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AMENDMENT 1
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Acrylonitrile-butadiene rubber (NBR) — Evaluation procedure

AMENDMENT 1

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

AMENDEMENT 1

Reference number
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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.

Amendment 1 to ISO 4658:1999 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*.

Introduction

This Amendment describes an additional procedure for preparing standard test formulations of NBR. The additional procedure uses an internal mixer followed by final mixing on a mill. The Amendment also includes precision data for formulations mixed in this manner.

Acrylonitrile-butadiene rubber (NBR) — Evaluation procedure

AMENDMENT 1

Page 1, Clause 2

Update the normative references as follows:

— Replace ISO 471:1995 by ISO 23529:2004, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods.*

— Insert the year of publication of ISO 1795 (2000) and delete the footnote.

Page 2, Subclause 5.2.1

Replace the second paragraph by the following text:

> 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.

Page 5, Subclause 5.2.3.3

In item j) of the list, replace ISO 471 by ISO 23529.

Page 5

Add a new subclause 5.2.4, as follows:

5.2.4 Procedure using an internal mixer followed by mixing on a mill

5.2.4.1 General

The standard test formulation is given in Table 1 of ISO 4658:1999.

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

If a type A_1, type A_2 or type B internal mixer, as specified in ISO 2393:1994, is used, the standard laboratory mill batch mass shall be seven times the formulation mass. If another type of internal mixer is used, the multiplying factor shall be established by agreement between the interested parties.

5.2.4.2 Mixing in internal mixer

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	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.

5.2.4.3 Batch conditioning

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

5.2.4.4 Final mill mixing procedure

a) Use the total mass of the batch conditioned as specified in 5.2.4.3.

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	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.

5.2.4.5 Batch conditioning

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.

Page 7

Add a new subclause 8.4, as follows:

8.4 Precision for procedure using an internal mixer followed by mixing on a mill

8.4.1 General

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272.

8.4.2 Precision details

A type 2 (interlaboratory) precision was determined for various cure characteristics. One material (NBR rubber) was used in the interlaboratory programme. This was tested in six laboratories on three different days.

8.4.3 Precision results

8.4.3.1 General information

The results of the precision calculations for repeatability and reproducibility are given in Table 3.

The symbols used in Table 3 are defined as follows:

r = repeatability, in measurement units. This is the value below which the absolute difference between two "within-laboratory" test results may be expected to lie, with a specified probability.

(r) = repeatability, in percent (relative).

The test results are obtained with the same method on nominally identical test materials under the same conditions (same operator, apparatus and laboratory) and within a specified time period. Unless stated otherwise, the probability is 95 %.

R = reproducibility, in measurement units. This is the value below which the absolute difference between two "between-laboratory" test results may be expected to lie, with a specified probability.

(R) = reproducibility, in percent (relative).

The test results are obtained with the same method on nominally identical test materials under different conditions (different operators, apparatus and laboratories) and within a specified time period. Unless stated otherwise, the probability is 95 %.

s_r = repeatability standard deviation, in measurement units.

s_R = reproducibility standard deviation, in measurement units.

8.4.3.2 Results

Table 3 — Type 2 precision for various test parameters

Property	Units	Mean of values (Δ)	Within laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
M_L	dN·m	8,34	0,18	0,49	5,92	0,82	2,31	27,7
M_H	dN·m	35,88	0,81	2,25	6,28	1,92	5,36	15,0
t_{s1}	min	3,58	0,11	0,29	8,23	0,39	1,11	30,9
$t'_c(50)$	min	5,19	0,13	0,37	7,05	0,51	1,43	27,6
$t'_c(90)$	min	13,44	0,49	1,38	10,26	1,14	3,20	23,8
NOTE 1 The curemeters used were the oscillating-disc type (test conditions: 160 °C, 1,7 Hz, 1° amplitude arc).								
NOTE 2 The midpoint of the range of values obtained was used for calculations of (r) and (R).								

Page 9, Bibliography

Replace ISO 6472:1994 by ISO 6472:2004 (same title).

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1999-12-01

Acrylonitrile-butadiene rubber (NBR) — Evaluation procedure

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

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Foreword

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International Standard 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.*

This third edition cancels and replaces the second edition (ISO 4658:1990), which has been technically revised.

INTERNATIONAL STANDARD ISO 4658:1999(E)

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.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties.*

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

ISO 248:1991, *Rubbers, raw — Determination of volatile-matter content.*

ISO 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity.*

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing.*

ISO 1795:—[1)], *Rubber, raw, natural and synthetic — Sampling and further preparative procedures.*

ISO 2393:1994, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

ISO 6502:1999, *Rubber — Guide to the use of curemeters.*

1) To be published. (Revision of ISO 1795:1992)

ISO 8312:1999, *Rubber compounding ingredients — Stearic acid — Definition and test methods.*

ISO 8332:1997, *Rubber compounding ingredients — Sulfur — Methods of test.*

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards.*

ISO 9298:1995, *Rubber compounding ingredients — Zinc oxide — Test methods.*

ISO 11235:1999, *Rubber compounding ingredients — Sulfenamide accelerators — 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.

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.

5.2 Procedure

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 either on a mill or in a miniature internal mixer, although slightly different results may be obtained.

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.

5.2.2 Mill mixing procedure

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.

		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 471.

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)

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.

5.2.2.3.3 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.

5.2.3.3 Mixing cycle

		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.

j) 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.

6 Evaluation of vulcanization characteristics by a curemeter test

6.1 Using an oscillating-disc curemeter

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, using the following test conditions:

oscillation frequency: 1,7 Hz (100 cycles per minute)

amplitude of oscillation: 1° of arc

selectivity: to be chosen to give at least 75 % of full-scale deflection

NOTE With some rubbers, 75 % may not be attainable.

die temperature: 160 °C ± 0,3 °C

pre-heat time: none

6.2 Using a rotorless curemeter

Measure the following standard test parameters:

F_L, F_H at defined time, t_{s1}, $t'_c(50)$ and $t'_c(90)$

in accordance with ISO 6502, using the following test conditions:

oscillation frequency: 1,7 Hz (100 cycles per minute)

amplitude of oscillation: 0,5° of arc

selectivity: to be chosen to give at least 75 % of full-scale deflection at F_H

NOTE 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 vulcanization 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 Precision

8.1 General

The precision results have been taken from ASTM D 3187:1990[3]. The repeatability and reproducibility have been calculated in accordance with ISO/TR 9272.

8.2 Precision details

A type 2 (interlaboratory) precision was determined. Three different materials or rubbers were used in the interlaboratory programme. These were tested in seven laboratories on two different days.

8.3 Precision results

The caclulated repeatability and reproducibility values are given in Table 2.

The symbols used in the table are defined as follows:

r = repeatability, in measurement units. This is the value below which the absolute difference between two "within-laboratory" test results may be expected to lie, with a specified probability.

(r) = repeatability, in per cent (relative).

The two test results are obtained with the same method on nominally identical test materials under the same conditions (same operator, apparatus and laboratory) and within a specified time period. Unless stated otherwise, the probability is 95 %.

R = reproducibility, in measurement units. This is the value below which the absolute difference between two "between-laboratory" test results may be expected to lie, with a specified probability.

(R) = reproducibility, in per cent (relative).

The two test results are obtained with the same method on nominally identical test materials under different conditions (different operators, apparatus and laboratories) and within a specified time period. Unless stated otherwise, the probability is 95 %.

Table 2 — Type 2 precision for vulcanization parameters and stress/strain properties of NBR

Property	Units	Range of values (Δ)	Within laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
M_L	dN·m	5,4 to 12,4	0,28	0,79	8,9	0,53	1,50	16,9
M_H	dN·m	36,0 to 46,7	0,85	2,41	5,8	2,14	6,05	14,6
t_{s1}	min	2,8 to 3,9	0,10	0,28	8,2	0,49	1,39	40,9
$t'_c(90)$	min	11,4 to 15,3	0,56	1,58	11,8	1,49	4,22	31,5
300 % modulus	MPa	11,1 to 16,3	0,63	1,78	13,0	1,11	3,14	22,9
Tensile strength	MPa	26,7 to 31,4	0,77	2,18	7,5	1,28	3,62	12,4
Elongation	%	493 to 577	13,5	38,2	7,1	31,8	90,0	16,8
ML(1+4)		54,4 to 104,3	1,30	3,68	4,63	7,8	22,1	27,8

NOTE 1 The curemeters used were the oscillating-disc type (test conditions: 160 °C, 1,7 Hz, 1° amplitude arc).

NOTE 2 The midpoint of the range Δ was used for calculations of (r) and (R).

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

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

c) the procedure used to prepare the standard test formulation (procedure 1, procedure 2 or MIM procedure);

d) the reference materials used;

e) the method used to determine the volatile-matter content (mill or oven);

f) the time used to measure M_H in clause 6;

g) the curemeter method used in clause 6 (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.

Bibliography

[1] ISO 1629:1995, *Rubber and lactices — Nomenclature.*

[2] ISO 6472:1994, *Rubber compounding ingredients — Abbreviations.*

[3] ASTM D 3187:1990, *Standard Test Methods for Rubber — Evaluation of NBR (Acrylonitrile-Butadiene Rubber).*

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