 °C.

2 Alternatively, the Mooney viscosity may be determined on a test portion prepared by the mill massing procedure of ISO 1795. This method will give poorer reproducibility, however, and the results may be different.

4.2 Volatile matter

Determine the volatile-matter content by the hot-mill method or by the oven method as specified in ISO 248.

---

1) To be published. (Revision of ISO 6502:1991)

2) To be published.

4.3 Ash

Determine the ash in accordance with method A or method B of ISO 247:1990.

5 Preparation of the test mixes

5.1 Standard test formulations

The standard test formulations are given in table 2.

The materials shall be national or international standard reference materials.

If no standard reference material is available, the materials to be used shall be agreed by the parties concerned.

5.2 Alternative formulations for oil-extended types

ASTM D 3185 specifies the test formulations given in table 3 for evaluation of general-purpose, oil-extended SBR, according to the oil content of the rubber. These test formulations may be used as alternatives to the test formulations given in table 2.

5.3 Procedure

5.3.1 Equipment and procedure

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

Two alternative mixing procedures are specified:

Method A — Mill mixing

Method C — Miniature internal mixer mixing

NOTE — A method B using an internal mixer for initial mixing and a mill for final mixing is presented in annex A for information only since insufficient experience has been gained with this method and the absence of information on precision does not allow it to be included as an integral part of the standard.

Table 2 — Test formulations

Material	Parts by mass	
	Series A	Series B
Styrene-butadiene rubber (SBR) (including oil in oil-extended SBR)	100,00	—
Type 1500 SBR[1)]	—	65,00
Series B SBR	—	35,00
Sulfur	1,75	1,75
Stearic acid	1,00	1,00
Current industry reference black[2)]	50,00	35,00
Zinc oxide	3,00	3,00
TBBS[3)]	1,00	1,00
	156,75	141,75

1) SBR 1500 EST, supplied by Enichem Elastomeri, Strada 3, Palazzo B1, 20090 Assago, Milan, Italy, is an example of a suitable product available commercially. This information is given for convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

2) Dried for 1 h at 125 °C ± 3 °C and stored in a tightly closed container.

3) *N-tert*-butylbenzothiazole-2-sulfenamide. This shall be supplied in powder form having an initial insoluble-matter content, determined in accordance with ISO 11235, of less than 0,3 %. The material shall be stored at room temperature in a closed container and the insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

Table 3 — Alternative test formulations for oil-extended types

Formulation No.	Quantity (parts by mass)					
	1B	2B	3B	4B	5B	6B
Parts oil	25	37,5	50	62,5	75	Y[1]
Oil-extended rubber	125,00	137,50	150,00	162,50	175,00	100 + Y
Zinc oxide	3,00	3,00	3,00	3,00	3,00	3,00
Sulfur	1,75	1,75	1,75	1,75	1,75	1,75
Stearic acid	1,00	1,00	1,00	1,00	1,00	1,00
Current industry reference black[2]	62,50	68,75	75,00	81,25	87,50	(100 + Y)/2
TBBS[3]	1,25	1,38	1,50	1,63	1,75	(100 + Y)/100
	194,50	213,38	232,25	251,13	270,00	
Batch factor for mill mix	2,4	2,2	2,0	1,9	1,7	
Batch factor for miniature internal mixer mix						
Cam head	0,37	0,34	0,31	0,29	0,27	
Banbury head	0,328	0,298	0,273	0,252	0,234	

1) Y = parts oil by mass per 100 parts base polymer in oil-extended rubber.

2) Dried for 1 h at 125 °C ± 3 °C and stored in a tightly closed container.

3) *N-tert*-butylbenzothiazole-2-sulfenamide. This shall be supplied in powder form having an initial insoluble-matter content, determined in accordance with ISO 11235, of less than 0,3 %. The material shall be stored at room temperature in a closed container and the insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

5.3.2 Method A — Mill mixing procedure

The standard laboratory mill batch mass, in grams, shall be based on four times the formulation mass (i.e. 4 × 156,75 g = 627 g or 4 × 141,75 g = 567 g). Maintain the surface temperature of the rolls at 50 °C ± 5 °C. Maintain a good rolling bank at the nip of the rolls during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings may be necessary.

	Series A		Series B	
	Duration	Cumulative time	Duration	Cumulative time
	(min)	(min)	(min)	(min)
a) Homogenize series B rubbers with the mill opening set at 1,1 mm at a temperature of 100 °C ± 5 °C.	—	—	1,0	—
b) Band the rubber with the mill opening set at 1,1 mm and make 3/4 cuts every 30 s from alternate sides.	7,0	7,0	—	—
After banding SBR 1500, add the rubber [homogenized as in 5.3.2 a)] and make 3/4 cuts from both sides every 30 s.	—	—	8,0	9,0
c) Add the sulfur slowly and evenly across the rubber.	2,0	9,0	2,0	11,0
d) Add the stearic acid. Make one 3/4 cut from each side.	2,0	11,0	2,0	13,0
e) 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. Be certain to add 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.	12,0	23,0	12,0	25,0