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Isobutene-isoprene rubber (IIR) — Evaluation procedures

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

Reference number
ISO 2302:2005(E)

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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 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 fifth edition cancels and replaces the fourth edition (ISO 2302:1995), which has been technically revised.

The precision data in this edition have been taken, with permission of ASTM, from ASTM D 3188-95, *Standard Test Methods for Rubber — Evaluation of IIR (Isobutene-Isoprene Rubber)*, copyright ASTM.

In this corrected version of ISO 2302:2005, the reference in the Bibliography to ASTM D 3188-95 has been transferred to Clause 1.

Isobutene-isoprene rubber (IIR) — 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, equipment and processing methods for evaluating the vulcanization characteristics of all types of isobutene-isoprene rubber (IIR).

The precision data in this International Standard have been taken, with permission of ASTM, from ASTM D 3188-95, *Standard Test Methods for Rubber — Evaluation of IIR (Isobutene-Isoprene Rubber)*, copyright ASTM.

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, *Rubbers, 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 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 sample shall be in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Prepare a test portion in accordance with the preferred procedure in ISO 1795, i.e. without milling, cutting the test portion directly from the laboratory sample. The test portion 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 sample (e.g. excessive porosity) makes milling necessary, 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 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.

4.3 Ash

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

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

5.2.2 Mixing procedures

5.2.2.1 General

Three alternative mixing procedures are specified:

- Method A: mixing with a laboratory mill;
- Method B: mixing with a miniature internal mixer (MIM);
- Method C: mixing with an internal mixer.

NOTE These procedures may 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.

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)	**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 — Mixing with a miniature internal mixer (MIM)

For a miniature internal mixer having a nominal mixing capacity of 64 cm^3, a batch mass corresponding to 0,47 times the formulation mass (i.e. $0,47 \times 156,75 = 73,67$ 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 rpm to 63 rpm).

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.

In the following procedure, compounding materials other than rubber, e.g. 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.

		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 mouth of the mixing chamber 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 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 head temperature.

e) Pass the batch through a mill set at 50 °C ± 5 °C twice with 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 $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

g) 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.

h) 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.

i) 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.

5.2.2.4 Method C — Internal mixer for initial and mill for final mixing

5.2.2.4.1 General

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 × 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).

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.

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

NOTE 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 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

8.1 General

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

NOTE The precision calculations are based on data from ASTM D 3188:1995.

8.2 Precision details

A type 2, class III interlaboratory precision was determined. Three different materials (IIR rubbers) were used in the interlaboratory programme. These were tested in four laboratories on three different days.

8.3 Precision results

The calculated repeatability and reproducibility values are given in Table 2.

The symbols used in Table 2 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 may be expected to lie with a specified probability.
(r)	is the repeatability in percent (relative).

The three test results were 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 may be expected to lie with a specified probability.
(R)	is the reproducibility in percent (relative).

The three test results were 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 %.

Table 2 — Precision for various test parameters

Property	**Units**	**Midpoint values** [a]	**Within laboratory**			**Between laboratories**		
			s_r	r	(r)	s_R	R	(R)
M_L	dN·m	14,95	0,22	0,61	4,33	0,51	1,45	10,18
M_H	dN·m	70,95	1,07	3,04	4,27	2,51	7,11	9,98
t_{s2}	min	2,50	0,11	0,32	14,37	0,21	0,58	26,53
$t'_c(50)$	min	9,40	0,13	0,36	3,90	0,29	0,81	8,69
$t'_c(90)$	min	26,05	0,95	2,70	10,21	1,41	3,98	15,07

s_r is the repeatability standard deviation, in measurement units;

s_R is the reproducibility standard deviation, in measurement units.

[a] Measured at 160 °C, 1,7 Hz, 3° of arc; midpoint of range used for (r) and (R) calculations.

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

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

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