

International Standard



2302

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber, isobutene-isoprene (IIR) — Evaluation procedures

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

Third edition — 1985-05-15

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CDU 678.5/.8 : 547.315.2

Ref. No. ISO 2302-1985 (E)

Descriptors : rubber, synthetic rubber, isobutene-isoprene, tests, vulcanizing tests.

Price based on 3 pages

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2302 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition, ISO 2302:1978, of which it constitutes a minor revision.

Rubber, isobutene-isoprene (IIR) — Evaluation procedures

1 Scope and field of application

This International Standard specifies

- a) physical and chemical tests on raw polymer;
- b) standard materials, equipment and processing methods for evaluating vulcanization characteristics of all types of isobutene-isoprene rubbers (IIR).

2 References

ISO 37, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 247, *Rubber — Determination of ash.*

ISO 248, *Rubbers, raw — Determination of volatile matter content.*

ISO 289, *Rubber, unvulcanized — Determination of Mooney viscosity.*¹⁾

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 1796, *Raw rubber — Sample preparation.*

ISO 2058, *Raw styrene-butadiene rubber (SBR) — Determination of volatile matter.*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417, *Rubber — Measurement of curing characteristics with the oscillating disc curemeter.*

3 Sampling and sample preparation

3.1 A sample of mass approximately 1 500 g shall be taken by the method specified in ISO 1795.

3.2 Sample preparation, if required, shall be in accordance with ISO 1796.

NOTE — No sample preparation is required for most types of isobutene-isoprene.

4 Physical and chemical tests on raw polymer

4.1 Mooney viscosity

Determine the viscosity on a portion from the original sample according to ISO 289.

Because the shearing disc viscosity of high molecular mass isobutene-isoprene rubber is non-linear, it is necessary to use different test temperatures for high and for low Mooney polymers. For low Mooney polymers (i.e. not exceeding 60 under these prescribed conditions), the viscosity shall be determined as ML 1 + 8 at 100 °C. For high Mooney polymers, the viscosity shall be determined as ML 1 + 8 at 125 °C.

4.2 Volatile matter

Determine the volatile matter by the hot-mill method as specified in ISO 2058 or by the oven method as specified in ISO 248.

4.3 Ash

Determine the ash in accordance with ISO 247.

1) At present at the stage of draft. (Revision of ISO/R 289-1963.)

5 Test recipe for evaluation of vulcanization characteristics

Duration (min)

5.1 Standard test formula

The standard test formula is given in the following table.

The materials shall be NBS¹⁾ Standard reference materials as indicated in the table, or shall be in accordance with equivalent national standards.

Material	NBS Standard reference material number	Parts by mass
Isobutene-isoprene rubber (IIR)	—	100,00
Stearic acid	372	1,00
Oil furnace black (HAF)*	378	50,00
Zinc oxide	370	3,00
Sulfur	371	1,75
TMTD**	374	1,00
		Total 156,75

* The current Industry Reference Black may be used in place of NBS 378, but this may give slightly different results.
** Tetramethylthiuram disulfide.

5.2 Procedure

5.2.1 Equipment and procedure

Equipment and procedure for the 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 four times the formula mass (i.e. 4 × 156,75 g = 627 g). The surface temperature of the rolls shall be maintained at 45 ± 5 °C throughout the mixing.

NOTE — All mill openings shall be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

5.2.2.1 Band the rubber with the mill opening set at 0,65 mm	1
5.2.2.2 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
5.2.2.3 Add the zinc oxide, the sulfur and the TMTD	3
5.2.2.4 Make three 3/4 cuts from each side	3
5.2.2.5 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
Total time	19

- 5.2.2.6 Sheet the batch to an approximate thickness of 6 mm and check weigh the batch.
- 5.2.2.7 Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.
- 5.2.2.8 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing.

6 Evaluation of vulcanization characteristics according to stress-strain properties

- Vulcanize sheets at 150 °C for 20, 40 and 80 min.
- Condition the vulcanized test slab for 16 to 72 h.
- Measure the stress-strain properties in accordance with ISO 37.

1) National Bureau of Standards of the USA.

7 Evaluation of vulcanization characteristics according to oscillating disc curemeter test

Measure the following standard 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° of arc is permitted as an alternative.
selectivity :	to be chosen to give at least 75 % full scale deflection at M_H
die temperature :	160 °C
pre-heat time :	none

8 Test recipe for evaluation of rate of cure and scorch testing

8.1 Standard test formula

The standard test formula is given in the following table.

The materials shall be NBS¹⁾ Standard reference materials as indicated in the table, or shall be in accordance with equivalent national standards.

Material	NBS Standard reference material number	Parts by mass
Isobutene-isoprene rubber (IIR)	—	100,00
Zinc oxide	370	2,00
Sulfur	371	2,00
TMTD*	374	0,60
Total		104,60

* Tetramethylthiuram disulfide.

8.2 Procedure

8.2.1 Equipment and procedure

Equipment and procedure for the preparation, mixing and vulcanization shall be in accordance with ISO 2393.

8.2.2 Mill mixing procedure

The standard laboratory mill batch mass, in grams, shall be based on twice the formula mass (i.e. $2 \times 104,60 \text{ g} = 209,20 \text{ g}$). The surface temperature of the rolls shall be maintained at $32,5 \pm 2,5 \text{ °C}$ throughout the mixing.

NOTE — All mill openings shall be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

	Duration (min)
8.2.2.1 Band the rubber with the mill opening set at 0,65 mm. Add the zinc oxide as soon as the rubber has banded	4
8.2.2.2 Make one 3/4 cut from each side	3
8.2.2.3 Add the sulfur and the TMTD	2
8.2.2.4 Make three 3/4 cuts from each side	2
8.2.2.5 Cut the batch from the mill and pass the rolled batch endwise through the rolls six times	2
Total time	14
8.2.2.6 Sheet the batch to an approximate thickness of 6 mm and check weigh the batch.	

NOTE — If the determination is not made within 1 h of mixing, the batch shall be kept wrapped in two layers of aluminium foil until tested. The test shall be made within 24 h of mixing.

1) National Bureau of Standards of the USA.

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