INTERNATIONAL STANDARD

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Rubber, halogenated isobutene-isoprene (BIIR and CIIR) — Evaluation procedure

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

Reference number
ISO 7663:1994(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 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 second edition cancels and replaces the first edition (ISO 7663:1985), which has been technically revised.

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Rubber, halogenated isobutene-isoprene (BIIR and CIIR) — Evaluation procedure

1 Scope

This International Standard specifies

— physical and chemical tests on raw rubbers;

— standard materials, standard test formulation, equipment and processing methods for evaluating the vulcanization characteristics of halogenated isobutene-isoprene rubbers (BIIR and CIIR).

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 37:1994, *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:—[1], *Rubber — Times, temperatures and humidities for conditioning and testing.*

ISO 1795:1992, *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 6502:1991, *Rubber — Measurement of vulcanization characteristics with rotorless curemeters.*

3 Sampling and further preparative procedures

Take a laboratory sample of approximately 1,5 kg in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

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

Determine the Mooney viscosity in accordance with ISO 289-1, using a running time of 8 min. Express the viscosity as ML (1 + 8) at 125 °C.

4.2 Volatile matter

Determine the volatile-matter content by the oven method specified in ISO 248.

1) To be published. (Revision of ISO 471:1983 and ISO 1826:1981)

4.3 Ash

Determine the ash in accordance with method B specified in ISO 247:1990.

5 Preparation of test mixes for evaluation of halogenated isobutene-isoprene rubbers

5.1 Standard test formulation

The standard test formulation is given in table 1.

The materials shall be national or international standard reference materials.

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

Material	Parts by mass
Halogenated isobutene-isoprene rubber (BIIR or CIIR)	100,0
Zinc oxide	5,0
Stearic acid	1,0
Industry reference black[1)]	40,0
	146,0
1) Use the current IRB.	

5.2 Procedure

5.2.1 Equipment and procedure

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

5.2.2 Mill mixing procedure

The standard laboratory mill batch mass, in grams, shall be based on twice the formulation mass. Maintain the surface temperature of the rolls at 40 °C ± 5 °C throughout the mixing.

Due to the high sensitivity of halogenated isobutene-isoprene rubber to moisture on vulcanization, care shall be taken when conditioning carbon black and during storage to minimize water absorption by placing it in closed containers.

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

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

	Duration (min)	Cumulative time (min)
a) Band the rubber on the slower 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. Be certain to add any mixture that has dropped into the mill pan.	9,5	10,5
c) When all of 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 of the zinc oxide has been incorporated, make three 3/4 cuts from each side.	2,0	16,0
f) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise through the rolls six times.	2,0	18,0

g) Sheet the batch to an approximate thickness of 6 mm and determine the mass of the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for curemeter testing.

h) Sheet the batch to approximately 2,2 mm for preparing test slabs 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, 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

Measure the following standard test parameters:

M_L, M_H at defined time, t_{s1} (or t_{s2}), $t'_c(50)$ 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° of arc An amplitude of oscillation of 3° of arc is permitted as an alternative
selectivity:	to be chosen to give at least 75 % of full-scale deflection at M_H With some polymers, 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 15 min, 30 min and 45 min.

Condition the vulcanized sheets for 16 h to 96 h, at a standard temperature, and, if possible, at a standard humidity, 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 temperature used for the Mooney viscosity determination;

d) the reference materials used;

e) the conditioning conditions used in 5.2.2 i) and clause 7;

f) in clause 6:

— the reference standard,

— the time for M_H and

— the amplitude of oscillation

used for the curemeter test;

g) any unusual features noted during the determination;

h) 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;

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

j) the date of the test.

ICS 83.060.00

Descriptors: rubber, crude rubber, synthetic rubber, isobutene-isoprene rubber, tests, chemical tests, physical tests, vulcanizing tests, estimation, characteristics.

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