INTERNATIONAL STANDARD

ISO 2302

Sixth edition
2014-10-15

Isobutene-isoprene rubber (IIR) — Evaluation procedure

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

Reference number
ISO 2302:2014(E)

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Published in Switzerland

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Foreword

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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).

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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 sixth edition cancels and replaces the fifth edition (ISO 2302:2005), which has been technically revised with the following changes:

- Clause 2 was updated.
- In 4.2, the method given in ISO 248-2 is now allowed.
- In 5.2.2.1, the addition of a statement that the mixing procedure with a laboratory internal mixer is the preferred procedure. Method B becomes "single stage mixing with a laboratory internal mixer".
- In 5.2.2.2, the alternative procedure using a batch mass of two times the formulation mass has been deleted.
- In 5.2.2.3, advice on mixing with various sizes of laboratory internal mixer is given along with a general mixing procedure.
- In Annex A, the precision data from ASTM D3188-95 have been used.

Isobutene-isoprene rubber (IIR) — Evaluation procedure

WARNING — Users of this International Standard should be familiar with the 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

- physical and chemical tests on raw rubbers and
- standardized materials, a standardized test formulation, equipment, and processing methods for evaluating the vulcanization characteristics of all types of isobutene-isoprene rubber (IIR).

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 in accordance with the preferred procedure in ISO 1795, i.e. without milling, cutting the test sample directly from the laboratory sample. The test sample shall be as free as possible from air and pockets that may trap air against the rotor and die surface.

If agreed between the interested parties or if the condition of the test sample (e.g. excessive porosity) makes milling necessary, 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 this test portion, 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 248-1 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 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. If no standard reference material is available, the materials to be used shall be agreed by the parties concerned.

Table 1 — Standard test formulation

Material	Parts by mass
Isobutene-isoprene rubber (IIR)	100,00
Stearic acid[a]	1,00
Industry reference black[b]	50,00
Zinc oxide[a]	3,00
Sulfur[a]	1,75
Tetramethylthiuram disulfide(TMTD)[a]	1,00
Total	156,75

[a] Powder materials shall be used (standard curing ingredients used in the industry).

[b] The current industry reference black shall be used.

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 procedures

5.2.2.1 General

Three 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 using a laboratory internal mixer (the preferred procedure);

— method C: two-stage mixing using a laboratory internal mixer for initial mixing and a mill for final mixing.

NOTE These procedures might not give identical results.

5.2.2.2 Method A — Mixing with a laboratory mill

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.

		Duration (min)	**Cumulative time** (min)
a)	Band the rubber with the mill opening set at 0,65 mm.	1,0	1,0
b)	Mix the carbon black and the stearic acid, and add evenly across the mill rolls at a uniform rate. Increase the mill opening at intervals to maintain a constant rolling bank. When all the carbon black has been incorporated, make a 3/4 cut from each side. Do not cut the batch while free carbon black is evident in the bank or on the milling surface. Be certain to return to the batch any materials that drop through the mill.	10,0	11,0
c)	Add the zinc oxide, the sulfur, and the TMTD.	3,0	14,0
d)	Make three alternating 3/4 cuts from each side.	2,0	16,0
e)	Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise through the mill six times.	2,0	18,0

f) Sheet the batch to approximately 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

g) 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 or dumbbell test pieces in accordance with ISO 37.

i) After mixing, condition the batch for at least 2 h but not more than 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 23529.

5.2.2.3 Method B — Single stage mixing using a laboratory internal mixer

For laboratory internal mixers having a 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.

The temperature of the batch discharged on completion of mixing shall not exceed 120 °C. If necessary, adjust the batch mass or the mixer head starting temperature so that this condition is met.

In the following procedure, compounding materials other than rubber and carbon black may be added to the batch more precisely and with greater ease if they are previously blended together in the proportions required by the formulation. Such blends may be made using one of the following:

— a mortar and pestle;

— a double-cone mixer (mix for 10 min with the intensifier bar turning);

— a blender (mix for five periods of 3 s each, scraping the inside of the blender to dislodge material stuck to the sides after each 3 s period) (a “Waring”-type blender has been found suitable for this method).

CAUTION — If the mixing periods are longer than 3 s, the stearic acid may melt, thus preventing good dispersion.

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

	Duration (min)	**Cumulative time** (min)
a) Load the rubber, lower the ram, and allow the rubber to be masticated.	0,5	0,5
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 down the mixer throat and lower the ram.	1,0	1,5
c) Allow the batch to mix.	2,0	3,5
d) Raise the ram, clean the mixer throat and the top of the ram, and lower the ram.	0,5	4,0
e) Allow the batch to mix.	1,0	5,0

f) Discharge the batch and immediately check the temperature with a suitable measuring device.

g) The final temperature of the discharged batch after the cumulative mixing time of 5 min shall not exceed 120 °C. If it does, discard the batch and repeat the procedure with a different batch mass or starting temperature.

h) Pass the batch through a mill set at 50 °C ± 5 °C twice with a 3,0 mm mill opening.

i) Check the batch mass (see ISO 2393) and record. If it differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

j) Cut out a test piece for determining the 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.

k) If required, sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring or dumbbell 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.

l) After mixing and prior to vulcanizing, condition the batch for at least 2 h but not more than 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 23529.

For a miniature internal mixer having a nominal mixing capacity of 65 cm^3, a batch mass corresponding to 0,47 times the formulation mass (i.e. 0,47 × 156,75 = 73,67 g) has been found to be suitable.

Mix with the head temperature of the miniature internal mixer maintained at 60 °C ± 3 °C and a starting (unloaded) rotor speed of 6,3 rad/s to 6,6 rad/s (60 rev/min to 63 rev/min).

Prepare the rubber by passing it once through a laboratory mill with the roll temperature set at 50 °C ± 5 °C and an opening of 0,5 mm. Cut the sheet thus produced into strips 25 mm wide.

For an 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 rev/min to 87 rev/min).

5.2.2.4 Method C — Two-stage mixing using a laboratory internal mixer for initial mixing and a mill for final mixing

5.2.2.4.1 General

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

The temperature of the batch discharged on completion of mixing shall be between 150 °C and 170 °C. If necessary, adjust the batch mass or the mixer head starting temperature so that this condition is met.

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

A general mixing procedure for the laboratory internal mixer is given in 5.2.2.4.2 and 5.2.2.4.3.

5.2.2.4.2 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.

h) Immediately check the temperature of the batch with a suitable measuring device. If the measured temperature is outside the range 150 °C to 170 °C, discard the batch and repeat the procedure with a different batch mass.

i) Pass the batch three times through a mill opening of 2,5 mm at 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 $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

j) Condition the batch for at least 30 min but not more than 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 23529.

5.2.2.4.3 Stage 2 — Final mill mixing procedure

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

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

	Duration (min)	**Cumulative time** (min)
a) Band the master-batch on the slow roll.	1,0	1,0
b) Add the sulfur and the TMTD. Do not cut the band until the sulfur and accelerator are completely dispersed.	1,5	2,5
c) Make three alternating 3/4 cuts from each side, allowing 15 s between each cut.	2,5	5,0
d) 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 alternately.	2,0	7,0

e) 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 $^{+0,5}_{-1,5}$ %, discard the batch and re-mix. Remove sufficient material for curemeter testing.

f) Sheet the batch to approximately 2,2 mm in order to prepare test sheets or to the appropriate thickness in order to prepare ISO ring or dumbbell test pieces in accordance with ISO 37. Check the batch mass and record it. If it differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

g) After mixing and prior to vulcanization, condition the batch for at least 2 h but not more than 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 23529.

6 Evaluation of vulcanization characteristics by a curemeter test

6.1 Using 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° of arc An amplitude of oscillation of 3° of 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 rotorless curemeter

Measure the following standard test parameters:

F_L, F_{max} at defined time, 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° of arc An amplitude of oscillation of 1° of 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

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 as defined in ISO 23529.

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

NOTE Method B (MIM method) 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 Precision data

See Annex A.

9 Test report

The test report shall include the following:

a) a reference to this International Standard, i.e. ISO 2302;

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

c) the method used for the volatile-matter determination (ISO 248-1 or ISO 248-2);

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

e) the reference materials used to prepare the test mix;

f) the mixing procedure used in 5.2.2;

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

h) for Clause 6:

  - the type of curemeter used;
  - the defined time at which M_H was measured;
  - the amplitude of oscillation used for the curemeter test;

i) any unusual features noted during the determination;

j) details of any operation not included in this International Standard or in the International Standards to which reference is made, as well as of any operation regarded as optional;

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

l) the date of the test.