INTERNATIONAL STANDARD

ISO 2303

Fourth edition
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Isoprene rubber (IR) — Non-oil-extended, solution-polymerized types — Evaluation procedure

Caoutchouc isoprène (IR) — Types polymérisés en solution et non étendus à l'huile — Méthode d'évaluation

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2303 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 2303:1990), which has been technically revised.

Annex A of this International Standard is for information only.

Isoprene rubber (IR) — Non-oil-extended, solution-polymerized types — Evaluation procedure

WARNING — Persons using this International Standard should be familiar with normal laboratory practices. 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, for general purpose non-oil-extended, solution-polymerized polyisoprene rubbers (IR):

— physical and chemical tests on raw rubbers;

— standard materials, standard test formulation, equipment and processing methods for evaluating the vulcanization characteristics.

Two mill-mixing procedures are described in this International Standard and an internal mixer-mill mixing procedure is described in annex A.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC 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 — Determination of Mooney viscosity*

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing*

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 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter*

ISO 6472:—[1], *Rubber compounding ingredients — Abbreviations*

ISO 6502:1999, *Rubber — Guide to the use of curemeters*

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards*

ISO 11235:1999, *Rubber compounding ingredients — Sulfenamide accelerators — Test methods*

3 Sampling and sample preparation

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

3.2 Preparation of the test portion shall be in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

The Mooney viscosity shall be determined in accordance with ISO 289-1 on a test portion prepared as described in ISO 1795 (without massing).

The result shall be recorded as ML(1 + 4) at 100 °C.

4.2 Volatile matter

The volatile matter content shall be determined in accordance with ISO 248.

4.3 Ash

The ash content shall be determined in accordance with ISO 247.

5 Preparation of the test mixes for evaluation of 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 concerned parties.

1) To be published. (Revision of ISO 6472:1994)

Table 1 — Standard test formulation for evaluation of IR rubbers

Material	Parts by mass
Isoprene rubber (IR)	100,00
Stearic acid	2,00
Zinc oxide	5,00
Sulfur	2,25
Industry Reference Black (N330)	35,00
TBBS [a]	0,70
Total	144,95

[a] TBBS or N-*tert*-butyl-2-benzothiazole sulfenamide in accordance with ISO 6472. This shall be supplied in powder form having an initial insoluble matter content, in accordance with ISO 11235, of less than 0,3 %. The material shall be stored at room temperature in a closed container and insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded. TBBS may be purified by reprocessing, e.g. by recrystallization; the procedure for this is beyond the scope of this International Standard.

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 procedures

5.2.2.1 General

Two mill mixing procedures are specified, A and B. The mixing time is shorter in method B than in method A.

The two methods do not necessarily give identical results. In laboratory cross checks or in a series of evaluations the same procedure shall be used in all cases.

In both methods the standard laboratory mill batch mass, in grams, shall be based on four times the formula mass. The surface temperature of the rolls shall be maintained at 70 °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 in 5.2.2.2 and 5.2.2.3, small adjustments to the mill openings may be necessary.

5.2.2.2 Procedure A

		Duration (min)	Cumulative time (min)
a)	Pass the rubber between the mill-rolls twice without banding, with the mill opening set at 0,5 mm for approximately 2 min and weigh the rubber.	2	2
b)	Band the rubber with the mill opening set at 1,4 mm and make two 3/4 cuts from each side.	2	4

NOTE Some types of isoprene rubber go to the back roll, in which case the stearic acid should be added and after its incorporation the rubber can usually be transferred to the front roll. In addition, certain tougher types of isoprene rubber may require slightly longer breakdown before the addition of other materials in order to obtain a good rolling bank.

c)	Set the mill opening to 1,7 mm and add the stearic acid. Make one 3/4 cut from each side.	2	6
d)	Add the zinc oxide and the sulfur. Make two 3/4 cuts from each side.	3	9
e)	Add the carbon black evenly across the mill at a uniform rate. When approximately half the black has been incorporated, open the mill to 1,9 mm and make one 3/4 cut from each side, then add the remainder of the carbon black. Be certain to add the black that has dropped into the mill pan. When all the black has been incorporated, make one 3/4 cut from each side.	13	22
f)	Add the TBBS with the mill opening still at 1,9 mm. Make three 3/4 cuts from each side.	3	25
g)	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.	3	28
Total time			**28**

h) 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.

i) 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.

j) 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.

5.2.2.3 Procedure B

		Duration (min)	Cumulative time (min)
a)	Pass the rubber between the rolls twice without banding, with the mill opening set at 0,5 mm ± 0,1 mm, then band the rubber between the rolls with the mill opening gradually increased to 1,4 mm.	2	2
b)	Add the stearic acid. Make one 3/4 cut from each side.	2	4
c)	Add the sulfur and the zinc oxide. Make two 3/4 cuts from each side.	3	7
d)	Add half of the carbon black. Make two 3/4 cuts from each side.	3	10
e)	Add the remaining half of the carbon black and the black that has dropped into the mill pan. Make three 3/4 cuts from each side.	5	15
f)	Add the TBBS. Make three 3/4 cuts from each side.	3	18
g)	Cut the batch from the mill. Set the mill opening to 0,5 mm ± 0,1 mm and pass the rolled batch endwise through the rolls six times.	2	20
Total time			**20**

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) Cut the batch from the mill 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.

j) 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

Warning — Formation of nitrosamines is possible during the cure.

6.1 Using an oscillating disc curemeter

Measure the following standard test parameters:

M_L, M_H, t_{s1}, $t'_c(50)$ and $t'_c(90)$ (see ISO 3417) using the following test conditions:

— oscillation frequency: 1,7 Hz (100 cycles per minute)

— amplitude of oscillation: 1° arc

— selectivity at M_H: to be chosen to give at least 75 % of full scale deflection

NOTE With some rubbers, 75 % may not be attainable.

— die temperature: 160 °C ± 0,3 °C

— pre-heat time: none

6.2 Using a rotorless curemeter

Measure the following standard test parameters:

F_L, F_{max} at defined time, $t'_c(50)$ 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° arc

— selectivity: to be chosen to give at least 75 % of full scale deflection at F_{max}.

NOTE With some rubbers, 75 % may not be attainable.

— die temperature: 160 °C ± 0,3 °C

— pre-heat time: none