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Isoprene rubber (IR) — Non-oil-extended, solution-polymerized types — Evaluation procedures

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Isoprene rubber (IR) — Non-oil-extended, solution-polymerized types — Evaluation procedures

Caoutchouc isoprène (IR) — Types polymérisés en solution et non étendus à l'huile — 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).

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 sixth edition cancels and replaces the fifth edition (ISO 2303:2011), which has been technically revised.

The main change compared to the previous edition is to allow the use of the method given in ISO 248-2 in 5.2 and in Clause 10.

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.

INTERNATIONAL STANDARD ISO 2303:2019(E)

Isoprene rubber (IR) — Non-oil-extended, solution-polymerized types — Evaluation procedures

WARNING — Persons using this document should be familiar with normal laboratory practices. 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, for general-purpose non-oil-extended, solution-polymerized polyisoprene rubbers (IR):

— physical and chemical tests on raw rubbers;

— standard materials, a standard test formulation, equipment and processing methods for evaluating the vulcanization characteristics.

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 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*

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 6502-1, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 1: Introduction*

ISO 6502-2, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 2: Oscillating disc 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 terminological databases for use in standardization at the following addresses:

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

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

4 Sampling and sample preparation

4.1 A laboratory sample of approximately 1,5 kg shall be taken by the method described in ISO 1795.

4.2 Preparation of the test portion shall be in accordance with ISO 1795.

5 Physical and chemical tests on raw rubber

5.1 Mooney viscosity

The Mooney viscosity shall be determined in accordance with ISO 289-1 on a test portion prepared as described in ISO 1795 (without massing).

The result shall be recorded as ML(1 + 4) at 100 °C.

5.2 Volatile matter

The volatile-matter content shall be determined in accordance with ISO 248-1 or ISO 248-2.

5.3 Ash

The ash content shall be determined in accordance with ISO 247-1.

6 Preparation of the test mixes for evaluation of isoprene rubbers

6.1 Standard test formulation

The standard test formulation is given in Table 1.

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 interested parties.

Table 1 — Standard test formulation for evaluation of IR rubbers

Material	Parts by mass
Isoprene rubber (IR)	100,00
Stearic acid	2,00
Zinc oxide	5,00
Sulfur	2,25
Industry reference black (N330)	35,00
TBBS[a]	0,70
Total	144,95

[a] TBBS or *N-tert*-butylbenzothiazole-2-sulfenamide in accordance with ISO 6472. This shall be supplied in powder form having an initial insoluble-matter content, in accordance with ISO 11235, of less than 0,3 %. The material shall be stored at room temperature in a closed container and insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded. TBBS may be purified by reprocessing, e.g. by recrystallization; the procedure for this is beyond the scope of this document.

6.2 Procedure

6.2.1 Equipment and procedure

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

6.2.2 Mill mixing procedures

6.2.2.1 General

Two mill mixing procedures are specified: methods A and B. The mixing time is shorter in Method B than in Method A.

The two methods do not necessarily give identical results. In laboratory cross-checks or in a series of evaluations, the same procedure shall be used in all cases.

In both methods, the standard laboratory mill batch mass, in grams, shall be based on four times the formula mass. The surface temperature of the rolls shall be maintained at 70 °C ± 5 °C throughout the mixing.

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified in 6.2.2.2 and 6.2.2.3, small adjustments to the mill openings might be necessary.

6.2.2.2 Method A

	Duration (min)	Cumulative time (min)
a) Pass the rubber between the mill-rolls twice without banding, with the mill opening set at 0,5 mm, for approximately 2 min and weigh the rubber;	2,0	2,0
b) Band the rubber with the mill opening set at 1,4 mm and make two 3/4 cuts from each side;	2,0	4,0

NOTE Some types of isoprene rubber go to the back roll, in which case the stearic acid should be added and, after its incorporation, the rubber can usually be transferred to the front roll. In addition, certain tougher types of isoprene rubber might require slightly longer breakdown before the addition of other materials in order to obtain a good rolling bank.

c) Set the mill opening to 1,7 mm and add the stearic acid. Make one 3/4 cut from each side;	2,0	6,0
d) Add the zinc oxide and the sulfur. Make two 3/4 cuts from each side;	3,0	9,0
e) Add the carbon black evenly across the mill at a uniform rate. When approximately half the black has been incorporated, open the mill to 1,9 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, make one 3/4 cut from each side;	13,0	22,0
f) Add the TBBS with the mill opening still at 1,9 mm. Make three 3/4 cuts from each side;	3,0	25,0

g)	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.	3,0	28,0

h) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

i) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 6502-1 or ISO 6502-2. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.

j) Sheet the batch to approximately 2,2 mm for preparing test slabs 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 prior to vulcanizing, if possible at a standard temperature and humidity as defined in ISO 23529.

6.2.2.3 Method B

		Duration (min)	Cumulative time (min)
a)	Pass the rubber between the rolls twice without banding, with the mill opening set at 0,5 mm ± 0,1 mm, then band the rubber between the rolls with the mill opening gradually increased to 1,4 mm.	2,0	2,0
b)	Add the stearic acid. Make one 3/4 cut from each side.	2,0	4,0
c)	Add the sulfur and the zinc oxide. Make two 3/4 cuts from each side.	3,0	7,0
d)	Add half of the carbon black. Make two 3/4 cuts from each side.	3,0	10,0
e)	Add the remaining half of the carbon black and any black that has dropped into the mill pan. Make three 3/4 cuts from each side.	5,0	15,0
f)	Add the TBBS. Make three 3/4 cuts from each side.	3,0	18,0
g)	Cut the batch from the mill. Set the mill opening to 0,5 mm ± 0,1 mm and pass the rolled batch endwise through the rolls six times.	2,0	20,0

h) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

i) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 6502-1 or ISO 6502-2. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.

j) Sheet the batch to approximately 2,2 mm for preparing test slabs 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 prior to vulcanizing, if possible at a standard temperature and humidity as defined in ISO 23529.

6.2.3 Laboratory internal mixer (LIM) mixing procedure

6.2.3.1 General

For a LIM having a nominal mixing capacity 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 compound density. The LIM conditions shall be the same for each batch mixed 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 LIM shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next. Temperature control settings shall not be altered during the mixing of a series of test batches.

6.2.3.2 Single-stage mixing procedure

The mixing technique shall be such as to obtain a good dispersion of all the ingredients. The final temperature of the batch discharged after mixing shall not exceed 120 °C. If necessary, adjust the batch mass, head temperature and/or rotor speed so that this condition is met.

NOTE 1 Compounding materials other than rubber, carbon black and oil can be added to LIM 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 a blender for five 3 s periods and scraping the inside of the blender to dislodge materials stuck to the sides after each 3 s mix. A Waring blender[1)] has been found suitable for this method. Caution: if mixed longer than 3 s, the stearic acid might melt and prevent good dispersion.

NOTE 2 An example of a mixing procedure for a LIM 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, sulfur, stearic acid and TBBS, 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.	7,0	9,0
d)	Turn off the motor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature.		
e)	After discharging the mixed batch, pass it through a mill set at 70 °C ± 5 °C once at a 0,5 mm mill opening, and then twice at a 0,3 mm mill opening.		
f)	Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.		
g)	Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 6502-1 or ISO 6502-2. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.		
h)	Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37.		
i)	Condition the batch for 2 h to 24 h prior to vulcanizing, if possible at a standard temperature and humidity as defined in ISO 23529.		

1) This 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.

6.2.3.3 Two-stage mixing including mill for final mixing procedure

6.2.3.3.1 General

The LIM shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next.

6.2.3.3.2 Stage 1 — Initial mixing stage

The mixing technique shall be such as to obtain a good dispersion of all the ingredients.
The final temperature of the batch discharged after mixing shall be between 150 °C and 170 °C. If necessary, adjust the batch mass, head temperature and/or rotor speed so that this condition is met.

NOTE An example of a mixing procedure for the initial mixing is as follows:

		Duration (min)	Cumulative time (min)
a)	Adjust the temperature of the LIM to a starting temperature of 60 °C ± 3 °C. Close the discharge door, set the rotor speed to 77 rpm, start the rotors and raise the ram.	—	—
b)	Load half of the rubber, all the carbon black, zinc oxide and stearic acid, then the remaining half of the rubber. Lower the ram.	0,5	0,5
c)	Allow the batch to mix.	3,0	3,5
d)	Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0,5	4,0
e)	Allow the batch to mix.	0,5	4,5
f)	Discharge the batch.	1,5	6,0

g) After discharging the batch, immediately check the temperature of the batch with a suitable measuring device. If the temperature as measured falls outside the range 150 °C to 170 °C, discard the batch.

h) Pass the batch three times through a mill with a mill opening of 2,5 mm and a temperature of 70 °C ± 5 °C.

i) Sheet the batch to an approximate thickness of 10 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

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

The smaller LIMs do not provide enough compound for the final mill mixing, as a batch mass of three times the formula mass is required. In these cases, the LIM may be used for the final mixing. The head temperature and/or the batch mass may be adjusted so that the final temperature of the discharged batch does not exceed 120 °C.

6.2.3.3.3 Stage 2 — Final mixing stage

Rest the batch for at least 30 min, or until it reaches room temperature, before proceeding with the final mixing stage. The mixing technique shall be such as to obtain a good dispersion of all the ingredients. The final temperature of the batch discharged after mixing shall not exceed 120 °C.

When a LIM is used, adjust, if necessary, the batch mass, the head temperature and/or the rotor speed so that this condition is met. When mill mixing is used, set the surface temperature of the rolls to 70 °C ± 5 °C and maintain it at this temperature during mixing. The standard laboratory mill batch

mass, in grams, shall be based on two times the formula mass. A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings given hereunder, small adjustments to the mill openings may be necessary.

NOTE 1 An example of a LIM mixing procedure for the final mixing stage is as follows:

		Duration (min)	Cumulative time (min)
a)	Close the discharge door, set the rotor speed and raise the ram.	—	—
b)	Load the rubber, the sulfur and the accelerator, and lower the ram.	0,5	0,5
c)	Allow the batch to mix.	1,5	2,0
d)	Raise the ram, open the mixing chamber and discharge the batch. Record the maximum batch temperature.	0,5	2,5
e)	After discharging the mixed batch, pass it four times through a mill at a roll temperature of 70 °C ± 5 °C and with a mill opening of 0,8 mm.	0,5	3,0

f) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

g) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 6502-1 or ISO 6502-2. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.

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

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

NOTE 2 An example of a mill mixing procedure for the final mixing is as follows:

		Duration (min)	Cumulative time (min)
a)	Set the mill temperature at 70 °C ± 5 °C and the mill opening to 1,9 mm. Band the masterbatch on the slow roll.	—	—
b)	Add the accelerators. Do not cut the band until the accelerators are completely dispersed. Then make three 3/4 cuts from each side.	3,0	3,0
c)	Add the sulfur. Do not cut the band until the sulfur is completely dispersed. Then make three 3/4 cuts from each side.	3,0	6,0
d)	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	8,0
e)	Set the mill opening at approximately 6 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternately. Sheet the batch.	1,0	9,0

f) Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

g) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 6502-1 or ISO 6502-2. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.

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

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

7 Evaluation of vulcanization characteristics by a curemeter test

WARNING — Formation of nitrosamines is possible during the cure.

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 % might 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:

F_L, F_H at defined time, $t'_{c(}50)$ and $t'_c(90)$

in accordance with ISO 6502-1, 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 F_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

Vulcanize sheets at 135 °C for three periods chosen from a cure series of 20 min, 30 min, 40 min and 60 min.

The three periods of cure shall be chosen to cover the undercure, optimum cure and overcure of the material 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 for the information on precision data for both mill mixer and laboratory internal mixer.

See Annex B for the information on additional precision data for natural rubber.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 2303:2019;

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

c) the time and temperature conditions used for the Mooney viscosity determination, and whether a massing process was used;

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

e) the method used for the ash determination (Method A or B of ISO 247-1:2018);

f) the standard test formulation used;

g) the reference materials used;

h) the mixing procedure used;

i) the conditioning conditions used in 6.2.2.2, 6.2.2.3, 6.2.3.2 or 6.2.3.3;

j) for Clause 7:

   — the reference standard,

   — the time for M_H or F_H;

k) the vulcanization periods used in Clause 8;

l) any unusual features noted during the determination;

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 data for both mill mixer and laboratory internal mixer

A.1 General

An interlaboratory test programme (ITP) was carried out using the procedures and guidelines described in ISO/TR 9272. Reference should be made to this Technical Report for other details and for terminology on precision determination.

A type 2 (interlaboratory) precision was determined for cure characteristics, using an oscillating-disc curemeter. A sample of isoprene rubber was used in the ITP. Five laboratories participated in a programme using the mill mixing procedure (method A) and eight laboratories in a programme using the two-stage LIM mixing procedure, utilizing a LIM for initial and a mill for final mixing. The test was done on three different days in each laboratory.

The precision results as determined from this ITP may not be applied to acceptance or rejection testing of any group of materials or products without documentation that the results of this precision determination actually apply to the materials or products tested.

A.2 Results

A.2.1 General

The results of the precision calculation for repeatability and reproducibility are given in Table A.1 for the mill mixing procedure and Table A.2 for the LIM mixing procedure.

A.2.2 Repeatability

The repeatability r of the test method has been established as the appropriate value tabulated in Table A.1 or A.2. Two single test results that differ by more than this value should be considered suspect and suggest that some appropriate investigative action be taken.

A.2.3 Reproducibility

The reproducibility R of the test method has been established as the appropriate value tabulated in Table A.1 or A.2. Two single test results that differ by more than this value should be considered suspect and suggest that some appropriate investigative action be taken.

Table A.1 — Precision for mill mixing (method A)

Property	Units	Mean level[a]	Within-laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
M_L	dN·m	6,05	0,15	0,40	6,61	0,36	1,01	16,69
M_H	dN·m	39,87	0,25	0,69	1,73	1,73	4,86	12,19
t_{s1}	min	3,19	0,19	0,53	16,61	0,36	1,00	31,35
$t'_c(50)$	min	4,97	0,07	0,20	4,02	0,14	0,39	7,85
$t'_c(90)$	min	7,09	0,08	0,23	3,24	0,10	0,28	3,95

s_r is the repeatability standard deviation;

r is the repeatability, in measurement units;

(r) is the repeatability, in percent (relative);

s_R is the reproducibility standard deviation;

R is the reproducibility, in measurement units;

(R) is the reproducibility, in percent (relative).

[a] Measured at 160 °C, 1,7 Hz, 1° of arc — midpoint of range used for (r) and (R) calculations.

Table A.2 — Precision for two-stage LIM-mill mixing

Property	Units	Mean level[a]	Within-laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
M_L	dN·m	6,85	0,09	0,26	3,80	0,18	0,50	7,30
M_H	dN·m	39,12	0,44	1,24	3,17	1,15	3,21	8,20
t_{s1}	min	3,82	0,09	0,26	6,80	0,24	0,66	17,28
$t'_c(50)$	min	6,23	0,07	0,19	3,04	0,45	1,25	20,06
$t'_c(90)$	min	8,47	0,10	0,27	3,19	0,44	1,23	14,52

For the meanings of the symbols used for the precision parameters in the column headings, see Table A.1.

[a] Measured at 160 °C, 1,7 Hz, 1° of arc — midpoint of range used for (r) and (R) calculations.

Annex B
(informative)

Additional precision data for natural rubber

The following precision data for mill mixing (Tables B.1 and B.2) and LIM mixing (Tables B.3 and B.4), obtained with natural rubber, have been taken from ISO 1658:2009[2)]. They were determined not only for curemeter properties but also for stress-strain properties. The reader is referred to ISO 1658:2009, Annex B, for full details of the ITP and a discussion of the results.

Table B.1 — Precision (type 2) — Mill mixing — Stress-strain properties

Parameter measured	Mean level	Within-laboratory			Between laboratories			Number of laboratories
		s_r	r	(r)	s_R	R	(R)	
S_{100}, MPa	2,70	0,029	0,080	3,00	0,092	0,26	9,70	5
S_{200}, MPa	7,10	0,12	0,33	4,60	0,40	1,13	21,90	5
S_{300}, MPa	13,50	0,16	0,45	3,30	0,93	2,60	19,30	5
Elongation at break, %	527	11,2	31,5	20,2	38,0	106	20,20	6
Tensile strength, MPa	28,7	0,39	1,09	3,80	3,31	9,30	32,30	6
Average		—	—	6,98	—	—	20,70	

S_{100} is the stress (modulus) at 100 % elongation;

S_{200} is the stress (modulus) at 200 % elongation;

S_{300} is the stress (modulus) at 300 % elongation.

For the meanings of the symbols used for the precision parameters in the column headings, see Table A.1.

Table B.2 — Precision (type 2) — Mill mixing — Curemeter properties

Parameter measured	Mean level	Within-laboratory			Between laboratories			Number of laboratories
		s_r	r	(r)	s_R	R	(R)	
M_H, dN·m	14,70	0,22	0,62	4,20	1,96	5,50	37,30	4
M_L, dN·m	1,62	0,09	0,25	15,4	0,29	0,82	50,6	5
t_{s1}, min	1,58	0,04	0,12	7,60	0,39	1,09	69,1	5
$t'_c(50)$, min	3,17	0,12	0,34	10,60	0,27	0,75	23,5	6
$t'_c(90)$, min	5,40	0,12	0,34	6,30	0,19	0,53	9,90	5
Mooney viscosity ML(1+4) at 100 °C	51,8	2,35	6,57	12,7	3,85	10,8	20,8	5
Average		—	—	8,82	—	—	38,1	

For the meanings of the symbols used for the precision parameters in the column headings, see Table A.1.

2) Withdrawn.

Table B.3 — Precision (type 2) — LIM mixing — Stress-strain properties

Parameter measured	Mean level	Within-laboratory			Between laboratories			Number of laboratories
		s_r	r	(r)	s_R	R	(R)	
S_{100}, MPa	2,55	0,05	0,13	5,10	0,23	0,64	25,2	8
S_{200}, MPa	6,69	0,15	0,43	6,40	0,61	1,70	25,4	8
S_{300}, MPa	13,0	0,20	0,56	4,30	0,83	2,33	18,0	8
Elongation at break, %	518	7,10	19,9	3,80	19,6	54,9	10,6	6
Tensile strength, MPa	29,2	0,44	1,24	4,20	2,66	7,46	25,5	8
Average		—	—	4,76	—	—	20,9	

S_{100} is the stress (modulus) at 100 % elongation;

S_{200} is the stress (modulus) at 200 % elongation;

S_{300} is the stress (modulus) at 300 % elongation.

For the meanings of the symbols used for the precision parameters in the column headings, see Table A.1.

Table B.4 — Precision (type 2) — LIM mixing — Curemeter properties

Parameter measured	Mean level	Within-laboratory			Between laboratories			Number of laboratories
		s_r	r	(r)	s_R	R	(R)	
M_H, dN·m	14,9	0,15	0,41	2,80	0,81	2,26	15,2	7
M_L, dN·m	1,94	0,06	0,17	8,80	0,18	0,49	25,2	8
t_{s1}, min	1,57	0,04	0,12	7,40	0,33	0,91	58,2	9
$t'_c(50)$, min	3,00	0,06	0,17	5,70	0,34	0,95	31,7	7
$t'_c(90)$, min	5,40	0,09	0,26	4,90	0,33	0,93	17,3	6
Mooney viscosity ML(1+4) at 100 °C	55,8	1,42	3,97	7,10	2,19	6,12	11,0	8
Average		—	—	5,92	—	—	29,5	

For the meanings of the symbols used for the precision parameters in the column headings, see Table A.1.

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