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British Standard

Test recipe and evaluation of vulcanization characteristics for styrene-butadiene rubber (SBR) masterbatches with carbon black or carbon black and oil

[ISO title: Rubber, styrene-butadiene (SBR) – Masterbatches with carbon black or carbon black and oil – Test recipe and evaluation of vulcanization characteristics]

Formule d'essai et évaluation des caractéristiques de vulcanisation des mélanges-maîtres de caoutchouc butadiène-styrène (SBR) avec du noir de carbone ou avec du noir de carbone et de l'huile

Prüfrezept und Auswertung der Vulkanisierungseigenschaften von Grundmischungen aus Styrol-Butadien-Kautschuk (SBR) mit Ruß oder mit Ruß und Öl

Foreword

This British Standard has been prepared under the direction of the Rubber Standards Committee. It is identical with ISO 4659 – 1977 'Rubber, styrene-butadiene (SBR) – Masterbatches with carbon black or carbon black and oil – Test recipe and evaluation of vulcanization characteristics'.

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.

Wherever the words 'International Standard', relating to this publication, 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 (on the baseline) as the decimal marker.

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

Reference to international standard	Corresponding British Standard
ISO 37 – 1977	BS 903 Methods of testing vulcanized rubber Part A2 : 1971 Determination of tensile stress-strain properties
	BS 1673 Methods of testing raw rubber and unvulcanized compounded rubber
ISO 667 – 1975	BS 1673 : Part 3 : 1969 Methods of physical testing
ISO 1795 – 1974 ISO 1796 – 1972	BS 1673 : Part 1 : 1976 Sampling and further preparative procedures
ISO 3417 – 1977	BS 1673 : Part 10 : 1977 Measurement of prevulcanizing and curing characteristics by means of curemeters
ISO 2393 – 1973	BS 1674 : 1976 Equipment and general procedure for mixing and vulcanizing rubber test mixes

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 clause **3.**

Additional information. It is customary in the UK to condition the rubber (see **4.2.9** and **5.1**) at 23 °C, which is one of the preferred temperatures given in ISO 2393 – 1973.

British Standards Institution

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the standard materials, equipment and processing methods for evaluating the vulcanization characteristics of masterbatches of styrene-butadiene rubber (SBR) with carbon black or carbon black and oil.

2 REFERENCES

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

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

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, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

3 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
Masterbatch	—	100 + x* + y**
Zinc oxide	370	3,00
Sulphur	371	1,75
Stearic acid	372	1,50
N-tert-butyl-2-benzothiazole sulphenamide (TBBS)	384	1,25
Total		107,50 + x + y

* x is the number of parts of carbon black to 100 parts of polymer in the masterbatch.

** y is the number of parts of oil to 100 parts of polymer in the masterbatch.

NOTE — TBBS : *N-tert*-butyl-2-benzothiazole 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.

4 PROCEDURE

4.1 Equipment and procedure

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

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

4.2 Mill mixing procedure

The standard laboratory mill batch mass factor shall be selected to the nearest 0,5 to give as large a total mass as possible that does not exceed 525 g. The surface temperature of the rolls shall be maintained at 50 ± 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 (minutes)
4.2.1 Band the rubber with the mill opening set at 1,4 mm	2
4.2.2 Add the sulphur slowly and evenly across the rubber	2
4.2.3 Add the stearic acid. Make one 3/4 cut from each side	2
4.2.4 Add the zinc oxide and the TBBS	3
4.2.5 Make three 3/4 cuts from each side	3
4.2.6 Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise between the rolls six times	2
Total time	14

4.2.7 Sheet the batch to an approximate thickness of 6 mm, and check weigh. Remove sufficient sample for shearing disc viscometer and/or oscillating disc curemeter testing.

4.2.8 Immediately sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

4.2.9 Condition the batch at normal laboratory temperature for 2 to 24 h after mixing and prior to vulcanizing.

5 EVALUATION OF VULCANIZATION CHARACTERISTICS

5.1 Evaluation according to stress-strain properties

Vulcanize sheets at 145 °C for three periods selected from a cure series of 15, 25, 35, 50 and 75 min (see ISO 2393).

NOTE – The three periods of cure selected should be chosen so that they ensure an undercure, optimum cure and overcure.

Condition the vulcanized test slabs for 16 to 72 h.

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

5.2 Evaluation according to oscillating disc curemeter test

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 degree
die temperature :	160 °C

5.3 Evaluation according to shearing disc viscometer test

Measure the times of t_5 and t_{35} at 150 °C in accordance with ISO 667.

NOTE – Curemeter and viscometer tests may be considered as alternative methods for the evaluation of 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 75/53268 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
Institution of Production Engineers
*Ministry of Defence
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
Plastics and Rubber Institute
Tyre Manufacturers' Conference (Service Committee)

Amendments issued since publication

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