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Natural rubber (NR) — Evaluation procedure

Caoutchouc naturel (NR) — 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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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 fifth edition cancels and replaces the fourth edition (ISO 1658:2015), which has been technically revised.

The main changes are as follows:

— in 6.3.4.1.2, change of the order of mixing process and reduction of the batch mass;

— in A.3, addition of the definition of the temperature according to ISO 23529.

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.

Natural rubber (NR) — Evaluation procedure

1 Scope

This document specifies

— physical and chemical tests on raw natural rubbers;

— standard materials, standard test formulae, equipment and processing methods for evaluating the vulcanization characteristics of natural rubber (NR).

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

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 2000:2020, *Rubber, raw natural — Guidelines for the specification of technically specified rubber (TSR)*

ISO 2007, *Rubber, unvulcanized — Determination of plasticity — Rapid-plastimeter method*

ISO 2393, *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 A laboratory sample of mass approximately 1,5 kg shall be prepared by the method described in ISO 1795.

4.2 The rubber shall be homogenized in accordance with ISO 1795.

4.3 Preparation of the test samples shall be 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 as indicated in 4.3. Record the result as ML(1+4) at 100 °C.

5.2 Volatile-matter content

Determine the volatile-matter content by the oven method specified in ISO 248-1 on a test sample prepared as indicated in 4.3.

5.3 Other specifications on requirements

Any specific values for physical and chemical properties other than the above (5.1 and 5.2) shall be based upon the grades given in ISO 2000:2020, Table 2.

6 Preparation of test mix

6.1 General

The following standard formulae and mixing procedures are recommended:

a) two gum-stock formulae for comparative testing of the vulcanization characteristics of natural rubber for use in non-black-filled compounds;

b) a black-filled formula for comparative testing of natural rubber for use in black-filled compounds;

c) a mill mixing procedure including masterbatches for the two gum-stock formulae;

d) a mill mixing procedure and a laboratory internal mixer (LIM) mixing procedure for the black-filled formula.

NOTE The black-filled formula [Formula (3) in Table 1] can also be used for comparative testing of isoprene rubbers (IRs).

6.2 Standard test formulae

The standard test formulae are given in Table 1.

The materials shall be national or international standard reference materials.

Table 1 — Standard test formulae

Material	Number of parts by mass		
	Formula (1) ACS 1	Formula (2) TBBS 1	Formula (3) Black-filled
Natural rubber	100,00	100,00	100,00
Zinc oxide	6,00	6,00	5,00
Sulfur	3,50	3,50	2,25
Stearic acid	0,50	0,50	2,00
Industry reference black (IRB) [a]	—	—	35,00
MBT [b]	0,50	—	—
TBBS [c]	—	0,70	0,70
Total	110,50	110,70	144,95

[a] The current IRB shall be used.

[b] 2-Mercaptobenzothiazole.

[c] *N*-tert-butyl-2-benzothiazole-sulfenamide. This shall be in powder form having an initial ether-insoluble or ethanol-insoluble matter content of less than 0,3 % (by mass). The material shall be stored at room temperature in a closed container and the ether-insoluble or ethanol-insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 % (by mass), the material shall be discarded or recrystallized.

6.3 Procedure

6.3.1 Equipment and procedure

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

6.3.2 Mill mixing procedure for Formulae (1) and 2 (gum compounds)

The standard laboratory mill batch mass, in grams, shall be four times the formulation batch mass [i.e. 110,5 g × 4 = 442 g, for Formula (1)]. Maintain the surface temperature of the rolls at 70 °C ± 5 °C and 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.

		Duration (min)
a)	Pass the rubber twice between the rolls without banding, with the mill opening set at 0,2 mm.	—
b)	Band the rubber with the mill opening set at 1,4 mm. When a smooth band has been obtained, adjust the mill opening to 1,8 mm.	4
c)	Add the zinc oxide, the stearic acid, the sulfur and the MBT or TBBS.	4
d)	Make three 3/4 cuts from each side.	3
e)	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
	Total time	**13**

f) Check the mass of the batch (see 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.

g) Cut sufficient material from the batch for curemeter testing and, if required, for determination of the Mooney viscosity of the unvulcanized batch in accordance with ISO 289-1. Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring test pieces.

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.

6.3.3 Mill mixing procedure for Formulae (1) and 2 (gum compounds) using masterbatches

Compounding materials such as accelerators, sulfur or fillers can be incorporated into the rubber as masterbatches. This technique improves the accuracy of compounding-material incorporation and is also more convenient.

The procedure for preparing masterbatches and test mixes for the gum compounds shall be as given in Annex A.

6.3.4 Mixing procedures for Formula (3) (black-filled compound)

6.3.4.1 Mill mixing

6.3.4.1.1 Measurement of rapid plasticity number

Load the rubber on to the mill with the mill opening set at 0,5 mm. Masticate until a smooth band and rolling bank are obtained.

After mastication, determine the rapid plasticity number in accordance with ISO 2007. The rapid plasticity number shall not exceed 45, which is approximately equivalent to a viscosity of 70 Mooney units determined in accordance with ISO 289-1.

6.3.4.1.2 Mixing

The standard laboratory mill batch mass, in grams, shall be two times the formulation batch mass (i.e. 144,95 g × 2 = 289,9 g, for Formula (3). Maintain the surface temperature of the rolls at 70 °C ± 5 °C and a good rolling bank at the nip of the rolls during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings may be necessary.

		Duration (min)
a)	Band the rubber with the mill opening set at 1,4 mm.	1
b)	Add the stearic acid. Make one 3/4 cut from each side.	1
c)	Add the zinc oxide. Make one 3/4 cut from each side.	2
d)	Add the carbon black evenly across the mill at a uniform rate. When about half the black has been incorporated, open the mill to 1,9 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, make one 3/4 cut from each side. Be certain to add the black that has dropped into the mill pan.	10
e)	Add the TBBS and the sulfur. Make three 3/4 cuts from each side.	3
f)	Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch lengthways through the mill six times.	3

	Total time	**20**

g) Check the mass of the batch. If the mass of the batch differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

h) Cut sufficient material from the batch for curemeter testing and, if required, for determination of the Mooney viscosity of the unvulcanized batch in accordance with ISO 289-1. Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring test pieces.

i) 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.

6.3.4.2 Mixing using a laboratory internal mixer (LIM)

6.3.4.2.1 General

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, in grams per cubic centimetre. This means that, if the laboratory internal mixer has a nominal capacity of 750 cm^3, the batch mass for Formula (3) is 750 × 1,103 g (= 827,25 g).

For each batch mixed during the preparation of a series of identical mixes, the laboratory internal mixer conditions shall be the same. 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 setting shall not be altered during the mixing of a series of test batches.

6.3.4.2.2 Single-stage mixing

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 or the head temperature so that this condition is met.

NOTE 1 The mixing conditions given in Table B.5 for each size of laboratory internal mixer can be helpful.

NOTE 2 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 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™ [1)] blender has been found suitable for this method. Caution: if mixed for longer than 3 s, the stearic acid can melt and prevent good dispersion.

An example of a mixing procedure for a laboratory internal mixer is given in a) to i) hereafter.

	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

1) Waring™ 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.

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 3,0 mm mill opening.

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

g) Cut 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 standard temperature and humidity as defined in ISO 23529, before testing.

h) If required, 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. To obtain the effects of mill direction, pass the folded batch four times between mill rolls set at 70 °C ± 5 °C and the appropriate mill opening. Cool on a flat, dry surface.

i) 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.

6.3.4.2.3 Two-stage mixing including mill for final mixing

6.3.4.2.3.1 General

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.

6.3.4.2.3.2 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 or head temperature so that this condition is met.

An example of a mixing procedure for the initial mixing is given in a) to k) hereafter.

		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 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.

h) 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.

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

j) Sheet the batch to an approximate thickness of 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.

k) 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 these cases, the laboratory internal mixer may be used for the final mixing. The head temperature or the batch mass may be adjusted so that the final temperature of the discharged batch does not exceed 120 °C.

6.3.4.2.3.3 Stage 2 — 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 laboratory internal mixer (LIM) is used, adjust, if necessary, the batch mass or the head temperature so that this condition is met.

When mill mixing is used, set the surface temperature of the rolls at 70 °C ± 5 °C and maintain it at this value during mixing. The standard laboratory mill batch mass, in grams, shall be based on three times the formula mass unless otherwise specified in the appropriate rubber evaluation procedure.

An example of an LIM mixing procedure for the final mixing is given in a) to h) hereafter.

		Duration (min)	**Cumulative time** (min)
a)	Close the discharge door, set the rotor and raise the ram.	—	—
b)	Load the rubber, the sulfur and the accelerator. Lower the ram and allow the rubber to be masticated.	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.

e) After discharging the mixed batch, pass it four times through the mill at a roll temperature of 70 °C ± 5 °C and with a mill opening of 0,8 mm.

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

g) 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.

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.

An example of a mill mixing procedure for the final mixing is given in a) to g) as follows.