
International Standard



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Rubber, acrylonitrile-butadiene (NBR) — Test recipe and evaluation of vulcanization characteristics

Caoutchouc acrylonitrile-butadiène (NBR) — Formule d'essai et évaluation des caractéristiques de vulcanisation

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4658 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in June 1978.

It has been approved by the member bodies of the following countries :

Australia	Hungary	<u>ISO 4658-1980</u> Sri Lanka
Austria	India	http://standards.iteh.ai/catalog/standards/sist/b48d3134-dcc6-415b-bdcd-f0b109449 Sweden
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Germany, F. R.	South Africa, Rep. of	

No member body expressed disapproval of the document.

Rubber, acrylonitrile-butadiene (NBR) — Test recipe and evaluation of vulcanization characteristics

1 Scope and field of application

This International Standard specifies the standard materials, equipment and processing methods for evaluating the vulcanization characteristics of acrylonitrile-butadiene rubber (NBR).

2 References

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

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 1796, *Rubber, raw — Sample preparation*¹⁾

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

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

3 Standard test recipe

3.1 Standard test formula

The standard test formula is given in the following table.

The materials used shall be NBS²⁾ Standard reference materials as indicated in the table, or shall be in accordance with equivalent national standards.

Material	NBS Standard reference material number	Parts by mass
NBR	—	100,0
Zinc oxide	370	3,0
Sulphur (see note 1)	—	1,5
Stearic acid	372	1,0
Oil furnace black (HAF)*	378	40,0
<i>N-tert</i> -butyl-2-benzothiazole sulphenamide (TBBS) (see note 2)	384	0,7
Total		146,2

* The current industry reference black may be used in place of NBS 378, but this may give slightly different results.

ISO 4658:1980 NOTES

1 The use of sulphur coated with 2 % magnesium carbonate is preferred. A standard lot of this material, reference M 266573-P is available from C.P. Hall and Co., 4460 Hudson Drive, Stow, Ohio 44224, USA.

2 *N-tert*-butyl-2-benzothiazole sulphenamide. This must be supplied in powder form with an initial ether- or ethanol-insoluble matter content of below 0,3 %. The material must be stored at room temperature in a closed container and the ether- or ethanol-insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 % the material should be discarded or recrystallized.

3.2 Procedure

3.2.1 Equipment and procedure

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

1) At present at the stage of draft. (Revision of ISO 1796-1972.)

2) National Bureau of Standards of the USA.

3.2.2 Mill mixing procedure

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

NOTE — All mill openings should be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

	Duration (min)
3.2.2.1 Band the rubber with the mill opening set at 1,4 mm	2,0
NOTE — For hot polymerized NBR, a period of mastication up to 4 min may be used.	
3.2.2.2 Add the zinc oxide, stearic acid and sulphur . .	2,0
3.2.2.3 Make three 3/4 cuts from each side	2,0
3.2.2.4 Add half the carbon black evenly across the rubber at a uniform rate	5,0
3.2.2.5 Make three 3/4 cuts from each side	2,0
NOTE — Do not cut the band until all visible free black has been incorporated. Be certain to return any material which has dropped into the mill pan to the mix.	
3.2.2.6 Add the remaining carbon black evenly across the rubber at a uniform rate	5,0
3.2.2.7 Add the accelerator	1,0
3.2.2.8 When all the accelerator has been incorporated, make three 3/4 cuts from each side	2,0
3.2.2.9 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

3.2.2.10 Sheet the batch to an approximate thickness of 6 mm and check weigh. Remove sufficient sample for oscillating disc curemeter testing.

3.2.2.11 Immediately sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

3.2.2.12 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing at a standard laboratory temperature (see ISO 471).

4 Evaluation of vulcanization characteristics

4.1 Evaluation according to stress-strain properties

Vulcanize sheets at 150 °C for three periods chosen from a cure series of 20, 30, 40, 50 and 60 min.

NOTES

1 Alternatively, vulcanize the sheets at 145 °C for 25, 35, 50 and 75 min; these conditions will not give the same results as the vulcanizations at 150 °C.

2 The three periods of cure selected should cover undercure, optimum cure and overcure of the polymer under test.

Condition the vulcanized test slab for 16 to 72 h at a standard laboratory temperature (see ISO 471).

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

4.2 Evaluation according to oscillating disc curemeter test

Measure the following standard test parameters :

$$M_L, M_H, t_s, t'_c(50) \text{ and } t'_c(90)$$

in accordance with ISO 3417, using the following test conditions :

oscillation frequency :	1,7 Hz (100 cycles per minute)
amplitude of oscillation :	1° arc
selectivity :	to be chosen to give at least 75 % full scale deflection at M_H
die temperature :	160 °C
pre-heat time :	None

NOTE — If macro dies are used, a pre-heat time of 1 min is necessary.

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