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# International Standard



# 3257

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Rubber compounding ingredients — Carbon black — Test recipe and method of evaluation in styrene- butadiene rubbers

*Ingrédients de mélange du caoutchouc — Noir de carbone — Formule d'essai et méthode d'évaluation dans les caoutchoucs  
butadiène-styrène*

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Price based on 2 pages

## 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 3257 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*. The first edition (ISO 3257-1975) had been approved by the member bodies of the following countries :

Australia	Hungary	Spain
Austria	India	Sweden
Belgium	Ireland	Switzerland
Bulgaria	Italy	Turkey
Canada	Mexico	United Kingdom
Czechoslovakia	Netherlands	USA
Egypt, Arab Rep. of	New Zealand	USSR
France	Portugal	Yugoslavia
Germany, F. R.	Romania	

No member body had expressed disapproval of the document.

This second edition, which cancels and replaces ISO 3257-1975, incorporates draft amendment 3, which was circulated to the member bodies in January 1980 and has been approved by the member bodies of the following countries :

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

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Netherlands  
USA

# Rubber compounding ingredients — Carbon black — Test recipe and method of evaluation in styrene-butadiene rubbers

## 1 Scope and field of application

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

NOTE — 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 References

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

ISO/R 289, *Determination of viscosity of natural and synthetic rubbers by the shearing disk viscometer.*

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

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 Test recipe

### 3.1 Standard test formula

The standard test formula is given in the table.

The materials shall be NBS<sup>1)</sup> 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
SBR 1500*	386	100,00
Zinc oxide	370	3,00
Sulphur	371	1,75
Stearic acid	372	1,00
Carbon black	—	50,00
TBBS**	384	1,00
		156,75

\* A European equivalent to NBS standard reference material 386 has been developed to match by ANIC. 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/R 289, of this standard reference material should have limits of  $\pm 1$  Mooney unit within the absolute range of 50 to 56, but with the preferred viscosity of 52 to 53.

\*\* *N-tert*-butyl 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 content 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, in grams, shall be based on four times the formula mass. The surface temperature of the rolls shall be maintained at  $50 \pm 5$  °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)
<b>3.2.2.1</b> Band the rubber with the mill opening set at 1,1 mm, and make 3/4 cuts every 30 s from alternate sides .....	7
<b>3.2.2.2</b> Add the sulphur slowly and evenly across the rubber .....	2
<b>3.2.2.3</b> Add the stearic acid. Make one 3/4 cut from each side .....	2
<b>3.2.2.4</b> 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 .....	12
<b>3.2.2.5</b> Add the zinc oxide and the TBBS with the mill opening at 1,8 mm .....	3
<b>3.2.2.6</b> Make three 3/4 cuts from each side .....	3
<b>3.2.2.7</b> 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
<b>Total time</b>	<b>31</b>
<b>3.2.2.8</b> Sheet the batch to approximately 6 mm and check weigh the batch.	
<b>3.2.2.9</b> Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.	
<b>3.2.2.10</b> Condition the batch for 2 to 24 h after mixing and prior to vulcanizing at a standard laboratory temperature (see ISO 471).	

NOTE — The mastication time of 7 min was designed to allow sufficient time to break down all varieties of polymers that may be used as standard or secondary standard rubbers. If the standard rubber used

can be masticated in less time, this period may be reduced to a minimum of 2 min, provided that the reduced mastication time gives equivalent results to the 7 min period.

**4 Evaluation of vulcanization characteristics**

**4.1 Evaluation according to stress-strain properties**

Vulcanize sheets at 145 °C for two (or, preferably, three) periods selected from a cure series of 25, 35, 50 and 75 min, depending on the characteristics of the carbon black under test.

NOTE — Alternatively, vulcanize sheets at 150 °C for two (or, preferably, three) periods selected from a cure series of 20, 25, 30, 35 and 50 min, depending on the characteristics of the carbon black under test. These conditions will give results that differ from those obtained with the standard vulcanization conditions.

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 :

$M_L$ , $M_H$ , $t_s$ , $t_c(50)$ 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.