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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 — Méthode 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).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This sixth edition cancels and replaces the fifth edition (ISO 2322:2009), which has been technically revised. In particular:

- 5.3.1 states that the laboratory internal mixer procedure is the preferred method;
- Note 2 in 5.3.3 has been updated for more clarity.

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

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard 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 and to ensure compliance with any national regulatory conditions.

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** % 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 247:2006, *Rubber — Determination of ash*

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

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

ISO 6502, *Rubber — Guide to the use of curemeters*

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

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 samples 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 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 mill massing procedure of ISO 1795. However, this method gives poorer reproducibility and the results might 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 and ISO 248-2.

4.3 Ash

Determine the ash in accordance with method A or method B of ISO 247:2006.

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.

Table 2 — 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	156,75	141,75

[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 6 mo. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

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, 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	194,50	213,38	232,25	251,13	270,00	

[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 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

5.3 Procedure

5.3.1 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 A: 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.

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 might 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 ISO 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.

5.3.3 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 might 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 has been found suitable for this method. Caution: if mixed longer than 3 s, the stearic acid can melt and prevent good dispersion.

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 3417 or ISO 6502, 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.

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

5.3.4.1 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 cm3 ± 40 cm3.

— 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.

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

	Duration (min)	**Cumulative time** (min)
a) Adjust the temperature of the laboratory internal mixer to a starting temperature of 50 °C ± 3 °C. Close the discharge door, set the rotor speed, and raise the ram.	—	—
b) Load the rubber, lower the ram, and allow the rubber to be masticated.	0,5	0,5
c) Raise the ram and load the zinc oxide, stearic acid, and carbon black. Lower the ram.	0,5	1,0
d) Allow the batch to mix.	2,0	3,0

		Duration (min)	**Cumulative time** (min)
e)	Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0,5	3,5
f)	Allow the batch to mix.	1,5	5,0
g)	Discharge the batch.		

After discharging the batch, immediately check the temperature of the batch with a suitable temperature-measuring device. If the temperature as measured falls outside the range 150 °C to 170 °C, discard the batch. Pass the batch three times through a mill with a mill opening of 2,5 mm and a roll temperature of 50 °C ± 5 °C. Sheet the batch to a thickness of approximately 10 mm and determine the mass of the batch. If the mass differs from the theoretical value by more than +0,5 % or −1,5 %, discard the batch and remix.

Leave the batch for at least 30 min and up to 24 h, if possible at standard temperature and humidity as defined in ISO 23529.

The smaller laboratory internal mixers do not provide enough compound for the final mill mixing, as a batch mass of three times the formula mass is required. In such cases, the laboratory internal mixer can be used for the final mixing. It might be necessary to adjust the head temperature or the batch mass so that the final temperature of the discharged batch does not exceed 120 °C.

5.3.4.2 Final mill-mixing procedure

During final mixing, maintain a good rolling bank at the nip of the rolls. If this is not attained with the nip settings specified, small adjustments to the mill openings might be necessary.

		Duration (min)	**Cumulative time** (min)
a)	The standard laboratory mill batch mass, in grams, shall be based on three times the formula mass.	—	—
b)	Set the mill temperature at 50 °C ± 5 °C and the mill opening to 1,5 mm.	—	—
c)	Band the masterbatch on the slow roll.	1,0	1,0
d)	Add the sulfur and the accelerator. Do not cut the band until the sulfur and accelerator are completely dispersed.	1,5	2,5
e)	Make three 3/4 cuts from each side, allowing 15 s between each cut.	2,5	5,0
f)	Cut the batch from the mill. Set the mill opening at 0,8 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternately.	2,0	7,0
g)	Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37. 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.		
h)	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.		