INTERNATIONAL STANDARD

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Rubber, acrylonitrile-butadiene (NBR) — Evaluation procedure

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

Reference number
ISO 4658:1990(E)

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.

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.

International Standard ISO 4658 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 4568:1980), of which it constitutes a technical revision.

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Rubber, acrylonitrile-butadiene (NBR) — Evaluation procedure

1 Scope

This International Standard specifies

- physical and chemical tests on raw rubbers;
- standard materials, a standard test formula, equipment and processing methods for evaluating the vulcanization characteristics of acrylonitrile-butadiene rubbers (NBR).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 247:1990, *Rubber — Determination of ash.*

ISO 248:1979, *Rubbers, raw — Determination of volatile matter content.*

ISO 289:1985, *Rubber, unvulcanized — Determination of Mooney viscosity.*

ISO 471:1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1795:1974, *Raw rubber in bales — Sampling.*

ISO 1796:1982, *Rubber, raw — Sample preparation.*

ISO 2393:1973, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417:1977, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

ISO 6502:1983, *Rubber — Measurement of vulcanization characteristics with rotorless curemeters.*

3 Sampling and sample preparation

3.1 A sample of mass approximately 1 500 g shall be taken by the method described in ISO 1795.

3.2 Preparation of the test portion shall be in accordance with ISO 1796.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289, 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 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 content

Determine the ash content in accordance with ISO 247.

5 Preparation of the test mix for evaluation of NBR

5.1 Standard test formula

The standard test formula is given in table 1.

The materials shall be NIST*) standard reference materials as indicated in table 1, or other, equivalent, national or international standard reference materials.

Table 1 — Standard test formula for evaluation of NBR

Material	NIST standard reference material number	Number of parts by mass
NBR	—	100,00
Zinc oxide	370	3,00
Sulfur[1)]	—	1,50
Stearic acid	372	1,00
Oil furnace black HAF[2)]	—	40,00
TBBS[3)]	384	0,70
Total		146,20

1) A standard lot of sulfur coated with 2 % magnesium carbonate, reference M 266573-P, is available from C.P. Hall and Co., 4460 Hudson Drive, Stow, Ohio 44224, USA. The use of this type of sulfur is mandatory for procedure 1 specified in 5.2.2.1.

In procedure 2 specified in 5.2.2.2, NIST standard reference material N 371 or another equivalent standard reference material shall be used.

2) The current industry reference black, or an equivalent national or international standard reference material, shall be used.

3) *N-tert*-Butyl-2-benzothiazole sulfenamide. This shall be supplied in powder form having an initial ether- or ethanol-insoluble matter content of less than 0,3 %. The material shall 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 shall be discarded or recrystallized.

5.2 Procedure

5.2.1 Equipment and procedure

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

5.2.2 Mill mixing procedures

The standard laboratory mill batch mass, in grams, shall be based on four times the recipe 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 specified hereunder, small adjustments to mill openings may be necessary.

Two alternative mixing procedures are specified:

5.2.2.1 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.

	Duration (min)
a) Band the rubber with the mill opening set at 1,4 mm ..... For hot-polymerized NBR, a period of mastication of up to 4 min may be used.	2,0
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	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 weight differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for curemeter testing.

*) National Institute of Standards and Technology (formerly the National Bureau of Standards) of the USA.

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.

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 471.

5.2.2.2 Procedure 2

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

5.2.2.2.1 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)

d) Cut the batch from the mill and allow it to rest, if possible at standard temperature and humidity as defined in ISO 471, for 0,5 h to 2,0 h.

5.2.2.2.2 Mixing procedure

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

	Duration (min)
a) Band the 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 5.2.2.1, c) to l).

6 Evaluation of vulcanization characteristics by a curemeter test

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 3417 or ISO 6502, 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 % of full scale deflection at M_H
	NOTE 1 With some rubbers, 75 % may not be attainable.
die temperature:	160 °C ± 0,3 °C
pre-heat time:	none

7 Evaluation of tensile stress-strain properties of vulcanized test mixes

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

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

The three periods of cure shall be selected to cover undercure, optimum cure and overcure of the rubber under test.

Condition the vulcanized test slabs for 16 h to 96 h, if possible at standard temperature and humidity as defined in ISO 471.

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

8 Test report

The test report shall include the following:

a) a reference to this International Standard;

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

c) the reference materials used;

d) the method used for the volatile matter content determination (mill or oven);

e) the procedure used to prepare the test mix (procedure 1 or procedure 2);

f) the time used for measuring M_H in clause 6 ;

g) the curemeter test used in clause 7 (ISO 3417 or ISO 6502);

h) the vulcanization temperature and times used in clause 7;

i) any unusual features noted during the determination;

j) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional;

k) the results and the units in which they have been expressed;

l) the date of the test.

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