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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

Reference number
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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.

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.

International Standard ISO 2303 was prepared by Technical Committee ISO/TC 45, *Rubber*.

This third edition cancels and replaces the second edition (ISO 2303 : 1983), of which it constitutes a minor revision.

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

1 Scope

This International Standard specifies

— physical and chemical tests on raw rubbers;

— standard materials, a standard test formula, equipment and processing methods for evaluating the vulcanization characteristics of non-oil-extended, solution-polymerized isoprene rubbers (IR).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37 : 1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 247 : 1978, *Rubber — Determination of ash.*

ISO 248 : 1979, *Rubbers, raw — Determination of volatile matter content.*

ISO 289 : 1985, *Rubber, unvulcanized — Determination of Mooney viscosity.*

ISO 471 : 1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1795 : 1974, *Raw rubber in bales — Sampling.*

ISO 1796 : 1982, *Rubber, raw — Sample preparation.*

ISO 2393 : 1973, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417 : 1977, *Rubber — Measurement of vulcanization 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 described in ISO 1795.

3.2 Preparation of the test portion shall be in accordance with ISO 1796.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine, without preliminary massing, the Mooney viscosity in accordance with ISO 289 on a test portion prepared as indicated in ISO 1796, avoiding areas that contain many bubbles. Record the result as ML (1 + 4) at 100 °C.

4.2 Volatile matter

Determine the volatile matter content in accordance with ISO 248.

4.3 Ash content

Determine the ash content in accordance with ISO 247.

5 Preparation of the test mix for evaluation of isoprene rubbers

5.1 Standard test formula

The standard test formula is given in table 1.

The materials shall be NIST[1] standard reference materials as indicated in table 1, or other, equivalent, national or international standard reference materials.

1) National Institute of Standards and Technology (formerly the National Bureau of Standards) of the USA.

Table 1 — Standard test formula for evaluation of IR rubbers

Material	NIST standard reference material number	Parts by mass
Isoprene rubber (IR)	—	100,00
Stearic acid	372	2,00
Zinc oxide	370	5,00
Sulfur	371	2,25
Oil furnace black (HAF)*)	378	35,00
TBBS**)	384	0,70
Total		144,95

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

**) *N-tert*-butyl-2-benzothiazole-sulfenamide. This shall be supplied in powder form having an initial ether- or ethanol-insoluble-matter content of less than 0,3 %. The material shall be stored at room temperature in a closed container and the ether- or ethanol-insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

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. 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 hereunder, small adjustments to the mill openings may be necessary.

	Duration (min)
5.2.2.1 Pass the rubber between the rolls twice without banding, with the mill opening set at 0,5 mm	2,0
5.2.2.2 Band the rubber with the mill opening set at 1,4 mm and make two 3/4 cuts from each side	2,0

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 may require slightly longer breakdown before the addition of other materials in order to obtain a good rolling bank.

5.2.2.3 Set the mill opening to 1,7 mm and add the stearic acid. Make one 3/4 cut from each side	2,0
5.2.2.4 Add the zinc oxide and the sulfur. Make one 3/4 cut from each side	3,0
5.2.2.5 Add the carbon black evenly across the mill at a uniform rate. When about 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	13,0
5.2.2.6 Add the TBBS with the mill opening still at 1,9 mm. Make three 3/4 cuts from each side	2,0
5.2.2.7 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
Total time	27,0

5.2.2.8 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 %, discard the batch and re-mix. Remove sufficient material for oscillating disc curemeter testing.

5.2.2.9 Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

5.2.2.10 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 with the oscillating disc curemeter

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
selectivity:	to be chosen to give at least 75 % full scale deflection at M_H
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 135 °C for three periods selected from a cure series of 20 min, 30 min, 40 min and 60 min.

The three periods of cure shall be chosen to cover the undercure, optimum cure and overcure of the material under test.