BSI

BS 4470 : 1980
ISO 2302-1978
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Methods of test for

Raw isobutene-isoprene rubbers (IIR or butyl)

[ISO title : Rubber, isobutene-isoprene (IIR) — Evaluation procedures]

Méthodes d'essai des caoutchoucs isobutènes-isoprénes (IIR ou butyl) bruts

Prüfverfahren für rohe Isobuten-Isopren-Kautschuke (IIR oder Butyl)

British Standards Institution

Gr 3

Contents

National foreword

This British Standard has been prepared under the direction of the Rubber Standards Committee. It is identical with ISO 2302-1978 'Rubber, isobutene-isoprene (IIR) — Evaluation procedures' and replaces the 1977 edition of BS 4470, which was identical with ISO 2302-1975.

The principal change from the previous edition is the inclusion of the oven method as an alternative to the hot-mill method for the determination of volatile matter. The title has been brought into line with current editorial practice.

Terminology and conventions. The text of the international standard has been approved as suitable for publication, without deviation, as a British Standard. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

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

Where the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

Cross-references

International standard	*Corresponding British Standard*
ISO 37-1977	BS 903 Methods of testing vulcanized rubber Part A2 : 1971 Determination of tensile stress-strain properties (Technically equivalent)
ISO 247-1978	BS 5923 Methods for chemical analysis of rubber Part 1 : 1980 Determination of ash (Identical)
ISO 248-1978	BS 1673 Methods for testing raw rubber and unvulcanized compounded rubber Part 2 : 1967 Chemical analysis of raw natural rubber (Technically equivalent)
ISO/R 289-1963	Part 3 : 1969 Methods of physical testing (Technically equivalent)
ISO 667-1975	Part 3 : 1969 Methods of physical testing (Technically equivalent)
ISO 1795-1974 and ISO 1796-1972	Part 1 : 1976 Sampling and further preparative procedures (Technically equivalent)
ISO 2058-1973	Part 5/5.2-4 : 1964 Analysis of styrene butadiene copolymers (SBR) (Relevant clauses are technically equivalent)
ISO 2393-1973	BS 1674 : 1976 Specification for equipment and general procedure for mixing and vulcanizing rubber test mixes (Technically equivalent)-(See note.)
ISO 3417-1977	BS 1673 Methods for testing raw rubber and unvulcanized compounded rubber Part 10 : 1977 Measurement of prevulcanizing and curing characteristics by means of curemeters (Technically equivalent)

NOTE. Although the mill speeds and friction ratios in ISO 2393 and BS 1674 differ from one another, these differences have not been found to affect vulcanizate properties significantly.

Additional information. ISO 2302 does not specify the temperature for conditioning the batch. It is customary in the UK to condition the batch at 23 ± 2 °C, which is one of the ISO preferred temperatures given in ISO 2393.

The NBS Standard reference materials referred to in **5.1** and **7.1** correspond to materials given in BS 4398 'Compounding ingredients for rubber test mixes'.

In the UK it is found convenient to sheet the batch to a thickness of 8 mm rather than 6 mm (see **7.2.2.6**). The thicker sheet can be used directly in the Mooney viscosity test for which BS 1673 : Part 3 requires a thickness of 8 mm.

British Standard Methods of test for

Raw isobutene-isoprene rubbers (IIR or butyl)

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/R 289, *Determination of viscosity of natural and synthetic rubbers by the shearing disk viscometer.*

ISO 667, *Compounded rubber — Determination of the rate of cure using the shearing disk viscometer.*

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

ISO 1796, *Raw rubber — Sample preparation.* [1)]

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 disk 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 rubber.

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/R 289.

Because the shearing disk 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.

5 TEST RECIPE FOR EVALUATION OF VULCANIZATION CHARACTERISTICS

5.1 Standard test formula

The standard test formula is given in the following table.

1) At present at the stage of draft. (Revision of ISO 1796-1972.)

The materials shall be NBS[1] 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
Sulphur	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 disulphide.

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.

	Duration (min)
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 sulphur 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.

7 TEST RECIPE FOR EVALUATION OF RATE OF CURE AND SCORCH TESTING

7.1 Standard test formula

The standard test formula is given in the following table.

The materials shall be NBS[1] 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
Sulphur	371	2,00
TMTD*	374	0,60
		Total 104,60

* Tetramethylthiuram disulphide.

7.2 Procedure

7.2.1 *Equipment and procedure*

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

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

7.2.2 *Mill mixing procedure*

The standard laboratory mill batch mass, in grams, shall be based on twice the formula mass (i.e. 2 × 104,60 g = 209,20 g). The surface temperature of the rolls shall be maintained at 32,5 ± 2,5 °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)
7.2.2.1 Band the rubber with the mill opening set at 0,65 mm. Add the zinc oxide as soon as the rubber has banded	4
7.2.2.2 Make one 3/4 cut from each side	3
7.2.2.3 Add the sulphur and the TMTD	2
7.2.2.4 Make three 3/4 cuts from each side	2
7.2.2.5 Cut the batch from the mill and pass the rolled batch endwise through the rolls six times	3
Total time	14

7.2.2.6 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.

8 EVALUATION OF VULCANIZATION CHARACTERISTICS ACCORDING TO VISCOSITY INDEX

Measure the times t_5 and t_{35} at 145 °C in accordance with ISO 667.

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

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ISO 2302-1978

This British Standard, having been prepared under the direction of the Rubber Standards Committee, was published under the authority of the Executive Board and comes into effect on 30 June 1980.

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The following BSI references relate to the work on this standard:
Committee reference RUC/20 Draft for comment 79/53188 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 (Chemicals and Textiles)
Medical Sterile Products Association
*Ministry of Defence
*Plastics and Rubber Institute
Rubber and Plastics Research Association of Great Britain
Rubber Growers' Association
Society of Motor Manufacturers and Traders Limited
The Malaysian Rubber Producers' Research Association

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
Tyre Manufacturers' Conference (Service Committee)

Amendments issued since publication

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