INTERNATIONAL STANDARD

ISO 2322

Seventh edition
2023-05

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

Reference number
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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.

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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.

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
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 can 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.

6.3.4.2 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 can 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 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.		

7 Evaluation of vulcanization characteristics by a curemeter test

7.1 Using an oscillating-disc curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, t_{s1}, $t'_c(50)$, and $t'_c(90)$

in accordance with ISO 6502-2, using the following test conditions:

- oscillation frequency: 1,7 Hz (100 cycles per minute);
- amplitude of oscillation: 1° of arc;
- selectivity: to be chosen to give at least 75 % of full-scale deflection at M_H (note that, with some rubbers, 75 % will possibly not be attainable);
- die temperature: 160 °C ± 0,3 °C;
- pre-heat time: none.

7.2 Using a rotorless curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, t_{s1}, $t'_c(50)$, and $t'_c(90)$

in accordance with ISO 6502-3, using the following test conditions:

- oscillation frequency: 1,7 Hz (100 cycles per minute);
- amplitude of oscillation: 0,5° of arc;
- selectivity: to be chosen to give at least 75 % of full-scale deflection at M_H (note that, with some rubbers, 75 % might not be attainable);
- die temperature: 160 °C ± 0,3 °C;
- pre-heat time: none.

8 Evaluation of tensile stress-strain properties of vulcanized test mixes

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

Alternatively, vulcanize sheets at 150 °C for three periods selected from a cure series of 10 min, 15 min, 20 min, 25 min, 30 min, 35 min and 50 min. Note, however, that these conditions give results which are different from those obtained by the preferred vulcanization conditions.

The three periods of cure selected shall cover the undercure, optimum cure and overcure of the rubber under test.

Condition the vulcanized sheets for 16 h to 96 h at a standard temperature and, if possible, a standard humidity as defined in ISO 23529.

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

9 Precision

See Annex A.

10 Test report

The test report shall include the following:

a) a reference to this document, i.e. ISO 2322:2023;

b) all details necessary for the identification of the sample;

c) the method used for the volatile-matter content determination (ISO 248-1 or ISO 248-2);

d) the method used for the ash determination (method A or method C of ISO 247-1:2018 or method A of ISO 247-2:2018);

e) the reference materials used;

f) the standard test formulation used;

g) the mixing procedure used;

h) the conditioning environment used in 6.3.2.1 k) or 6.3.2.2 k) or 6.3.3 or 6.3.4.2 h) and in Clause 8;

i) the curemeter test used in Clause 6 (ISO 6502-2 or ISO 6502-3);

j) the time at which M_H was determined in the curemeter test;

k) the vulcanization temperature and times used in Clause 8;

l) any unusual features noted during the determinations;

m) any operation not included in this document or in the International Standards to which reference is made, as well as any operation regarded as optional;

n) the results and the units in which they have been expressed;

o) the date of the test.

Annex A
(informative)

Precision

A.1 General

Two interlaboratory test programmes (ITPs) were organized in 1986 and in 2003. The 1986 ITP was conducted with mill mixing. The other one was conducted with mill mixing and laboratory internal mixer mixing.

The precision results as determined by these ITPs cannot be applied to acceptance or rejection testing of any group of materials or products without documentation that the results of the precision determination actually apply to the products or materials tested.

A.2 Precision obtained for mill mixing in the 1986 ITP

A.2.1 Details of the ITP

A.2.1.1 Two series A formulations were tested:

- A-1, containing an oil-extended SBR, type 1712;
- A-2, containing a non-oil-extended SBR, type 1500.

In addition, one series B formulation was tested:

- A-3, with a high-styrene SBR.

Mixes of these formulations were prepared in each of the 13 laboratories participating in the ITP on each of two days approximately one week apart. The mixes were prepared from special samples of all the necessary materials sent to each laboratory prior to actual testing. For each material, the samples were drawn from a homogeneous batch. Stress-strain tests were conducted on cured sheets prepared from each of the mixes or compounds as specified by the test programme.

A.2.1.2 The modulus (stress at 300 % strain), tensile strength, and percent elongation at break were determined on dumb-bell test pieces in accordance with ISO 37, taking as the test result the median value of five individual determinations. The precision thus calculated is a type 2 precision, and the time period for repeatability and reproducibility is on a scale of days.

See Annex B for comments on the precision results.

A.2.2 Precision results

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272:1986[2].

The precision results are given in Table A.1. The symbols used in Table A.1 are defined as follows:

- *r* = repeatability, in measurement units. This is the value below which the absolute difference between two within-laboratory test results can be expected to lie with a specified probability.

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[2] Withdrawn standard. Replaced by ISO/TR 9272:2005.

- (*r*) = repeatability, in percent (relative).

The two within-laboratory test results are obtained using the same method on nominally identical test materials under the same conditions (same operator, apparatus and laboratory) and within a specified time period. Unless stated otherwise, the specified probability is 95 %.

- *R* = reproducibility, in measurement units. This is the value below which the absolute difference between two between-laboratory test results can be expected to lie with a specified probability.

- (*R*) = reproducibility, in percent (relative).

The two between-laboratory test results are obtained using the same method on nominally identical test materials under different conditions (different operators, apparatus and laboratories) and within a specified time period. Unless stated otherwise, the specified probability is 95 %.

Table A.1 — Type 2 precision for stress-strain testing after mixing with a mill

Formulation	**Average value**	**Within-laboratory**		**Between laboratories**	
		r	(*r*)	*R*	(*R*)
Modulus (at 300 % strain), MPa					
SBR A-2	12,3	1,62	13,1	3,83	31,1
SBR A-1	14,6	1,80	12,3	3,86	26,5
SBR A-3	16,0	2,36	14,8	6,12	38,2
Tensile strength, MPa					
SBR A-2	20,3	2,05	10,1	3,09	15,2
SBR A-3	23,4	4,70	20,1	4,70	20,1
SBR A-1	25,5	2,50	9,79	3,60	14,1
Elongation at break, %					
SBR A-3	434	52,0	11,9	200	46,2
SBR A-2	481	56,6	11,8	103	21,5
SBR A-1	481	51,6	10,7	66,2	13,8

A.3 Precision obtained for both mill and internal mixer in the 2003 ITP

A.3.1 Details of the ITP

The ITP to determine the precision using a laboratory mill and various sizes of internal mixer was conducted on series A test formulations containing SBR 1500 (Europrene 1500 EST8). The ITP was conducted as part of the revision of ISO 2393:1994.

NOTE The internal mixers used covered those types often described as “miniature”, “internal” and “laboratory”.

The ITP was carried out using the precision procedures and guidelines described in ISO/TR 9272:2005[3] (current edition). Reference should be made to ISO/TR 9272 for other details and terminology on precision evaluation.

Two types of measurement were used to evaluate each type of mixing procedure: stress-strain testing and curemeter testing. Stress-strain property precision was determined for stress (modulus) at 100 %, 200 %, and 300 % elongation, elongation at break, and tensile strength. Curemeter precision was determined for M_H (maximum torque), M_L (minimum torque), t_{s1} (scorch time or time to a 1 dN·m rise), $t'_c(50)$ (time to 50 % of full torque), and $t'_c(90)$ (time to 90 % of full torque). A type 2 precision was determined. The compounds were prepared and tested on a day 1/day 2 basis one week apart.

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[3] Withdrawn standard. Replaced by ISO 19983:2017.

On each day, homogeneous materials were used to prepare a separate mix for each type of compound, and the appropriate testing (stress-strain and curemeter) was conducted on each of these mixes for each of the two test days. The test result was taken as the average of two replicate test values obtained on each of the two separate test days and the precision was calculated from these test results.

For the internal-mixer testing, nine laboratories participated in the test programme. However, each laboratory did not have the same type or size of mixer. The sizes of the mixers used in the ITP were 80 cm^3, 270 cm^3, 379 cm^3, 588 cm^3, 1 580 cm^3 and 1 600 cm^3.

In addition to determining the precision, data analysis was carried out to determine if, or how, the type of mixer (size and other operating conditions) influenced the test results (see further discussion of this issue in ISO 2393:2014, Annex C). Some laboratories submitted data from more than one size of mixer, and the data from these separate mixers were incorporated into the ITP as data from individual laboratories. This gave a database equivalent to 12 laboratories. Each of the mixer-laboratory combinations was designated as a pseudo-laboratory.

A.3.2 Precision results

The precision results for both types of mixing, for both stress-strain and curemeter testing, are given in Tables A.2 to A.5. These results were obtained using the option 1 outlier deletion procedures described in ISO/TR 9272. General statements for the use of the precision results are given below. These are given in terms of both the absolute precision, *r* and *R*, and also for relative precision, (*r*) and (*R*) (see additional discussion below).

- Repeatability — The repeatability, or local domain precision, for each of the test methods has been established as the values given in Tables A.2 to A.5 for each measurement parameter listed in the tables. Two individual test results obtained in the same laboratory (by the proper use of this document) that differ by more than the tabulated values for *r*, in measurement units, or (*r*), in percent, should be considered as suspect, i.e. to have come from different populations. Such a situation should suggest that some appropriate investigative action be taken.
- Reproducibility — The reproducibility, or global domain precision, for each of the test methods has been established as the values found in Tables A.2 to A.5 for each measurement parameter listed in the tables. Two individual test results obtained in different laboratories (by the proper use of this document) that differ by more than the tabulated values for *R*, in measurement units, or (*R*), in percent, should be considered as suspect, i.e. to have come from different populations. Such a situation should suggest that some appropriate investigative action be taken.

A.3.3 Comparison of relative reproducibility

The grand average or overall relative reproducibility, (*R*), values in Tables A.2 to A.5 give an indication of the difference in relative reproducibility between mill and internal-mixer mixing.

The overall (*R*) values in Tables A.2 and A.3 for mill-mixing stress-strain and curemeter properties are 19,0 % and 20,3 %, respectively, and those in Tables A.4 and A.5 for internal-mixer stress-strain and curemeter properties are 17,9 % and 28,7 %, respectively.

For the stress-strain properties, the overall relative reproducibility is essentially the same for mill mixing and internal-mixer mixing: 19,0 % and 17,9 %, respectively. For curemeter testing, however, the relative reproducibility for mill mixing is different from that for internal-mixer mixing: 20,3 % versus 28,7 %, respectively. This higher between-laboratory variation is no doubt primarily attributable to the differences between the internal mixers used in the various laboratories.

A.3.4 Bias

Bias is the difference between a measured average test result and a reference or true value for the measurement in question. Reference values do not exist for this test method and therefore bias cannot be evaluated.