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

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

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document 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 seventh edition cancels and replaces the sixth edition (ISO 2322:2014), which has been technically revised.

The main changes are as follows:

— update of the normative references in Clause 2 and addition of a Clause 3 for terms and definitions;

— addition of batch factor of alternative test formulations for oil-extended types in Table 3 (6.2);

— addition of mill-mixing procedure of the test formulations for oil-extended types (6.3.2.2).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

1 Scope

This document 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 (SBRs), 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** % mass fraction	**Block content** % mass fraction
Series A			
Emulsion SBR	Random	≤50	0
Solution SBR	Random	≤50	0
Solution SBR	Partial block	≤50	≤30
Series B			
Emulsion SBR	Random	>50	0
Solution SBR	Random	>50	0
Solution SBR	Partial block	≤50	>30

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 247-1:2018, *Rubber — Determination of ash — Part 1: Combustion method*

ISO 247-2:2018, *Rubber — Determination of ash — Part 2: Thermogravimetric analysis (TGA)*

ISO 248-1, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 248-2, *Rubber, raw — Determination of volatile-matter content — Part 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit*

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

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

ISO 2393:2014, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*

ISO 6502-2, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 2: Oscillating disc curemeter*

ISO 6502-3, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 3: Rotorless curemeter*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Sampling and further preparative procedures

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

4.2 Prepare test samples in accordance with ISO 1795.

5 Physical and chemical tests on raw rubber

5.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1 on a test sample prepared in accordance with the preferred method of ISO 1795 (unmilled test sample). Record the result as ML(1 + 4) at 100 °C.

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

Alternatively, the Mooney viscosity can be determined on a test sample prepared by the milling procedure of ISO 1795. However, this method gives poorer reproducibility and the results can be different.

5.2 Volatile matter

Determine the volatile-matter content as specified in ISO 248-1 or ISO 248-2.

5.3 Ash

Determine the ash in accordance with either method A, or method C of ISO 247-1:2018, or method A of ISO 247-2:2018.

6 Preparation of the test mixes

6.1 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.

Table 2 — Standard test formulations

Material	Parts mass fraction	
	Series A	Series B
Styrene-butadiene rubber (SBR) (including oil in oil-extended SBR)	100,00	—
Standard SBR 1500[a]	—	65,00
Series B SBR	—	35,00
Sulfur	1,75	1,75
Stearic acid	1,00	1,00
Industry reference black[b]	50,00	35,00
Zinc oxide	3,00	3,00
TBBS[c]	1,00	1,00
Total (formulation mass)	156,75	141,75
Mill batch factor	4	4

[a] The previously used SBR 1500 EST is no longer available. It is therefore necessary to use another commercially available SBR 1500. The type shall be agreed between the interested parties.

[b] Use the current industrial reference black. Dry the material for 1 h at 125 °C ± 3 °C and store in a tightly closed container.

[c] *N*-tert-butylbenzothiazole-2-sulfenamide. This is 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 checked every six months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

6.2 Alternative formulations for oil-extended types

ASTM D3185 specifies the test formulations given in Table 3 for evaluation of general-purpose, oil-extended SBR, depending on the oil content of the rubber. These test formulations can be used as alternatives to the test formulations given in Table 2.

Table 3 — Alternative test formulations for oil-extended types

	Quantity mass fraction					
Formulation number	**1B**	**2B**	**3B**	**4B**	**5B**	**6B**
Parts of oil	**25**	**37,5**	**50**	**62,5**	**75**	***Y***[a]
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
Industry reference black[b]	62,50	68,75	75,00	81,25	87,50	(100 + *Y*)/2
TBBS[c]	1,25	1,38	1,50	1,63	1,75	(100 + *Y*)/100
Total (formulation mass)	194,50	213,38	232,25	251,13	270,00	
Batch factor[d]	2,4	2,2	2,0	1,9	1,7	

[a] *Y* = parts of oil, mass fraction, per 100 parts of base polymer in the oil-extended rubber.

[b] Use the current industrial reference black. Dry the material for 1 h at 125 °C ± 3 °C and store in a tightly closed container.

[c] *N*-tert-butylbenzothiazole-2-sulfenamide. This is 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 checked every six months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

[d] The batch factor is the coefficient for calculating the mill batch mass, which maintains that the amount of mill batch mass is mostly similar for mixing tests. The mill batch mass is equal to the formulation mass multiplied by the batch factor. The value is used only for Method A2 in 6.3.2.2 (mill mixing).

6.3 Procedure

6.3.1 Equipment and procedure

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

Three alternative mixing procedures are specified, but in accordance with ISO 2393, the laboratory internal mixer procedure is preferred.

— Method A1 and A2: mill mixing.

— Method B: single-stage mixing using a laboratory internal mixer (the preferred procedure).

— Method C: two-stage mixing using a laboratory internal mixer for initial mixing and a mill for final mixing.

6.3.2 Method A1 and A2 — Mill-mixing procedure

6.3.2.1 Method A1 — Procedure for the standard test formulations

The standard laboratory mill batch mass, in grams, shall be based on four times the formulation mass in Table 2 (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 can be necessary.

		Series A		Series B	
		Duration (min)	Cumulative time (min)	Duration (min)	Cumulative time (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	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 the SBR 1500, add the rubber [homogenized as in step a) above] 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 any 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
f)	Add the zinc oxide and the TBBS with the mill opening still at 1,8 mm.	3,0	26,0	3,0	28,0
g)	Make three 3/4 cuts from each side.	2,0	28,0	2,0	30,0
h)	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.	2,0	30,0	2,0	32,0

i) Sheet the batch to an approximate thickness of 6 mm by opening the mill and passing the stock through the mill four times, folding it back on itself each time. Determine the mass of the batch in accordance with ISO 2393. If the mass of the batch differs from the theoretical value by more than +0,5 % or −1,5 %, discard the batch and remix. Remove sufficient material for curemeter testing.

j) Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ring test pieces in accordance with ISO 37.

k) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible, at standard temperature and humidity as defined in ISO 23529.

6.3.2.2 Method A2 — Procedure for the test formulations of oil-extended types

The standard laboratory mill batch mass, in grams, shall be based on the test formulations mass of oil-extended types times batch factor in Table 3. 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 can be necessary.

		Series A	
		Duration (min)	Cumulative time (min)
a)	Set the mill opening at 1,15 mm and band the rubber on the slow roll and make 3/4 cuts every 30 s from alternate sides.	7,0	7,0
b)	Add the sulfur slowly and evenly across the mill at a uniform rate.	2,0	9,0
c)	Add the stearic acid. Make one 3/4 cut from each side after the stearic acid has been incorporated.	2,0	11,0

		Series A	
		Duration (min)	Cumulative time (min)
d)	Add the carbon black evenly across the mill at a uniform rate. When about half the black is incorporated, open the mill to 1,25 mm and make one 3/4 cut from each side. Then add the remainder of the carbon black. When all the black has been incorporated, open the mill to 1,40 mm and make one 3/4 cut from each side.	10,0	21,0
e)	Add the zinc oxide and the TBBS with the mill opening still at 1,4 mm.	3,0	24,0
f)	Make three 3/4 cuts from each side and cut the batch from the mill.	2,0	26,0
g)	Set the rolls at 0,8 mm. Pass the rolled batch endwise through the mill six times.	2,0	28,0
h)	Sheet the batch to an approximate thickness of 6 mm by opening the mill and passing the stock through the mill four times, folding it back on itself each time.	1,0	29,0
i)	Determine the mass of the batch in accordance with ISO 2393. If the mass of the batch differs from the theoretical value by more than +0,5 % or −1,5 %, discard the batch and remix. Remove sufficient material for curemeter testing.		
j)	Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ring test pieces in accordance with ISO 37.		
k)	Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible, at standard temperature and humidity as defined in ISO 23529.		

6.3.3 Method B — Single-stage mixing using a laboratory internal mixer

For laboratory internal mixers having nominal capacities of 65 cm^3 to about 2 000 cm^3, the batch mass shall be equal to the nominal mixer capacity, in cubic centimetres, multiplied by the density of the compound. For each batch mixed, the laboratory internal mixer conditions shall be the same during the preparation of a series of identical mixes. At the beginning of each series of test mixes, a machine-conditioning batch shall be mixed using the same formulation as the mixes under test. The laboratory internal mixer shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next. The temperature control conditions shall not be altered during the mixing of a series of test batches.

The mixing technique shall be such as to obtain a good dispersion of all the ingredients.

The temperature of the batch discharged on completion of mixing shall not exceed 120 °C. If necessary, adjust the batch mass or the mixer head starting temperature so that this condition is met.

NOTE 1 The mixing conditions given in Table A.6 for various sizes of laboratory internal mixer can be helpful.

Compounding materials other than rubber, carbon black and oil can be added to laboratory internal mixer batches more precisely and with greater ease if they are previously blended together in the proportions required by the formulation. Such blends can be made using a mortar and pestle, by mixing for 10 min in a biconical blender with the intensifier bar turning, or by mixing in another type of blender for five 3 s periods, scraping the inside of the blender to dislodge material stuck to the sides after each 3 s mix. A Waring® blender[1)] has been found suitable for this method.

WARNING — If mixed longer than 3 s, the stearic acid can melt and prevent good dispersion.

1) A Waring blender is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

NOTE 2 A general mixing procedure for the laboratory internal mixer is as follows:

		Duration (min)	Cumulative time (min)
a)	Load the rubber, lower the ram, and allow the rubber to be masticated.	1,0	1,0
b)	Raise the ram and add the pre-blended zinc oxide and stearic acid taking care to avoid any loss. Then add the carbon black, sweep the orifice, and lower the ram.	1,0	2,0
c)	Allow the batch to mix.	5,0	7,0
d)	Raise the ram and add the pre-blended sulfur and TBBS taking care to avoid any loss. Sweep the orifice and lower the ram.	2,0	9,0
e)	Turn off the motor, raise the ram, remove or open the mixing chamber, and discharge the batch. Record the maximum batch temperature.		

After discharging the mixed batch, pass it through a mill set at 50 °C ± 5 °C, once at a 0,5 mm mill opening and then twice at a 3,0 mm mill opening.

Determine the mass of the batch and record it. If it differs from the theoretical value by more than +0,5 % or –1,5 %, discard the batch and remix.

Prepare a test piece for determining the vulcanization characteristics in accordance with ISO 6502-2 or ISO 6502-3, if required. Condition the test piece for 2 h to 24 h, if possible, at a standard temperature and humidity as defined in ISO 23529, before testing.

If required, sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ring test pieces in accordance with ISO 37. To obtain the effects of mill direction, pass the folded batch four times between mill rolls set at the appropriate opening and a temperature of 50 °C ± 5 °C. Allow the sheet to cool on a flat, dry surface.

Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible, at standard temperature and humidity as defined in ISO 23529.

6.3.4 Method C — Two-stage mixing using a laboratory internal mixer for initial mixing and a mill for final mixing

6.3.4.1 Stage 1 — Initial mixing procedure

The mixing technique shall be such as to obtain a good dispersion of all the ingredients.

The temperature of the batch discharged on completion of mixing shall be between 150 °C and 170 °C. If necessary, adjust the batch mass or the mixer head starting temperature so that this condition is met.

NOTE 1 The following mixing conditions have been found to be suitable for a laboratory internal mixer with a nominal capacity of 1 170 cm^3 ± 40 cm^3.

— batch mass: 8,5 times the formulation mass (8,5 × 156,75 g = 1 332,37 g) for series A rubbers, 9,5 times the formulation mass (9,5 × 141,75 g = 1 346,62 g) for series B rubbers;

— rotor speed: 77 r/min ± 10 r/min.