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ISO 4659-1981
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British Standard Method for

Evaluation of vulcanization characteristics for raw styrene-butadiene rubber (SBR) masterbatches with carbon black or carbon black and oil (including test recipe)

[ISO title : Rubber, raw styrene-butadiene (carbon black or carbon black and oil masterbatches) – Test recipe and method of evaluation]

Méthode d'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 (y inclus formule d'essai)

Verfahren zur Bewertung der Vulkanisierungseigenschaften von Grundmischungen aus Styrol-Butadien-Kautschuk (SBR) mit Ruß oder mit Ruß und Öl (einschließlich Prüfverfahren)

National foreword

This revision of this British Standard has been prepared under the direction of the Rubber Standards Committee and is identical with ISO 4659-1981 'Rubber, raw styrene-butadiene (carbon black or carbon black and oil masterbatches) – Test recipe and method of evaulation', published by the International Organization for Standardization (ISO).

The principal changes from the 1978 edition, which is now withdrawn, are as follows.

(a) 150 °C has been introduced as an alternative temperature of vulcanization. This is in line with the aim of ultimately establishing a single vulcanization temperature for all synthetic rubbers of 150 °C.

(b) The evaluation of vulcanization characteristics according to the shearing disc viscometer test has been deleted since this method is rarely used.

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

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

Wherever 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 471-1977	Part A35 : 1978 Standard temperatures, humidities and times for the conditioning and testing of test pieces (Identical)
ISO 1795-1974 ISO 1796-1980	BS 6315 : 1982 Methods for sampling and sample preparation of raw rubber (Technically equivalent)
ISO 2393-1973	BS 1674 : 1976 Specification for equipment and general procedure for mixing and vulcanizing rubber test mixes (Technically equivalent)
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)

British Standards Institution

The NBS Standard reference materials referred to in clause **3** correspond to materials given in BS 4398 : 1969 'Compounding ingredients for rubber test mixes'.

Additional information. It is customary in the UK to condition the rubber (see **3.2.2.9** and **4.1**) at 23 °C, which is one of the preferred temperatures given in ISO 2393-1973.

Textual error. Attention is drawn to an error in the ISO text, given below, which ISO have confirmed will be corrected in the next edition. The note to **4.2** should be deleted as macro-dies are not covered in ISO 3417.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

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

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 recipe

3.1 Standard test formula

The standard test formula is given in the table.

The materials shall be NBS[1] standard reference materials as indicated in the table, or shall be in accordance with equivalent national standards.

Table

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
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.

*** 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.

3.2 Procedure

3.2.1 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 USA.

3.2.2 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 should be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

	Duration (min)
3.2.2.1 Band the rubber with the mill opening set at 1,4 mm	2
3.2.2.2 Add the sulphur slowly and evenly across the rubber	2
3.2.2.3 Add the stearic acid. Make one 3/4 cut from each side	2
3.2.2.4 Add the zinc oxide and the TBBS	3
3.2.2.5 Make three 3/4 cuts from each side	3
3.2.2.6 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

3.2.2.7 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.

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

3.2.2.9 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing at a standard laboratory temperature (see ISO 471).

4 Evaluation of vulcanization characteristics

4.1 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.

NOTES

1 Alternatively, vulcanize the sheets at 150 °C for three periods selected from a cure series of 15, 20, 25, 30, 35 and 50 min. These conditions will give results different from those obtained with the standard vulcanization conditions.

2 The three periods of cure selected should cover the undercure, optimum cure and overcure of the masterbatch under test.

Condition the vulcanized test slabs for 16 to 72 h at a standard laboratory temperature (see ISO 471).

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

4.2 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° arc
selectivity :	to be chosen to give at least 75 % full scale deflection at M_H
die temperature :	160 °C
pre-heat time :	none
	NOTE — If macro-dies are used, a pre-heat time of 1 min is necessary *

5 Precision

To be added later.

*See national foreword

Publications referred to

See national foreword.

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The following BSI references relate to the work on this standard:
Committee reference RUC/20 Draft for comment 80/51229 DC

Committees responsible for this British Standard

This British Standard was published under the direction of the Rubber Standards Committee RUC/-. Its preparation was entrusted to Technical Committee RUC/20 upon which the following bodies were represented:

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British Rubber Manufacturers' Association
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Ministry of Defence
Plastics and Rubber Institute

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