INTERNATIONAL STANDARD

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Isobutene-isoprene rubber (IIR) — Evaluation procedures

Caoutchouc isobutène-isoprène (IIR) — Méthode d'évaluation

Reference number
ISO 2302:1995(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 2302 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 2302:1985), which has been technically revised.

Annex A of this International Standard is for information only.

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Isobutene-isoprene rubber (IIR) — Evaluation procedures

1 Scope

This International Standard specifies

— physical and chemical tests on raw rubbers;

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

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 471:1995, *Rubber — Temperatures, humidities and times 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

A laboratory sample of approximately 1,5 kg shall be taken by the method described in 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 in ISO 1795.

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

Because the Mooney viscosity of high-molecular-mass isobutene-isoprene rubbers does not vary linearly with their molecular mass, it is necessary to use different test temperatures for high- and low-Mooney rubbers. For low-Mooney rubbers (i.e. those not exceeding 60 under the conditions given here), the viscosity shall be determined as ML (1 + 8) at 100 °C.

For high-Mooney rubbers, 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 or by the oven method as specified in ISO 248.

4.3 Ash

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

5 Preparation of test mixes for evaluation of 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 (or as agreed by the interested parties).

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

Material	Parts by mass
Isobutene-isoprene rubber (IIR)	100,0
Stearic acid	1,00
Current industry reference black	50,00
Zinc oxide	3,00
Sulfur	1,75
TMTD[1)]	1,00
Total	156,75

1) Tetramethylthiuram disulfide.

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.

Three alternative mixing procedures are specified:

Method A — Mill mixing

Method B — Internal mixer mixing

Method C — Miniature internal mixer mixing.

Method B is presented as informative annex A since insufficient experience has been gained with this method to include it as part of the standard.

NOTE 1 These procedures may not give identical results.

5.2.2 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 × 156,75 g = 627 g). The surface temperature of the rolls shall be maintained at 45 °C ± 5 °C throughout the mixing.

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 openings may be necessary.

A mill batch mass based on two times the formulation mass may also be used, but, in this case, more adjustments to the mill openings will be necessary.

	Duration (min)
a) Band the rubber with the mill opening set at 0,65 mm.	1,0
b) Mix the carbon black and the stearic acid and add evenly across the rolls at a uniform rate. Increase the mill opening at intervals to maintain a constant rolling bank. When all the black has been incorporated, make one 3/4 cut from each side. Be certain to add all the black that has dropped into the mill pan.	10,0
c) Add the zinc oxide, the sulfur and the TMTD.	3,0
d) Make three 3/4 cuts from each side.	3,0
e) 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
Total time	19,0

f) Sheet the batch to an approximate thickness of 6 mm and check-weigh 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.

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

h) Condition the batch for 2 h to 24 h, after mixing and prior to vulcanizing, if possible at standard laboratory temperature and humidity as defined in ISO 471.

5.2.3 Method C — Miniature internal mixer procedure

For a miniature internal mixer having a nominal mixing capacity of 64 cm^3, a batch mass corresponding to 0,46 times the formulation mass (i.e. 0,46 × 156,75 g = 72,10 g) has been found to be suitable.

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).

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 strips that are 25 mm wide.

NOTE 2 Compounding materials other than rubber, e.g. carbon black, may be added to miniature internal mixer batches more precisely and with greater ease if they are previously blended together in proportion to the mass required by the formulation. Such blends may be made using one of the following:

a) A mortar and pestle.

b) A biconical mixer. Agitate for 10 min with the intensifier bar turning.

c) A blender. Mix for five periods of 3 s each, scraping the inside of the blender to dislodge materials stuck to the sides after each 3 s period. (A "Waring"-type blender has been found suitable for this method.) CAUTION — If mixed longer than 3 s at a time, the stearic acid may melt, thus preventing good dispersion.

	Duration (min)	Cumulative time (min)
a) Load the rubber, lower the ram and allow the rubber to be masticated.	1,0	1,0
b) Raise the ram and add the zinc oxide, sulfur, stearic acid and TMTD, taking care to avoid any loss. Then add the carbon black, sweep the orifice and lower the ram.	1,0	2,0
c) Allow the batch to mix.	3,0	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) Pass the batch through a mill set at 50 °C ± 5 °C twice at a 3,0 mm mill opening.

f) Check the batch mass (see ISO 2393) and record. If it differs from the theoretical value by more than 0,5 %, discard the batch and remix.

g) Cut a test piece for testing vulcanization characteristics in accordance with ISO 3417 or ISO 6502, if required. Condition the test piece for 2 h to 24 h at 23 °C ± 3 °C before testing.

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. To obtain the effects of mill direction, pass the folded batch four times between mill rolls set at 50 °C ± 5 °C and at the appropriate mill opening. Cool on a flat, dry surface.

i) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard laboratory temperature and humidity as defined in ISO 471.

6 Evaluation of vulcanization characteristics

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° arc 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 (torsion shear) curemeter

Measure the following standard test parameters:

F_L, F_{max} at defined time, t_{s1}, $t'_{0,50}$ and $t'_{0,90}$

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 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_{max}.
die temperature:	160 °C ± 0,3 °C
pre-heat time:	None

NOTE 3 The two types of curemeter may not give identical results.

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

Vulcanize sheets at 150 °C for 20 min, 40 min and 80 min.

Condition the vulcanized sheets for 16 h to 96 h, at a standard laboratory temperature, and, if possible, at a standard laboratory humidity defined in ISO 471.

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

NOTE 4 Method C provides sufficient compounded material for evaluation of vulcanization characteristics by a curemeter test and the evaluation of stress-strain properties on one vulcanized sheet. The recommended vulcanization time is 40 min at 150 °C, but other values may be appropriate.

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 method used for the volatile-matter-content determination (mill or oven);

e) the method used for the ash determination (method A or B of ISO 247:1990);

f) the reference materials used;

g) the mixing procedure used;

h) the mill batch factor used in 5.2.2;

i) the conditioning environment used in 5.2.2 h), 5.2.3 i), clause 7 and A.1 i);

j) in clause 6:

— the type of curemeter used and the reference standard,

— the time for M_H or F_{max} and

— the amplitude of oscillation used for the curemeter test;

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, 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)

Method B — Internal mixer for initial and mill for final mixing

For an internal mixer of Type A1 (see ISO 2393) having a nominal capacity of 1 170 cm^3 ± 40 cm^3, a batch mass corresponding to 8,5 times the formulation mass (i.e. 8,5 × 156,75 g = 1332 g) has been found to be suitable.

The speed of the fast rotor shall be set at 7 rad/s to 9 rad/s (67 rpm to 87 rpm).

The final temperature of the batch discharged after 5 min mixing time shall be between 150 °C and 170 °C. If necessary, adjust the batch mass to achieve the mixing conditions specified.

During final mixing, a good rolling bank at the nip of the rolls shall be maintained. If this is not attained with the nip settings specified, small adjustments to the mill openings may be necessary.

A.1 Stage 1 — Initial mixing procedure

	Duration (min)	Cumulative time (min)
a) Adjust the temperature of the internal mixer to a starting temperature of 50 °C. Close the discharge door, start the rotors and raise the ram.		
b) Load the rubber, lower the ram and allow the rubber to be masticated.	0,5	0,5
c) Raise the ram, load the zinc oxide, stearic acid and carbon black, and lower the ram.	0,5	1,0
d) Allow the batch to mix.	2,0	3,0
e) Raise the ram, clean the mixer throat and the top of the ram, and lower the ram.	0,5	3,5
f) Allow the batch to mix.	1,5	5,0
g) Discharge the batch.		
Total time	5,0	

h) Immediately check the temperature of the batch with a suitable measuring device. If the temperature as measured falls outside the range 150 °C to 170 °C, discard the batch. Pass the batch three times through a mill with a mill opening of 2,5 mm and a temperature of 50 °C ± 5 °C. Sheet the batch to an approximate thickness of 10 mm and check-weigh the batch (see ISO 2393). If the mass differs from the theoretical value by more than 0,5 %, discard the batch and re-mix.

i) Leave the batch for at least 30 min and up to 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 471.

A.2 Stage 2 — Final mill mixing procedure

a) The standard laboratory-mill batch mass, in grams, shall be based on three times the formula mass (462 g masterbatch).

b) Set the mill temperature at 50 °C ± 5 °C and the mill opening to 1,5 mm.

	Duration (min)	Cumulative time (min)
c) Band the masterbatch on the slow roll.	1,0	1,0
d) Add the sulfur and the TMTD. Do not cut the band until the sulfur and accelerator are completely dispersed.	1,5	2,5
e) Make three 3/4 cuts from each side, allowing 15 s between each cut.	2,5	5,0

f) Cut the batch from the mill. Set the mill opening at 0,8 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternatively. 2,0 7,0

Total time 7,0

g) Sheet the batch to an approximate thickness of 6 mm and check-weigh 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 sheets or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37. Check the batch mass and record. If it differs from the theoretical value by more than 0,5 %, discard the batch and re-mix.

i) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard laboratory temperature and humidity as defined in ISO 471.