
International Standard



4659

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Rubber, raw styrene-butadiene (carbon black or carbon black and oil masterbatches) — Test recipe and method of evaluation

Caoutchouc butadiène-styrène brut (mélanges-maîtres avec du noir de carbone ou avec du noir de carbone et de l'huile) — Formule d'essai et méthode d'évaluation

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4659 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*.

The first edition (ISO 4659-1977) had been approved by the member bodies of the following countries :

Australia	India	South Africa, Rep. of
Belgium	Italy	Spain
Bulgaria	Korea, Rep. of	Sweden
Canada	Mexico	Turkey
Czechoslovakia	Netherlands	United Kingdom
France	Poland	USA
Germany, F. R.	Portugal	Yugoslavia
Hungary	Romania	

No member body expressed disapproval of the document.

This second edition, which supersedes ISO 4659-1977, incorporates draft Amendment 1, which was circulated to the member bodies in January 1980 and which has been approved by the member bodies of the following countries :

Belgium	India	Spain
Brazil	Italy	Sri Lanka
China	Korea, Rep. of	Sweden
Czechoslovakia	Mexico	Switzerland
Egypt, Arab Rep. of	Netherlands	Thailand
France	Poland	Turkey
Germany, F. R.	Romania	United Kingdom
Hungary	South Africa, Rep. of	USSR

The member body of the following country expressed disapproval of the document on technical grounds :

USA

Rubber, raw styrene-butadiene (carbon black or carbon black and oil masterbatches) — Test recipe and method of evaluation

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 NBS¹⁾ 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.