INTERNATIONAL STANDARD

ISO 7663

Fifth edition
2014-10-01

Halogenated isobutene-isoprene rubber (BIIR and CIIR) — Evaluation procedures

Caoutchoucs isobutène-isoprène halogénés (BIIR et CIIR) — Méthodes d'évaluation

Reference number
ISO 7663:2014(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fifth edition cancels and replaces the fourth edition (ISO 7663:2005), which has been technically revised with the following changes:

— Clause 2 has been updated;

— in 4.2, the method given in ISO 248-2 is now allowed;

— in 5.2.2.1, addition of a statement that the mixing with a laboratory internal mixer is the preferred procedure. Method B becomes "Single stage mixing with a laboratory internal mixer";

— in 5.2.2.3, advice on mixing with various sizes of laboratory internal mixer is given along with a general mixing procedure.

Halogenated isobutene-isoprene rubber (BIIR and CIIR) — Evaluation procedures

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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 the following:

— physical and chemical tests on raw rubbers;

— standardized materials, a standardized test formulation, and the equipment and processing methods for evaluating the vulcanization characteristics of all types of halogenated isobutene-isoprene rubber (BIIR and CIIR).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 247, *Rubber — Determination of ash*

ISO 248-1, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 248-2, *Rubber, raw — Determination of volatile-matter content — Part 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit*

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

ISO 1795:2007, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

ISO 2393, *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 further preparative procedures

3.1 Selection of the sample from the lot shall be in accordance with ISO 1795.

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

3.3 Prepare test samples in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Prepare a test sample, without milling, in accordance with the preferred procedure in ISO 1795.

If milling is deemed necessary, either because of the condition of the laboratory sample (e.g. excessive porosity) or by agreement between the interested parties, it shall be performed in accordance with ISO 1795:2007, 7.3.2.2, paragraphs 1 and 2.

Determine the Mooney viscosity in accordance with ISO 289-1 on a test piece cut from the test sample which shall be as free as possible from air and pockets that may trap air against the rotor and die surface.

The viscosity shall be determined as ML(1+8) at 125 °C.

4.2 Volatile matter

Determine the volatile-matter content by the hot-mill method as specified in ISO 248-1 or by the method specified in ISO 248-2.

4.3 Ash

Determine the ash in accordance with either method A or method B of ISO 247.

5 Preparation of test mixes

5.1 Standard test formulation

The standard test formulation is given in Table 1. The materials shall be national or international standard reference materials. If no standard reference material is available, the materials to be used shall be agreed by the parties concerned.

Table 1 — Standard test formulation for evaluation of halogenated isobutene-isoprene rubbers

Material	Parts by mass
Halogenated isobutene-isoprene rubber (BIIR or CIIR)	100,00
Stearic acid[a, b]	1,00
Industry reference black[c]	40,00
Zinc oxide[a, d]	5,00
Total	146,00

[a] Use powder materials.

[b] The standard reference material for stearic acid is specified in ISO 8312. Use class A.

[c] Use the current industry reference black.

[d] The standard reference material for zinc oxide is specified in ISO 9298. Use the indirect (French) process.

5.2 Procedure

5.2.1 Equipment and procedure

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

5.2.2 Mixing procedure

5.2.2.1 General

Two alternative mixing procedures are specified, but in accordance with ISO 2393 the laboratory internal mixer procedure is preferred:

— method A: mill mixing;

— method B: single stage mixing with a laboratory internal mixer.

NOTE The above procedures may not give identical results.

5.2.2.2 Method A (mill mixing procedure)

The standard laboratory mill batch mass, in grams, shall be based on four times the formulation mass, i.e. 4 × 146,00 g (= 584 g). The surface temperatures of the rolls shall be maintained at 40 °C ± 5 °C throughout the mixing.

The vulcanization of halogenated isobutene-isoprene rubber with zinc oxide is highly sensitive to moisture. Therefore, care shall be taken when conditioning the carbon black.

Condition the carbon black for 1 h at 125 °C ± 3 °C. The thickness of the carbon black layer shall not exceed 10 mm. Store the conditioned black in a moisture-proof container.

Maintain a good rolling bank at the nip of the rolls during mixing. If this is not obtained with the nip settings specified below, small adjustments to the mill openings can be necessary.

Mix the stearic acid and the carbon black together in a suitable container before starting to mix.

	Duration (min)	**Cumulative time** (min)
a) Band the rubber on the slow roll with the mill opening set at 0,65 mm	1,0	1,0
b) Add the mixture of stearic acid and carbon black evenly across the mill at a uniform rate. Return any materials that drop through the mill to the batch.	9,5	10,5
c) When all the mixture of stearic acid and carbon black has been incorporated, make one 3/4 cut from each side. Do not cut the band until all visible free black has been incorporated.	0,5	11,0
d) Add the zinc oxide.	3,0	14,0
e) When all the zinc oxide has been incorporated, make three 3/4 cuts from each side, alternately.	2,0	16,0
f) Cut the batch from the mill. Set the mill opening at 0,8 mm and pass the rolled batch endwise through the mill six times.	2,0	18,0

g) Sheet the batch to a thickness of approximately 6 mm. 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.

h) Sheet the batch to a thickness of approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37.

i) Condition the batch for 2 h to 24 h prior to vulcanizing and curemeter testing at standard laboratory temperature and humidity as defined in ISO 23529.

5.2.2.3 Method B (single stage mixing with a laboratory internal mixer procedure)

For laboratory internal mixers having nominal capacities of 65 cm^3 to about 2 000 cm^3, the batch mass shall be equal to the nominal mixer capacity, in cubic centimetres, multiplied by the density of the compound. For each batch mixed, the laboratory internal mixer conditions shall be the same during the preparation of a series of identical mixes. At the beginning of each series of test mixes, a machine-conditioning batch shall be mixed using the same formulation as the mixes under test. The laboratory internal mixer shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next. The temperature control conditions shall not be altered during the mixing of a series of test batches.

The mixing technique shall be such as to obtain a good dispersion of all the ingredients.

Condition the carbon black as described in 5.2.2.2.

NOTE A general mixing procedure for the laboratory internal mixer is as follows:

	Duration (min)	**Cumulative time** (min)
a) Charge the stearic acid, zinc oxide, and carbon black first, followed by 3/4 of the rubber, lower the ram and start the timer.	0,0	0,0
b) Allow the batch to mix, raise the ram to sweep down, if necessary. Add the rest of the rubber.	1,5	1,5
c) Allow the batch to mix.	3,5	5,0

d) Turn off the rotor, raise the ram, open the mixing chamber, and discharge the batch. Record the maximum batch temperature.

The final temperature of the batch discharged after 5 min shall not exceed 120 °C. If necessary, adjust the batch mass or the head temperature so that this condition is achieved.

e) Immediately pass the batch twice through a mill set at 40 °C ± 5 °C with a mill opening of 3,0 mm or compress the batch between two stainless-steel plates with a force of 100 kN for 5 s at 30 °C ± 5 °C.

f) 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 remix.

g) Condition the batch for 2 h to 24 h at standard laboratory temperature and humidity as defined in ISO 23529. Cut a test piece for curemeter testing in accordance with either 6.1 or 6.2.

h) If required, sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ring test pieces in accordance with ISO 37. Condition the batch in accordance with g) above.

For a laboratory internal mixer having a nominal capacity of 65 cm^3 a batch mass corresponding to 0,48 times the formulation mass, i.e. 0,48 × 146,00 (= 70,08 g) has been found to be suitable.

Prepare the rubber by passing it once through a mill with the temperature set at 50 °C ± 5 °C and an opening of 0,5 mm. Cut the sheet into 20 mm wide strips.

Mix with the head temperature maintained at 60 °C ± 3 °C and the unloaded rotor speed at 6,3 rad/s to 6,6 rad/s (60 rpm to 63 rpm).

For a laboratory internal mixer having a nominal capacity of 1170 cm^3 ± 40 cm^3, a batch mass corresponding to (8,5 × 156,75 g = 1 332 g) has been found to be suitable.

The speed of the fast rotor shall be set at 7 rad/s to 8 rad/s (67 rpm to 87 rpm).

6 Evaluation of vulcanization characteristics by a curemeter test

6.1 Using an oscillating-disc curemeter

Measure the following standardized test parameters:

M_L, M_H, 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° arc

NOTE An amplitude of oscillation of 3° arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1}.

— Selectivity: to be chosen to give at least 75 % of full-scale deflection at M_H

— Die temperature: 160 °C ± 0,3 °C

— Pre-heat time: none

6.2 Using a rotorless curemeter

Measure the following standardized test parameters:

F_L, F_{HR}, 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° arc

NOTE An amplitude of oscillation of 1° arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1}.

— Selectivity: to be chosen to give at least 75 % of full-scale deflection at F_{HR}

— Die temperature: 160 °C ± 0,3 °C

— Pre-heat time: none

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

Vulcanize test sheets at 150 °C for 15 min, 30 min and 45 min, respectively.

Condition the vulcanized sheets for at least 16 h and up to 96 h at standard laboratory temperature and at standard humidity, as defined in ISO 23529.

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

NOTE Method B (the miniature internal mill mixing procedure) provides sufficient compounded material for the evaluation of vulcanization characteristics by a curemeter test and the evaluation of stress-strain properties on one vulcanized sheet. The recommended vulcanization time is 45 min at 150 °C, but other values may be appropriate.

8 Precision

See Annex A.

9 Test report

The test report shall include the following:

a) a reference to this International Standard (i.e. ISO 7663:2014);

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

c) the method used for the ash determination (method A or method B of ISO 247);

d) the method used for the determination of the volatile-matter content; (ISO 248-1, mill or oven or ISO 248-2);

e) the reference materials used;

f) the ambient conditions in the laboratory during preparation of the test mix;

g) the mixing procedure used in 5.2.2;

h) the size (nominal mixer capacity) of the mixer used for method B;

i) for Clause 6:

— the type of curemeter used and the reference standard;

— the amplitude of oscillation used;

— the values obtained for the standardized test parameters measured in 6.1 or 6.2;

j) the vulcanization periods used in Clause 7;

k) any unusual features noted during the determination;

l) any operation not included in this International Standard or in the International Standards to which reference is made, including non-conformity with ISO 23529, as well as any operation regarded as optional;

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

n) the date of the test.

Annex A
(informative)

Precision

A.1 General

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

NOTE The precision results are based on data from ASTM D 3958:1995.

A.2 Precision details

A type 2 interlaboratory precision was determined. Two different materials (BIIR and CIIR rubbers) were used in the interlaboratory programme; these were tested in five laboratories on two different days.

A.3 Precision results

The results of the precision calculations for repeatability and reproducibility are given in Table A.1.

The symbols used in Table A.1 are defined as follows:

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

(r) is the 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 is the reproducibility, in measurement units. This is the value below which the absolute difference between two "between-laboratory" test results can be expected to lie with a specified probability;

(R) is the reproducibility, in percent (relative).

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

s_r is the repeatability standard deviation, in measurement units;

s_R is the reproducibility standard deviation, in measurement units.