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Second edition
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Natural rubber (NR) — Evaluation procedure

Caoutchouc naturel (NR) — Méthode d'évaluation



Reference number
ISO 1658:1994(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 1658 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 second edition cancels and replaces the first edition (ISO 1658:1973), plus Amendment 1:1976 to the first edition, which have been technically revised.

Annex A forms an integral part of this International Standard.

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Natural rubber (NR) — Evaluation procedure

1 Scope

This International Standard specifies

- physical and chemical tests on raw natural rubbers;
- standard materials, standard test formulae, equipment and processing methods for evaluating the vulcanization characteristics of natural rubber (NR).

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:—¹⁾, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties.*

ISO 248:1991, *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 1795:1992, *Rubber, raw, natural and synthetic — Sampling and further preparative procedures.*

ISO 2007:1991, *Rubber, unvulcanized — Determination of plasticity — Rapid-plastimeter method.*

ISO 2393:—²⁾, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

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

ISO 6502:1991, *Rubber — Measurement of vulcanization characteristics with rotorless curemeters.*

3 Sampling and further preparative procedures

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

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

1) To be published. (Revision of ISO 37:1977)

2) To be published. (Revision of ISO 2393:1973)

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289 on a test portion prepared as indicated in 3.2. Record the result as ML (1 + 4) at 100 °C.

4.2 Volatile-matter content

Determine the volatile-matter content by the oven method specified in ISO 248 on a test portion prepared as indicated in 3.2.

5 Preparation of the test mix

Three formulae are recommended:

- a) two gum-stock formulae for comparative testing of the vulcanization characteristics of natural rubber for use in non-black-filled compounds;

- b) a black-filled formula for comparative testing of natural rubber for use in black-filled compounds.

NOTE 1 Formula b) can also be used for comparative testing of isoprene rubbers (IR).

5.1 Standard test formulae

The standard test formulae are given in table 1.

The materials shall be national or international standard reference materials.

5.2 Procedure

5.2.1 Equipment and procedure

Equipment and procedure for the preparation, mixing and vulcanizing shall be in accordance with ISO 2393. The surface temperature of the mill rolls shall be maintained at $70\text{ °C} \pm 5\text{ °C}$ throughout the mixing. The rubber shall be homogenized in accordance with ISO 1795.

Table 1 — Standard test formulae

Material	Number of parts by mass		
	Formula 1 ACS 1	Formula 2 TBBS	Formula 3 Black-filled
Natural rubber	100,00	100,00	100,00
Zinc oxide	6,00	6,00	5,00
Sulfur	3,50	3,50	2,25
Stearic acid	0,50	0,50	2,00
Oil furnace black (HAF) IRB	—	—	35,00
MBT ¹⁾	0,50	—	—
TBBS ²⁾	—	0,70	0,70
Total	110,50	110,70	144,95

1) 2-Mercaptobenzothiazole.

2) *N*-tert-butyl-2-benzothiazole-sulfenamide. This shall be supplied in powder form having an initial ether-insoluble 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-insoluble or ethanol-insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 % (*m/m*), the material shall be discarded or recrystallized.

5.2.2 Mixing procedure for formulae 1 and 2 (gum compounds)

	Duration (min)
a) Pass the rubber twice between the rolls without banding, with the mill opening set at 0,2 mm.	—
b) Band the rubber with the mill opening set at 1,4 mm. When a smooth band has been obtained, adjust the mill opening at 1,8 mm.	4
c) Add the zinc oxide, the stearic acid, the sulfur and the MBT or TBBS.	4
d) Make three 3/4 cuts from each side.	3
e) 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	13
f) Check the mass of 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 remix.	
g) Cut sufficient material from the batch for curemeter testing and, if required, for determination of the Mooney viscosity of the unvulcanized batch in accordance with ISO 289. Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.	
h) 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.	

5.2.3 Mixing procedure for formulae 1 and 2 (gum compounds) using masterbatches

Compounding materials such as accelerators, sulfur or fillers can be incorporated into the rubber as masterbatches. This technique will improve the accuracy of compounding-material incorporation and is also more convenient.

The procedure for preparing masterbatches and test mixes for the gum compounds is given in annex A.

5.2.4 Mixing procedure for formula 3 (black-filled compound)

Load the rubber on to the mill with the mill opening set at 0,5 mm. Masticate until a smooth band and rolling bank are obtained.

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

	Duration (min)
a) Band the rubber with the mill opening set at 1,4 mm.	1
b) Add the stearic acid. Make one 3/4 cut from each side.	1
c) Add the zinc oxide and the sulfur. Make one 3/4 cut from each side.	2
d) 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
e) Add the TBBS. Make three 3/4 cuts from each side.	3
f) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch lengthways through the mill six times.	3
Total time	20

g) Check the mass of the batch. If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and remix.

h) Cut sufficient material from the batch for curemeter testing and, if required, for determination of the Mooney viscosity of the unvulcanized batch in accordance with ISO 289. Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

- i) 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 by a curemeter test

Measure the following standard test parameters:

$$M_L, M_{HR}, t_{s1}, t'_c(50), t'_c(90)$$

in accordance with ISO 3417 or ISO 6502, using the following test conditions:

oscillation frequency:	1,7 Hz (100 cycles per minute)
amplitude of oscillation:	1° arc (3° arc optional)
selectivity:	to be chosen to give at least 75 % of full-scale deflection at M_{HR}
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 140 °C for periods of 20 min, 30 min, 40 min and 60 min. 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:

- a reference to this International Standard;
- all details necessary for the identification of the sample;
- the standard test formula used;
- the reference materials used;
- the method used for the volatile-matter content determination;
- the curemeter test used in clause 6 (ISO 3417 or ISO 6502);
- any unusual features noted during the determination;
- 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, e.g. whether masterbatches were used;
- the results and the units in which they have been expressed;
- the date of the test.

Annex A

(normative)

Procedure for preparing gum compounds through use of masterbatches

A.1 Masterbatch formulation

The rubber for preparing masterbatches shall be of similar quality to that being tested. The proportions by mass of the compounding materials used are given in table A.1.

Table A.1 — Masterbatch formulation

Masterbatch	MBT	Sulfur	TBBS
Material	Number of parts by mass		
Natural rubber	100	100	100
Zinc oxide	120	120	120
Stearic acid	10	10	10
MBT	20	—	—
TBBS	—	—	28
Sulfur	—	140	—
Total	250	370	258

A.2 Test mix formulation

The test mixes are made up as given in table A.2.

Table A.2 — Test mix formulation

Material	Number of parts by mass	
	ACS 1	TBBS
Test rubber	95,00	95,00
MBT masterbatch	6,25	—
TBBS masterbatch	—	6,45
Sulfur masterbatch	9,25	9,25
	110,50	110,70

A.3 Masterbatch preparation by mill mixing procedure

The mill rolls are kept cool with running water throughout the mixing.

- a) Band the rubber with the mill opening set at 0,6 mm and masticate. Duration (min) 1
- b) Add the zinc oxide, stearic acid and MBT or TBBS or sulfur. Gradually widen the mill opening so as to maintain a rolling bank. When about 80 % of the material has been incorporated (mill opening approximately 1,0 mm), make one 3/4 cut from each side. 2
- c) Incorporate the rest of the material and, when no free powder is evident, make 3/4 cuts from each side until dispersion appears fully uniform. 5

Total time 8

- d) Cut the masterbatch from the mill and check the mass.
- e) Cool the rolls to $27\text{ °C} \pm 5\text{ °C}$ using running water at ambient temperature.
- f) Close the rolls of the mill to a tight nip and grind the masterbatch three times through the rolls, rolling the rubber into a ball between each pass.
- g) Sheet the masterbatch through a mill opening of 1,4 mm.

- h) Store the masterbatches in airtight containers at an ambient temperature of $23\text{ °C} \pm 2\text{ °C}$. The storage period should preferably not exceed three months.

A.4 Test mix preparation

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

	Duration (min)		Duration (min)
a) Band the rubber with the mill opening set at 0,8 mm. Make two 3/4 cuts from each side.	0,75	b) Add the MBT or TBBS and sulfur masterbatches. Make six 3/4 cuts from each side.	2,00
		c) Cut the batch from the mill and pass the rolled batch endwise through the rolls two times.	0,25
		Total time	3,00
		d) Sheet the batch through a mill opening of 1,4 mm.	
		e) 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.	

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