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

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 2303:2011](https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-a54a671b8039/iso-2303-2011)

[https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-
a54a671b8039/iso-2303-2011](https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-a54a671b8039/iso-2303-2011)



iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 2303:2011

<https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-a54a671b8039/iso-2303-2011>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2011

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Sampling and sample preparation	2
4 Physical and chemical tests on raw rubber	2
4.1 Mooney viscosity	2
4.2 Volatile matter	2
4.3 Ash	2
5 Preparation of the test mixes for evaluation of isoprene rubbers	2
5.1 Standard test formulation	2
5.2 Procedure	3
5.2.1 Equipment and procedure	3
5.2.2 Mill mixing procedures	3
5.2.3 Laboratory internal mixer (LIM) mixing procedure	4
6 Evaluation of vulcanization characteristics by a curemeter test	8
6.1 Using an oscillating-disc curemeter	8
6.2 Using a rotorless curemeter	8
7 Evaluation of tensile stress-strain properties of vulcanized test mixes	8
8 Precision	9
9 Test report	9
Annex A (informative) Precision data for both mill mixer and laboratory internal mixer	10
Annex B (informative) Additional precision data for natural rubber	12
Bibliography	14

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 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 fifth edition cancels and replaces the fourth edition (ISO 2303:2003), in which the following technical changes have been made:

- a laboratory internal mixer (LIM) mixing procedure has been added as Subclause 5.2.3;
- old Annex A, which gave an alternative mixing method using an internal mixer-mill mixing procedure, has been deleted;
- the existing precision data have been moved from Clause 8 to a new Annex A;
- precision data for the LIM mixing procedure have been added as Table A.2;
- precision data for natural rubber, obtained using mill mixing and LIM mixing, have been taken from ISO 1658:2009 and added as Annex B.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 2303:2011](https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-a54a671b8039/iso-2303-2011)

<https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-a54a671b8039/iso-2303-2011>

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

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

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 247:2006, *Rubber — Determination of ash*

ISO 248-1, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 289-1, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

ISO 1795, *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 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 248-1.

4.3 Ash

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

iTeh STANDARD PREVIEW

5 Preparation of the test mixes (for evaluation of isoprene rubbers)

5.1 Standard test formulation

ISO 2303:2011

[https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-](https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-14a671b8039/iso-2303-2011)

The standard test formulation is given in Table 1. [14a671b8039/iso-2303-2011](https://standards.iteh.ai/catalog/standards/sist/a95db07c-4bf9-4da1-b948-14a671b8039/iso-2303-2011)

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 interested parties.

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-butylbenzothiazole-2-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: methods 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 $70\text{ °C} \pm 5\text{ °C}$ 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 might be necessary.

5.2.2.2 Method 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.	2,0	2,0
b) Band the rubber with the mill opening set at 1,4 mm and make two 3/4 cuts from each side.	2,0	4,0
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 might 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.	2,0	6,0
d) Add the zinc oxide and the sulfur. Make two 3/4 cuts from each side.	3,0	9,0
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 any black that has dropped into the mill pan. When all the black has been incorporated, make one 3/4 cut from each side.	13,0	22,0
f) Add the TBBS with the mill opening still at 1,9 mm. Make three 3/4 cuts from each side.	3,0	25,0
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.	3,0	28,0
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 $\begin{matrix} +0,5\% \\ -1,5\% \end{matrix}$, discard the batch and re-mix.		

ISO 2303:2011(E)

- i) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 3417 or ISO 6502. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.
- j) 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.
- k) Condition the batch for 2 h to 24 h prior to vulcanizing, if possible at a standard temperature and humidity as defined in ISO 23529.

5.2.2.3 Method 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.	2,0	2,0
b) Add the stearic acid. Make one 3/4 cut from each side.	2,0	4,0
c) Add the sulfur and the zinc oxide. Make two 3/4 cuts from each side.	3,0	7,0
d) Add half of the carbon black. Make two 3/4 cuts from each side.	3,0	10,0
e) Add the remaining half of the carbon black and any black that has dropped into the mill pan. Make three 3/4 cuts from each side.	5,0	15,0
f) Add the TBBS. Make three 3/4 cuts from each side.	3,0	18,0
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.	2,0	20,0
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 $\begin{matrix} +0,5 \\ -1,5 \end{matrix}$ %, discard the batch and re-mix.		
i) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 3417 or ISO 6502. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.		
j) 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.		
k) Condition the batch for 2 h to 24 h prior to vulcanizing, if possible at a standard temperature and humidity as defined in ISO 23529.		

5.2.3 Laboratory internal mixer (LIM) mixing procedure

5.2.3.1 General

For a LIM having a nominal mixing capacity of 65 cm³ to about 2 000 cm³, the batch mass shall be equal to the nominal mixer capacity, in cubic centimetres, multiplied by the compound density. The LIM conditions shall be the same for each batch mixed 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 LIM shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next. Temperature control settings shall not be altered during the mixing of a series of test batches.

5.2.3.2 Single-stage mixing procedure

The mixing technique shall be such as to obtain a good dispersion of all the ingredients. The final temperature of the batch discharged after mixing shall not exceed 120 °C. If necessary, adjust the batch mass, head temperature and/or rotor speed so that this condition is met.

NOTE 1 Compounding materials other than rubber, carbon black and oil can be added to LIM batches more precisely and with greater ease if they are previously blended together in the proportions required by the formulation. Such blends can be made using a mortar and pestle, by mixing for 10 min in a biconical blender with the intensifier bar turning, or by mixing in a blender for five 3 s periods and scraping the inside of the blender to dislodge materials stuck to the sides after each 3 s mix. A Waring blender¹⁾ has been found suitable for this method. Caution: if mixed longer than 3 s, the stearic acid might melt and prevent good dispersion.

NOTE 2 An example of a mixing procedure for a LIM is as follows:

	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 pre-blended zinc oxide, sulfur, stearic acid and TBBS, 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.	7,0	9,0
d) Turn off the motor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature.		
e) After discharging the mixed batch, pass it through a mill set at 70 °C ± 5 °C once at a 0,5 mm mill opening, and then twice at a 0,3 mm mill opening.		
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 $\pm 1,5\%$, discard the batch and re-mix.		
g) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 3417 or ISO 6502. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.		
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) Condition the batch for 2 h to 24 h prior to vulcanizing, if possible at a standard temperature and humidity as defined in ISO 23529.		

5.2.3.3 Two-stage mixing including mill for final mixing procedure

5.2.3.3.1 General

The LIM shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next.

5.2.3.3.2 Stage 1 — Initial mixing stage

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

1) This is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

ISO 2303:2011(E)

The final temperature of the batch discharged after mixing shall be between 150 °C and 170 °C. If necessary, adjust the batch mass, head temperature and/or rotor speed so that this condition is met.

NOTE An example of a mixing procedure for the initial mixing is as follows:

	Duration (min)	Cumulative time (min)
a) Adjust the temperature of the LIM to a starting temperature of 60 °C ± 3 °C. Close the discharge door, set the rotor speed to 77 rpm, start the rotors and raise the ram.	—	—
b) Load half of the rubber, all the carbon black, zinc oxide and stearic acid, then the remaining half of the rubber. Lower the ram.	0,5	0,5
c) Allow the batch to mix.	3,0	3,5
d) Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0,5	4,0
e) Allow the batch to mix.	0,5	4,5
f) Discharge the batch.	1,5	6,0
g) After discharging the batch, 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.		
h) Pass the batch three times through a mill with a mill opening of 2,5 mm and a temperature of 70 °C ± 5 °C.		
i) Sheet the batch to an approximate thickness of 10 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $\begin{matrix} +0,5 \\ -1,5 \end{matrix}$ %, discard the batch and re-mix.		
j) Leave the batch for at least 30 min and up to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529.		

The smaller LIMs do not provide enough compound for the final mill mixing, as a batch mass of three times the formula mass is required. In these cases, the LIM may be used for the final mixing. The head temperature and/or the batch mass may be adjusted so that the final temperature of the discharged batch does not exceed 120 °C.

5.2.3.3.3 Stage 2 — Final mixing stage

Rest the batch for at least 30 min, or until it reaches room temperature, before proceeding with the final mixing stage. The mixing technique shall be such as to obtain a good dispersion of all the ingredients. The final temperature of the batch discharged after mixing shall not exceed 120 °C.

When a LIM is used, adjust, if necessary, the batch mass, the head temperature and/or the rotor speed so that this condition is met. When mill mixing is used, set the surface temperature of the rolls to 70 °C ± 5 °C and maintain it at this temperature during mixing. The standard laboratory mill batch mass, in grams, shall be based on two times the formula mass. A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings given hereunder, small adjustments to the mill openings may be necessary.

NOTE 1 An example of a LIM mixing procedure for the final mixing stage is as follows:

	Duration (min)	Cumulative time (min)
a) Close the discharge door, set the rotor speed and raise the ram.	—	—
b) Load the rubber, the sulfur and the accelerator, and lower the ram.	0,5	0,5

- | | | |
|---|-----|-----|
| c) Allow the batch to mix. | 1,5 | 2,0 |
| d) Raise the ram, open the mixing chamber and discharge the batch. Record the maximum batch temperature. | 0,5 | 2,5 |
| e) After discharging the mixed batch, pass it four times through a mill at a roll temperature of $70\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ and with a mill opening of 0,8 mm. | 0,5 | 3,0 |
| 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 $\begin{matrix} +0,5 \\ -1,5 \end{matrix}$ %, discard the batch and re-mix. | | |
| g) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 3417 or ISO 6502. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing. | | |
| 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) Condition the batch for 2 h to 24 h prior to vulcanizing, if possible at a standard temperature and humidity as defined in ISO 23529. | | |

NOTE 2 An example of a mill mixing procedure for the final mixing is as follows:

	Duration (min)	Cumulative time (min)
a) Set the mill temperature at $70\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ and the mill opening to 1,9 mm. Band the masterbatch on the slow roll.	—	—
b) Add the accelerators. Do not cut the band until the accelerators are completely dispersed. Then make three 3/4 cuts from each side.	3,0	3,0
c) Add the sulfur. Do not cut the band until the sulfur is completely dispersed. Then make three 3/4 cuts from each side.	3,0	6,0
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	8,0
e) Set the mill opening at approximately 6 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternately. Sheet the batch.	1,0	9,0
f) Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $\begin{matrix} +0,5 \\ -1,5 \end{matrix}$ %, discard the batch and re-mix.		
g) Remove sufficient material from the batch for evaluating the vulcanization characteristics in accordance with ISO 3417 or ISO 6502. Condition this material for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529, before testing.		
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) Condition the batch for 2 h to 24 h prior to vulcanizing, if possible at a standard temperature and humidity as defined in ISO 23529.		