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

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

AMENDEMENT 1

Reference number
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Foreword

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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.

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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).