INTERNATIONAL STANDARD

ISO 3257

Third edition
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Rubber compounding ingredients — Carbon black — Method of evaluation in styrene-butadiene rubbers

Ingrédients de mélange du caoutchouc — Noir de carbone — Méthode d'évaluation dans les caoutchoucs butadiène-styrène

Reference number
ISO 3257:1992(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 3257 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Sub-Committee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This third edition cancels and replaces the second edition (ISO 3257:1982). The main technical differences introduced in this new edition of ISO 3257 in comparison with the second edition are as follows:

— in note 1 to table 1, the limits for the Mooney viscosity of the reference rubber have been modified;

— in 3.2.2 h) (previously 3.2.2.8), a condition governing the final mass of the test mix has been added;

— in clause 4 (previously subclause 4.2), the limit on the selectivity of the oscillating disc curemeter has been modified, and a tolerance added to the die temperature;

— a test report clause has been added (clause 6).

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Rubber compounding ingredients — Carbon black — Method of evaluation in styrene-butadiene rubbers

1 Scope

This International Standard specifies standard materials, equipment and processing methods for evaluating carbon black in styrene-butadiene rubbers (SBR).

NOTE 1 Variations in equipment and testing procedure permitted in this International Standard can lead to discrepant results. Therefore, carbon black is preferably compared to a reference carbon black tested under the same conditions.

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

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

3 Preparation of test mixes for evaluation of carbon black in styrene-butadiene rubbers

3.1 Standard test formula

The standard test formula is given in table 1.

The materials shall be NIST*) standard reference materials as indicated in table 1, or shall be in accordance with equivalent national or international standards.

3.2 Procedure

3.2.1 Equipment and procedure

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

3.2.2 Mill mixing procedure

The standard laboratory mill batch mass, in grams, shall be based on four times the formula mass (see table 1). The surface temperature of the rolls shall be maintained at 50 °C ± 5 °C throughout the mixing.

All mill openings shall be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

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

	Duration (min)	Cumulative time (min)
a) Band the rubber with the mill opening set at 1,1 mm, and make 3/4 cuts every 30 s from alternate sides	2	2
b) Add the sulfur slowly and evenly across the rubber	2	4
c) Add the stearic acid. Make one 3/4 cut from each side	2	6
d) Add carbon black evenly across the mill at a uniform rate. When about half the black has been incorporated, open the mill to 1,4 mm and make one 3/4 cut from each side. Then add the remainder of the carbon black. When all the black has been incorporated, open the mill to 1,8 mm and make one 3/4 cut from each side. Be certain to add the black that has dropped into the mill pan	10	16
e) Add the zinc oxide and the TBBS with the mill opening at 1,8 mm	3	19
f) Make three 3/4 cuts from each side	3	22
g) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise through the rolls six times	2	24
Total time	24	

h) Sheet the batch to approximately 6 mm and check-weigh the batch. 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.

i) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring test pieces and other test specimens.

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

Table 1 — Standard test formula for evaluation of carbon black in butadiene-styrene rubbers

Material	NIST standard reference material number	Parts by mass
SBR 1500[1]	386	100,00
Zinc oxide	370	3,00
Sulfur	371	1,75
Stearic acid	372	1,00
Carbon black (except N 700 series)[2]	—	50,00
TBBS[3]	384	1,00
Total		156,75

1) A European equivalent to NIST standard reference material 386 has been developed to match by Erichem Elastomeri SpA. This EST (European Standard Type) rubber is an SBR 1500 type using a rosin acid emulsifier and a staining stabilizer.

The Mooney viscosity [ML (1 + 4) at 100 °C], determined in accordance with ISO 289, of this standard reference material shall have limits of ± 1 Mooney unit within the absolute range of 48 to 52, but with the preferred viscosity of 50 to 51.

2) If N 700 series carbon black is used, the number of parts by mass shall be 80,00, making a total parts by mass of 186,75. The carbon black shall be dried for 30 min at 105 °C or 125 °C and cooled to room temperature in a dessicator prior to use.

3) *N-tert*-butylbenzothiazole-2-sulfenamide. This shall be supplied in powder form, with an initial ether- or ethanol-insoluble-matter content of less than 0,3 % (*m/m*). The material shall be stored at room temperature in a closed container and the ether- or ethanol-insoluble-matter content shall be checked every 6 months. If this is found to exceed 0,75 % (*m/m*), the material shall be discarded or recrystallized.

4 Evaluation of vulcanization characteristics by the oscillating disc curemeter test

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: ± 1,00° ± 0,03° of arc