INTERNATIONAL STANDARD

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

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

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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.

ISO 7663 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 fourth edition cancels and replaces the third edition (ISO 7663:1995), which has been technically revised.

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

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:

— 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 referenced documents are indispensable for the application of this document. 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 247:1990, *Rubber — Determination of ash*

ISO 248:2005, *Rubber, raw — Determination of volatile-matter content*

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

ISO 1795:2000, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

ISO 2393:1994, *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 8312, *Rubber compounding ingredients — Stearic acid — Definition and test methods*

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

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

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Sampling and further preparative procedures

A laboratory sample of approximately 1,5 kg shall be taken in accordance with the method described in ISO 1795. Preparation of the test samples shall be 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 1795:2000, Subclause 8.3.2.2, 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 hot-mill method A as specified in ISO 248:2005, Clause 4.

A 450 g sized test unit may be used if required by an agreement between the interested parties.

4.3 Ash

Determine the ash in accordance with one of the methods specified in 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 (or as agreed by the interested parties).

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 2393:1994, Clauses 6, 7, 8 and 9.

5.2.2 Mixing procedure

5.2.2.1 General

Two permitted mixing procedures are specified:

— method A: mill mixing;

— method B: miniature internal mixer (MIM) mixing.

Additionally, method A and method B may be used with different mixing procedures, by agreement between the interested parties.

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 may 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.	0,5	11,0
	Do not cut the band until all visible free black has been incorporated.		
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 (miniature internal mixer procedure)

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 for a miniature internal mixer having a nominal capacity of 64 cm^3.

Condition the carbon black as described in 5.2.2.2.

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 of the miniature internal mixer 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).

		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, remove 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.

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.