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INTERNATIONAL STANDARD

ISO 4659

Third edition
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Rubber, styrene-butadiene (carbon black or carbon black and oil masterbatches) — Evaluation procedure

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

Reference number
ISO 4659 : 1989 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

This third edition cancels and replaces the second edition (ISO 4659 : 1981). The main technical differences introduced in this new edition of ISO 4659 in comparison with the second edition are as follows:

— a new clause covering sampling and sample preparation has been introduced (clause 3);

— a new clause specifying physical and chemical tests on the raw masterbatch has been introduced (clause 4);

— the duration of the procedure has been reduced from 3 min to 2 min (see 5.2.2.5);

— a new clause giving the required format for a test report has been introduced (clause 8).

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Rubber, styrene-butadiene (carbon black or carbon black and oil masterbatches) — Evaluation procedure

1 Scope

This International Standard specifies

a) physical and chemical tests on raw masterbatches;

b) standard materials, a standard test formula, equipment and processing methods for evaluating vulcanization characteristics of masterbatches of styrene-butadiene rubbers (SBR) with carbon black or carbon black and oil.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37 : 1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 247 : 1978, *Rubber — Determination of ash.*

ISO 248 : 1979, *Rubbers, raw — Determination of volatile matter content.*

ISO 289 : 1985, *Rubber, unvulcanized — Determination of Mooney viscosity.*

ISO 471 : 1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1795 : 1974, *Raw rubber in bales — Sampling.*

ISO 1796 : 1982, *Rubber, raw — Sample preparation.*

ISO 2393 : 1973, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417 : 1977, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

3 Sampling and sample preparation

3.1 A sample of mass approximately 1 500 g shall be taken by the method described in ISO 1795.

3.2 Preparation of the test portion shall be in accordance with ISO 1796.

4 Physical and chemical tests on masterbatches

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289, on a test portion prepared as indicated in ISO 1796.

4.2 Volatile matter

Determine the volatile matter content in accordance with ISO 248.

4.3 Ash

Determine the ash content in accordance with ISO 247.

5 Preparation of the test mix for evaluation of masterbatches of styrene-butadiene rubber

5.1 Standard test formula

The standard test formula is given in table 1.

The materials shall be NIST[1] standard reference materials as indicated in table 1, or other, equivalent national or international standard reference materials.

Table 1 — Standard test formula for evaluation of masterbatches of styrene-butadiene rubber

Material	NIST standard reference material number	Parts by mass
Masterbatch	—	100 + x*) + y**)
Zinc oxide	370	3,00
Sulfur	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 rubber in the masterbatch.

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

***) *N-tert*-butyl-benzothiazole-2-sulfenamide. 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.

5.2 Procedure

5.2.1 Equipment and procedure

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

5.2.2 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 °C ± 5 °C throughout the mixing.

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings may be necessary.

	Duration (min)
5.2.2.1 Band the masterbatch with the mill opening set at 1,4 mm	2,0
5.2.2.2 Add the sulfur slowly and evenly across the masterbatch	2,0
5.2.2.3 Add the stearic acid. Make one 3/4 cut from each side	2,0
5.2.2.4 Add the zinc oxide and the TBBS	3,0
5.2.2.5 Make three 3/4 cuts from each side	2,0
5.2.2.6 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	2,0
Total time	13,0

5.2.2.7 Sheet the batch to approximately 6 mm. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for oscillating disc curemeter testing.

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

5.2.2.9 Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 471.

6 Evaluation of vulcanization characteristics with the oscillating disc curemeter test

Measure the following standard test parameters:

M_L, M_H or M_{HR}, t_{s1}, t'_c (50) and t'_c (90)

1) National Institute of Standards and Technology (formerly the National Bureau of Standards) of the USA.

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 % of full scale deflection at M_H or M_{HR}
	NOTE — With some masterbatches, 75 % may not be attainable.
die temperature:	160 °C ± 0,3 °C
pre-heat time:	none

7 Evaluation of tensile stress-strain properties of vulcanized test mixes

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

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

The three periods of cure shall be chosen to cover the undercure, optimum cure and overcure of the masterbatch under test.

Condition the vulcanized sheets for 16 h to 96 h, if possible at standard temperature and humidity as defined in ISO 471.

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

8 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the idendification of the sample;

c) the reference materials used;

d) the method used for volatile matter determination (mill or oven);

e) the vulcanizing temperature and times used in clause 7;

f) any unusual features noted during the determination;

g) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional;

h) the results and the units in which they have been expressed;

i) the date of the test.

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