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Acrylonitrile-butadiene rubber (NBR) — Evaluation procedure

Caoutchouc acrylonitrile-butadiène (NBR) — 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 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 fourth edition cancels and replaces the third edition (ISO 4658:1999), which has been technically revised. It also incorporates the Amendment ISO 4658:1999/Amd.1:2004.

The main changes compared to the previous edition are as follows:

— Clause 2 has been updated;

— the mixing procedure has been specified: method A1 for single stage mixing with LIM is the preferred method; method A2 is for two stages mixing using a LIM for initial mixing and mill mixing for final mixing; method B deals with mill mixing;

— the precision data have been moved to Annex A.

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.

Acrylonitrile-butadiene rubber (NBR) — Evaluation procedure

1 Scope

This document specifies, for acrylonitrile-butadiene rubbers (NBRs):

— physical and chemical tests on raw rubbers;

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

The mixing preferred method is the single stage mixing with LIM (laboratory internal mixer).

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, *Rubber — Determination of ash — Part 1: Combustion method*

ISO 247-2, *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, *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 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 Take a sample of mass approximately 1,5 kg by the method described in ISO 1795.

4.2 Prepare the test portion 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 portion prepared as indicated in 4.2. Record the result as $M_L(1+4)$ at 100 °C.

5.2 Volatile matter

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

5.3 Ash

Determine the ash in accordance with ISO 247-1 or ISO 247-2.

6 Preparation of the test mix for evaluation

6.1 Standard test formulation

The standard test formulation is given in Table 1.

The materials shall be national or international standard reference materials, unless no standard reference materials are available in which case the materials to be used shall be agreed between the interested parties.

Table 1 — Standard test formulation for evaluation on NBRs

Material	Parts by mass
NBR	100,00
Zinc oxide[a]	3,00
Sulfur[b]	1,50
Stearic acid[c]	1,00
Carbon black[d]	40,00
TBBS[e]	0,70
Total	146,20

[a] Class B1a (see ISO 9298:2017, Annex D).

[b] See ISO 8332.

[c] See ISO 8312.

[d] The current industry reference black (IRB), or an equivalent national or international standard reference material, is used.

[e] *N-tert*-Butyl-2-benzothiazole sulfenamide. This is supplied in powder form having an initial insoluble-matter content, determined in accordance with ISO 11235. The material is stored at room temperature in a closed container and the insoluble-matter content is checked every 6 months. If this is found to exceed 0,75 %, the material is discarded or recrystallized.

6.2 Equipment and procedure

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

6.3 Mixing procedures

6.3.1 General

The three following alternative mixing procedures are specified:

— method A1: single stage mixing with LIM (laboratory internal mixer) which is the preferred method;

— method A2: two stage mixing using a LIM for initial mixing and mill mixing for final mixing;

— method B: mill mixing.

6.3.2 LIM mixing for methods A1 and A2

6.3.2.1 General

The mixing technique in each method may be modified to achieve a good dispersion of all the ingredients. The LIM conditions shall be the same during the preparation of a series of identical mixes for each batch mixed. 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 condition shall not be altered during the mixing of a series of test.

6.3.2.2 Method A1 — Single stage mixing with LIM

The final temperature of the batch discharged after mixing shall not exceed 120 °C. If necessary, adjust the batch mass, head temperature or rotors speed, so that this condition is met.

Compounding materials other than rubber, carbon black and oil may 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 may 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 blender has been found suitable for this method.

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

		Duration (min)	Cumulative time (min)
a)	Load the mixing chamber with the rubber strips, lower the ram and start the timer.	0,0	0,0
b)	Masticate the rubber.	1,0	1,0
c)	Raise the ram and add the previously 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
d)	Allow the batch to mix, raising the ram momentarily to sweep down material if necessary.	7,0	9,0
	Total time	9,0	

e) Turn off the motor, raise the ram, open the mixing chamber and discharge the batch. Record the maximum batch temperature indicated, if desired.

f) After discharging the mixed batch, immediately pass it through a laboratory mill set at 50 °C ± 5 °C with a mill opening of 0,8 mm.

g) Pass the rolled batch endwise through the rolls six times.

h) Sheet the batch to approximately 6 mm thickness. Check-weight the batch (see ISO 2393). If 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 for cure 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 after mixing and prior to vulcanizing, if possible, at standard temperature and humidity as defined in ISO 23529.

6.3.2.3 Method A2 — Two stage mixing using a LIM for initial mixing and mill mixing for final mixing

6.3.2.3.1 Stage 1 — Initial mixing procedure with LIM

Mix with the head temperature of the internal mixer maintained at 50 °C ± 5 °C and adjust the rotor speed, if necessary, to maintain the temperature.

a) Load the mixing chamber with the rubber strips, lower the ram and start the timer.

	Duration (min)	Cumulative time (min)
b) Masticate the rubber.	1,0	1,0
c) Raise the ram and add the previously blended zinc oxide, stearic acid and carbon black, taking care to avoid any loss. Lower the ram.	2,0	3,0
d) Raise the ram, clean the orifice and the top of the ram and lower the ram.	0,5	3,5
e) Allow the batch to mix.	1,5	5,0
Total time	5,0	

f) Discharge the batch and record the maximum batch temperature indicated, if desired.

g) Pass the batch once through a mill set at 50 °C ± 5 °C with a mill opening of 1,9 mm. Remove the batch from the mill.

h) Reset the mill opening to 3,0 mm and pass the batch through the mill once. Cut the batch from the mill.

i) Check the batch mass and record. If it differs from the theoretical value by more than +0,5 %/-1,5 %, discard the batch.

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

6.3.2.4 Stage 2 — Final mill mixing procedure

a) Use the total mass of the batch conditioned as specified in 6.3.2.3.1.

b) Set the mill temperature at 50 °C ± 5 °C and the mill opening at 1,9 mm.

	Duration (min)	Cumulative time (min)
c) Band the batch on the mill. Make two 3/4 cuts from each side.	2,0	2,0
d) Add the sulfur and TBBS evenly and slowly across the batch.	0,5	2,5
e) Make three 3/4 cuts from each side.	3,0	5,5
f) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise between the rolls six times.	2,0	7,5
Total time	7,5	

g) Set the mill opening to 3,0 mm and pass the batch through the mill once. Cut the batch from the mill.

h) Check the batch mass and record. If it differs from the theoretical value by more than +0,5 %/–1,5 %, discard the batch.

i) Set the mill temperature at 50 °C ± 5 °C and the mill opening at 1,5 mm.

j) Sheet the batch to approximately 2,0 mm for test sheets.

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

6.3.3 Method B — Mill mixing

The standard laboratory mill batch mass, in grams, shall be based on four times the formulation mass. The surface temperature of the rolls shall be maintained at 50 °C ± 5 °C throughout the mixing.

	Duration (min)	Cumulative time (min)
a) Band the rubber on the roll with the mill set at 50 °C ± 5 °C and 1,4 mm opening.		
b) Add zinc oxide, stearic acid and sulfur.	2,0	2,0
c) Make three 3/4 cuts from each side.	2,0	4,0
d) Add about half of carbon black evenly across the mill.	2,0	6,0
e) Make three 3/4 cuts from each side.	5,0	11,0
f) Add remainder of carbon black evenly across the mill. Be sure to add any material that has fallen into the mill-pan.	2,0	13,0
g) Add the accelerators.	5,0	18,0
h) After accelerators has mixed, make three 3/4 cuts from each side.	1,0	19,0