INTERNATIONAL STANDARD

ISO 4659

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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 4659:1997(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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4659 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fourth edition cancels and replaces the third edition (ISO 4659:1989), which has been technically revised.

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

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies

— physical and chemical tests on raw rubbers;

— standard materials, standard test formulations, equipment and processing methods for evaluating the vulcanization characteristics of styrene-butadiene rubber masterbatched with carbon black or with 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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties.*

ISO 247:1990, *Rubber — Determination of ash.*

ISO 248:1991, *Rubbers, raw — Determination of volatile-matter content.*

ISO 289-1:1994, *Rubber, unvulcanized — Determination using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity.*

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing.*

ISO 1795:1992, *Rubber, raw, natural and synthetic — Sampling and further preparative procedures.*

ISO 2393:1994, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

ISO 6502:—[1], *Rubber — Measurement of vulcanization characteristics with rotorless curemeters.*

ISO 11235:—[2], *Rubber compounding ingredients — Sulfenamide-type accelerators — Methods of test.*

3 Sampling and sample preparation

3.1 A laboratory sample of approximately 1,5 kg shall be taken by the method described in ISO 1795.

3.2 Preparation of test portions shall be in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1 on a test portion prepared as indicated in ISO 1795 (without massing).

If massing is necessary, use a mill with its roll surfaces maintained at a temperature of 35 °C ± 5 °C and record this fact in the test report.

Record the result as ML (1 + 4) at 100 °C.

4.2 Volatile matter

Determine the volatile-matter content in accordance with ISO 248.

4.3 Ash content

Determine the ash content in accordance with ISO 247.

5 Preparation of test mixes for evaluation of masterbatches of styrene-butadiene rubber

5.1 Standard test formulations

The standard test formulations are given in table 1.

The materials shall be national or international standard reference materials. If no standard reference material is available, the materials to be used shall be agreed by the interested parties.

1) To be published. (Revision of ISO 6502:1991)

2) To be published.

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

Material	Parts by mass
Masterbatch	100 + x*) + y**)
Zinc oxide	3,00
Sulfur	1,75
Stearic acid	1,50
TBBS***)	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 insoluble-matter content, determined in accordance with ISO 11235, of less than 0,3 %. The material shall be stored at room temperature in a closed container and the insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the TBBS shall be discarded or recrystallized.

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 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)	Cumulative time (min)
a)	Band the masterbatch with the mill opening set at 1,4 mm.	2,0	12,0
b)	Add the sulfur slowly and evenly across the masterbatch.	2,0	14,0
c)	Add the stearic acid. Make one 3/4 cut from each side.	2,0	16,0
d)	Add the zinc oxide and the TBBS.	3,0	19,0
e)	Make three 3/4 cuts from each side.	2,0	11,0
f)	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	13,0

g) Sheet the batch to approximately 6 mm and determine the mass of the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than +0,5 % or –1,5 %, discard the batch and re-mix.

h) Remove sufficient material for curemeter testing.

i) Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring specimens in accordance with ISO 37.

6 Conditioning of compounds

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.

7 Evaluation of vulcanization characteristics by a curemeter test

7.1 Using an oscillating-disc curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, 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° of arc
selectivity:	to be chosen to give at least 75 % of full-scale deflection at M_H With some rubbers, 75 % may not be attainable
die temperature:	160 °C ± 0,3 °C
pre-heat time:	none

7.2 Using a rotorless curemeter

Measure the following standard test parameters:

F_L, F_{max} at defined time, t_{s1}, $t'_c(50)$ and $t'_c(90)$

in accordance with ISO 6502, using the following test conditions:

oscillation frequency:	1,7 Hz (100 cycles per minute)
amplitude of oscillation:	0,5° of arc
selectivity:	to be chosen to give at least 75 % of full-scale deflection at F_{max} With some rubbers, 75 % may not be attainable
die temperature:	160 °C ± 0,3 °C
pre-heat time:	none

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

Vulcanize sheets at 145 °C for 25 min, 35 min and 50 min. Alternatively, vulcanize sheets at 150 °C for 20 min, 30 min and 50 min.

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