BS 5474 : 1977
ISO 2303 – 1975
UDC 678.762.3 : 678.01

British Standard

Test recipe and evaluation of vulcanization characteristics for raw non oil-extended isoprene rubbers

[ISO title: Rubber, isoprene (IR) – Non oil-extended, solution polymerized types – Test recipe and evaluation of vulcanization characteristics]

Formule d'essai et évaluation des caractéristiques de vulcanisation des caoutchoucs polyisoprènes bruts et non étendus à l'huile

Prüfformel und Bewertung der Vulkanisierungseigenschaften für rohe Isoprenkautschuke ohne Ölstreckung

Foreword

This British Standard has been prepared under the authority of the Rubber Standards Committee. It is identical with ISO 2303–1975 'Rubber, isoprene (IR)–Non oil-extended, solution polymerized types–Test recipe and evaluation of vulcanization characteristics' and incorporates Amendment 1 published 15 August 1975 to that standard (but see 'Textual error' below).

Terminology and conventions. The text of the international standard has been accepted as suitable for publication, without deviation*, as a British Standard. Certain terminology and conventions are used, however, that are not identical with those used in British Standards. Attention is therefore drawn to the following.

Wherever the words 'International Standard', relating to this standard, appear, they should be interpreted as 'British Standard'.

The comma has been used throughout as a decimal marker. In British Standards it is current practice to use a full point (full stop on the baseline) as the decimal marker.

Cross references. For each of the following references to other international standards that are given in the text, there is an equivalent British Standard; these are as listed below.

Reference to international standard	Equivalent British Standard
ISO/R 37–1968 Determination of tensile stress-strain properties of vulcanized rubbers	BS 903 Methods of testing vulcanized rubber Part A2 : 1971 Determination of tensile stress-strain properties
ISO 1795–1974 Raw rubber in bales–Sampling *and* ISO 1796–1972 Raw rubber–Sample preparation	BS 1673 Methods for testing raw rubber and unvulcanized compounded rubber Part 1 : 1976 Sampling and further preparative procedures
ISO 2393–1973 Rubber test mixes–Preparation, mixing and vulcanization–Equipment and procedures	BS 1674 : 1976 Equipment and general procedure for mixing and vulcanizing rubber test mixes
ISO 3417–1977 Raw rubber–Measurement of curing characteristics with the oscillating disk curemeter	BS 1673 : Part 10† Measurement of prevulcanizing and curing characteristics by means of curemeters

Compounding ingredients complying with the requirements of BS 4398 : 1969 'Compounding ingredients for rubber test mixes' are suitable to replace the NBS Standard reference materials referred to in **3.1.**

Additional information. Ring specimens (see **3.2.2.9**) are not included in the British Standard for determining stress-strain properties (BS 903 : Part A2) because the dumb-bell type specimen is used almost exclusively in the UK.

It is customary in the UK to condition the rubber (see **3.2.2.10** and clause **4**) at 23 °C, which is one of the preferred temperatures given in ISO 2393.

Textual error. Attention is drawn to a printing error in the second footnote to the table in **3.1** (as amended by Amendment 1), where '*N-Tert*-butyl sulphenamide' should be printed as '*N-Tert*-butyl-2-benzothiazole sulphenamide'. When an amendment has been issued to the international standard to correct this error, this standard will be likewise amended; users are advised in the meantime to identify the error in their copy by a suitable mark in the text.

*The page numbering of the international standard has been altered to allow for a 4-page presentation of this standard.
†In course of preparation.

British Standards Institution

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1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies standard materials, equipment and processing methods for evaluating vulcanization characteristics of non oil-extended, solution-polymerized isoprene rubbers (IR).

2 REFERENCES

ISO/R 37, *Determination of tensile stress-strain properties of vulcanized rubbers.*

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

ISO 1796, *Raw rubber — Sample preparation.*

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

ISO 3417, *Raw rubber — Measurement of curing characteristics with the oscillating disk curemeter.*[1)]

3 TEST RECIPE FOR EVALUATION OF VULCANIZATION CHARACTERISTICS

3.1 Standard test formula

The standard test formula is given in the following table.

The materials shall be NBS[2)] 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
Isoprene rubber (IR)	—	100,00
Stearic acid	372	2,00
Zinc oxide	370	5,00
Sulphur	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 NBS 378, but this may give slightly different results.

** *N-Tert-butyl sulphenamide. 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.

3.2 Procedure

3.2.1 *Equipment and procedure*

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

3.2.2 *Mill mixing procedure*

The standard laboratory mill batch mass, in grams, shall be based on four times the formula mass. The surface

1) At present at the stage of draft.

2) National Bureau of Standards of the U.S.A.

temperature of the rolls shall be maintained at 70 ± 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.

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

NOTE — Some types of isoprene rubber go to the back roll, in which case the stearic acid shall 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.

3.2.2.3 Set the mill opening to 1,7 mm and add the stearic acid. Make one 3/4 cut from each side	2
3.2.2.4 Add the zinc oxide and the sulphur. Make one 3/4 cut from each side	3
3.2.2.5 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
3.2.2.6 Add the TBBS with the mill opening still at 1,9 mm. Make three 3/4 cuts from each side . .	2
3.2.2.7 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
Total time	27

3.2.2.8 Sheet the batch to an approximate thickness of 6 mm and check weigh.

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

3.2.2.10 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing.

4 EVALUATION OF VULCANIZATION CHARACTERISTICS ACCORDING TO STRESS-STRAIN PROPERTIES

Vulcanize sheets at 135 °C for three periods selected from a cure series of 20, 30, 40 and 60 min.

Condition the vulcanized test slab for 16 to 72 h.

Measure the stress-strain properties in accordance with ISO/R 37.

NOTE — The three periods of cure selected shall cover the undercure, optimum cure and overcure of the polymer under test.

Curemeter testing in accordance with ISO 3417 may be considered as an alternative method for measuring the vulcanization characteristics.

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Contract requirements
Attention is drawn to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

Revision of British Standards
British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. **It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.**

The following BSI references relate to the work on this standard:
Committee reference RUC/20 Draft for comment 76/51484 DC

Cooperating organizations

The Rubber Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

*British Association of Synthetic Rubber Manufacturers
*British Rubber Manufacturers' Association
Department of Industry
Institution of Production Engineers
Malaysian Rubber Producers' Research Association
*Ministry of Defence
Rubber and Plastics Research Association of Great Britain
Rubber Growers' Association
Society of Motor Manufacturers and Traders Ltd.

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

Chemical Industries Association
Plastics and Rubber Institute
Tyre Manufacturers' Conference
Individual experts

Amendments issued since publication

Amd. No.	Date of issue	Text affected

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