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Acrylonitrile-butadiene rubber (NBR) — Evaluation procedure

Caoutchouc acrylonitrile-butadiène (NBR) — Méthode d'évaluation

(Revision of third edition of ISO 4658:1999 and of ISO 4658:1999/Amd.1:2004)

ICS 83.060

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 4658 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*.

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This second edition cancels and replaces the first edition (ISO 4658:1999), in which:

— ISO 4658:1999/Amd1:2004 was incorporated in the standard;

— the normative references were updated;

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— the precision data (Clause 8) were moved to an informative Annex A.

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Acrylonitrile-butadiene rubber (NBR) — Evaluation procedure

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies, 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.

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2 Normative references

The following referenced documents are indispensable for the application 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.

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ISO 37, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

ISO 247, *Rubber — Determination of ash*

ISO 248, *Rubbers, raw — Determination of volatile-matter content*

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 2393:2008, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*

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

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

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

3 Sampling and sample preparation

3.1 Take a sample of mass approximately 1,5 kg by the method described in ISO 1795.

3.2 Prepare the test portion in accordance with ISO 1795.

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4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1, on a test portion prepared as indicated in 3.2. Record the result as ML(1+4) at 100 °C.

4.2 Volatile matter

Determine the volatile-matter content preferably by the hot-mill method specified in ISO 248. Certain rubbers tend to stick to the rolls during the hot-mill method; if so, the oven method at 105 °C ± 5 °C may be used.

4.3 Ash

Determine the ash in accordance with ISO 247.

5 Preparation of the test mix for evaluation

5.1 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.

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5.2 Procedure

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5.2.1 Equipment and procedure

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

The compound may be prepared on a mill, in a miniature mixer, or using an internal mixer followed by final mixing on a mill, although slightly different results may be obtained when using one method rather than another.

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Table 1 — Standard test formulation for evaluation of 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 9298:1995, 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, shall be used.

[e] *N*-*tert*-Butyl-2-benzothiazole sulfenamide. This shall be 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 content shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

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5.2.2 Mill mixing procedure

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5.2.2.1 General

The standard laboratory mill batch mass shall be based on four times the recipe mass in grams.

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 hereunder, small adjustments to the mill opening may be necessary.

Two alternative mill mixing procedures are specified.

5.2.2.2 Procedure 1

In this procedure, sulfur coated with magnesium carbonate shall be used and the surface temperature of the rolls shall be maintained at 50 °C ± 5 °C throughout the mixing.

NOTE A standard lot of sulfur coated with 2 % magnesium carbonate, reference M 266573-P, is available from C.P. Hall Co., 4460 Hudson Drive, Stow, Ohio 44224, USA.

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		Duration (min)
a)	Band the rubber with the mill opening set at 1,4 mm	2,0

For hot-polymerized NBR, a period of mastication of up to 4 min may be used.

b)	Add the zinc oxide, stearic acid and sulfur	2,0
c)	Make three 3/4 cuts from each side	2,0
d)	Add half the carbon black evenly across the rubber at a uniform rate	5,0
e)	Make three 3/4 cuts from each side	2,0
f)	Add the remaining carbon black evenly across the rubber at a uniform rate. Sweep up and add any material which has fallen into the pan	5,0
g)	Add the accelerator	1,0
h)	When all the accelerator has been incorporated, make three 3/4 cuts from each side	2,0
i)	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
	Total time	23,0 (max. 25,0)

j) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the batch mass differs from the theoretical value by more than +0,5 %/–1,5 %, discard the batch and re-mix. Remove sufficient material for curemeter testing.

k) Sheet the batch to an approximate thickness of 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens in accordance with ISO 37.

l) 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.

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5.2.2.3 Procedure 2

5.2.2.3.1 General

In this procedure, uncoated sulfur is used. In order to obtain a good dispersion, the sulfur is premixed with the rubber.

5.2.2.3.2 Preparation of the sulfur premix

For this operation, the surface temperature of the rolls shall be maintained at 80 °C ± 5 °C.

		Duration (min)
a)	Band the rubber with the mill opening set at 1,4 mm	2,0

For hot-polymerized NBR, a period of mastication of up to 4 min may be used.

b)	Add the sulfur evenly and slowly across the rubber	3,0
c)	Make three 3/4 cuts from each side	2,0
	Total time	7,0 (max. 9,0)

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d) Cut the batch from the mill and allow it to rest, if possible at standard temperature and humidity as defined in ISO 23529, for 0,5 h to 2,0 h.

5.2.2.3.3 Mixing procedure

The surface temperature of the rolls shall be maintained at 50 °C ± 5 °C throughout the mixing.

		Duration (min)
a)	Band the sulfur premix with the mill opening set at 1,4 mm	2,0
b)	Add the zinc oxide and stearic acid	2,0

Continue in accordance with 5.2.2.2, items c) to l).

5.2.3 Miniature internal mixer (MIM) procedure

5.2.3.1 Mix with the head temperature of the miniature internal mixer maintained at 60 °C ± 3 °C and the rotor speed at 6,3 rad/s to 6,6 rad/s (60 r/min to 63 r/min).

5.2.3.2 Prepare the rubber by passing it through the mill once with the temperature set at 50 °C ± 5 °C and an opening that will give an approximately 5 mm thick sheet. Cut the sheet into strips that are approximately 25 mm wide.

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5.2.3.3 Mixing cycle

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

e) Turn off the rotors, raise the ram, open the mixing chamber and discharge the batch.

f) Immediately pass the batch through a laboratory mill with its opening set at 0,8 mm and at a temperature of 50 °C ± 5 °C.

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

h) Sheet the batch to approximately 6 mm thickness. 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. Remove sufficient material for curemeter testing.

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

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