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# INTERNATIONAL STANDARD



# 1658

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

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## Natural rubber (NR) – Test recipes and evaluation of vulcanization characteristics

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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 1658 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*, and circulated to the Member Bodies in January 1971.

It has been approved by the Member Bodies of the following countries :

Australia	Israel	Spain
Canada	Italy	Sri Lanka
Czechoslovakia	Netherlands	Sweden
Egypt, Arab Rep. of	New Zealand	Switzerland
Germany	Portugal	Thailand
Greece	Romania	United Kingdom
India	South Africa, Rep. of	U.S.S.R.

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

France  
Hungary  
U.S.A.

# Natural rubber (NR) – Test recipes and evaluation of vulcanization characteristics

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies standard materials, equipment and processing methods for evaluating natural rubbers (NR).

Two recipes are recommended :

a) **Gum stock recipe**, for comparative testing of the vulcanization characteristics of natural rubber for use in non-black-filled compounds.

b) **Black-filled recipe**, for comparative testing of natural rubber for use in black-filled compounds. It can also be used for comparative testing with isoprene rubbers (IR).

## 2 REFERENCES

ISO/R 37, *Determination of tensile stress-strain properties of vulcanized rubbers.*

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

ISO 1796, *Raw rubber – Sample preparation.*

ISO/R 2007, *Raw rubber and unvulcanized rubber – Rapid plasticity test.*

ISO 2393, *Rubber test mixes – Preparation, mixing and vulcanization – Equipment and procedures.*

## 3 GUM STOCK RECIPE

### 3.1 Standard test recipe

The standard recipe is given in the following table.

The materials shall be NBS\* Standard reference materials as indicated in the table, or shall be in accordance with equivalent national standards.

Material	NBS Standard reference material number	Parts by mass
Natural rubber (NR)	—	100,00
Zinc oxide	370	6,00
Sulphur	371	3,50
Stearic acid	372	0,50
MBT <sup>1)</sup>	383	0,50
		Total 110,50

1) 2-Mercaptobenzothiazole.

### 3.2 Procedure

#### 3.2.1 Equipment and procedure

Equipment and procedure for the 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 recipe mass.

The surface temperature of the rolls shall be maintained at  $70 \pm 5$  °C throughout the mixing.

The rubber shall be homogenized in accordance with ISO 1796.

NOTE – Smaller batch sizes may be used by reducing the clearance between the guides without adjustment of the mixing cycle or nip openings. If a reduced batch mass is used without adjustment of the distance between the guides, it will be necessary to adjust the nip openings and times. All nip openings shall be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

\* National Bureau of Standards of the U.S.A.

Duration  
(min.)

3.2.2.1 Pass the rubber twice between the rolls without banding with the mill opening set at 0,20 mm. . . . .	—
3.2.2.2 Band the rubber with the mill opening set at 1,4 mm. When a smooth band has been obtained, adjust the mill opening to 1,9 mm. . . . .	4
3.2.2.3 Add the zinc oxide, the stearic acid, the sulphur and the MBT. . . . .	4
3.2.2.4 Make three 3/4 cuts from each side. . . . .	3
3.2.2.5 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
Total time	<u>13</u>

Material	NBS Standard reference material number	Parts by mass
Natural rubber	—	100,00
Zinc oxide	370	5,00
Sulphur	371	2,25
Stearic acid	372	2,00
Oil furnace black (HAF) <sup>1)</sup>	378	35,00
TBBS <sup>2)</sup>	384	0,70
		Total 144,95

1) The current industry reference black may be used in place of NBS 378, but this may give slightly different results.  
2) *N-tert-butylbenzothiazole-2-sulphenamide*

- 3.2.2.6 Sheet the batch to approximately 6 mm and check weigh the batch.
- 3.2.2.7 Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.
- 3.2.2.8 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing.

**3.3 Evaluation of vulcanization characteristics**

**3.3.1 Stress-strain properties**

Vulcanize sheets at 140 °C for 40 min.  
Condition the vulcanized test slab for 16 to 72 h.  
Measure the stress-strain properties in accordance with ISO/R 37.

**3.3.2 Viscosity**

Determine the viscosity of the unvulcanized batch in accordance with ISO/R 289.

**4.2 Procedure**

**4.2.1 Equipment and procedure**

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

**4.2.2 Mill mixing procedure**

The standard laboratory mill batch mass, in grams, shall be based on four times the recipe mass.

The surface temperature of the rolls shall be maintained at 70 ± 5 °C throughout the mastication and mixing operation.

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

**4.2.2.1 MASTICATION STAGE**

With the mill roll set at 70 ± 5 °C and the mill opening set at 0,5 mm, load the rubber on to the mill and masticate until a smooth band and rolling bank is obtained.

After mastication, the rapid plasticity number (RPN) determined in accordance with ISO/R 2007 shall not exceed 45, which is approximately equivalent to a viscosity of 70 Mooney units determined in accordance with ISO/R 289.

**4.2.2.2 MIXING STAGE**

Duration  
(min)

1) Band the rubber with the mill opening set at 1,4 mm. . . . .	1
2) Add the stearic acid. Make one 3/4 cut from each side. . . . .	1



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**Natural rubber (NR) – Test recipes and evaluation of vulcanization characteristics****AMENDMENT 1**

Amendment 1 to International Standard ISO 1658 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*. It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

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Sub-clause 4.1 : Replace note 2) below the table by the following :

"2) *N-Tert*-butyl 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."

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- 3) Add the zinc oxide and the sulphur. Make one 3/4 cut from each side. . . . . 2
  - 4) Add the carbon black evenly across the mill at a uniform rate. When about half the black has been incorporated, open the mill to 1,9 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, make one 3/4 cut from each side. Be certain to add the black that has dropped into the mill pan.. . . . . 10
  - 5) Add the TBBS. Make three 3/4 cuts from each side. . . . . 3
  - 6) 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. . . . . 3
- Total time 20
- 7) Check weigh the batch.

- 8) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.
- 9) Condition the batch for 2 to 24 h after mixing and prior to vulcanizing.

**4.3 Evaluation of vulcanization characteristics**

**4.3.1 Stress-strain properties**

- Vulcanize sheets at 135 °C for periods of 20, 30, 40 and 60 min.
- Condition the vulcanized test slab for 16 to 72 h.
- Measure the stress-strain properties in accordance with ISO/R 37.

**4.3.2 Viscosity**

- Determine the viscosity of the unvulcanized batch in accordance with ISO/R 289.

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