IS 7499 : 2001
ISO 1658 : 1994

भारतीय मानक

प्राकृतिक रबड़ (एन आर) — मूल्यांकन प्रक्रिया

(दूसरा पुनरीक्षण)

Indian Standard

NATURAL RUBBER (NR) — EVALUATION PROCEDURE

(Second Revision)

ICS 83.060

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 3

Rubber Sectional Committee, PCD 14

NATIONAL FOREWORD

The Indian Standard (Second Revision) which is identical with ISO 1658 :1994 'Natural rubber (NR) — **Evaluation procedure' issued** by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Rubber Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The first revision of this standard was published in 1981 which was essentially based on ISO 1658. Since ISO 1658 has subsequently been revised as ISO 1658 : 1994, the Committee decided to revise this standard to completely align it with ISO 1658 : 1994 and publish as a dual number standard.

The text of ISO standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated. However, that International Standard cross-referred in this adopted ISO Standard which has subsequently been revised, position in respect of latest ISO Standard has been given:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 37 : 1994 Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties	IS 3400 (Part 1) : 1987 Methods of test for vulcanized rubbers: Part 1 Tensile stress-strain properties (*second revision*)	Technically equivalent with minor deviations
ISO 248 : 1991 Rubbers, raw — Determination of volatile matter content	IS 3660 (Part 2) : 1985 Methods of test for natural rubber: Part 2 Determination of volatile matter (NR : 2) (*second revision*)	do
ISO 289-1 : 1994 Rubber, unvulcanized — Determinations using a shearing disc viscometer — Part 1 : Determination of Mooney viscosity	IS 3660 (Part 7) :1988 Methods of test for natural rubber: Part 7 Determination of Moony viscosity (NR : 8) (*second revision*)	do
ISO 471 : 1995 Rubber — Temperatures, humidities and times for conditioning and testing	IS 13867 : 1993 Rubber standard temperatures, humidities and times for the conditioning and time interval between vulcanization and testing of test pieces	do
ISO 1795 : 1992 Rubber, raw natural and synthetic — Sampling and further preparative procedures	IS 5599 : 1999 Rubber — Raw, natural and synthetic — Methods of sampling and sample preparation (*first revision*)	do
ISO 2007 : 1991, Rubber, unvulcanized Determination of plasticity — Rapid-plastimeter method	IS 3660 (Part 11) : 1999 Methods of test for natural rubber : Part 11 Determination of plasticity (NR : 12) (*first revision*)	do

(Continued on third cover)

IS 7499 : 2001
ISO 1658 : 1994

Indian Standard

NATURAL RUBBER (NR) — EVALUATION PROCEDURE

(Second Revision)

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.

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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 °C ± 5 °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) a reference to this International Standard;

b) all details necessary for the identification of the sample;

c) the standard test formula used;

d) the reference materials used;

e) the method used for the volatile-matter content determination;

f) the curemeter test used in clause 6 (ISO 3417 or ISO 6502);

g) any unusual features noted during the determination;

h) 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;

i) the results and the units in which they have been expressed;

j) 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.

	Duration (min)
a) Band the rubber with the mill opening set at 0,6 mm and masticate.	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 °C ± 5 °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 °C ± 2 °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 °C ± 5 °C throughout the mixing.

	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.

(Continued from second cover)

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 2393 : 1994 Rubber test mixes — *Preparation, mixing and vulcanization* — Equipment and procedures	IS 3660 (Part 8) : 1999 Methods of *test for natural rubber : Part 8 Mixing* and vulcanizing of rubber in standard compound (NR : 9) (*second revision*)	**Technically equivalent with minor deviations**
ISO 3417 : 1991 Rubber — Measurement *of vulcanization characteristics with the* oscillating disc curemeter	Nil	
ISO 6502:1991 Rubber — Measurement of vulcanization characteristics with *rotorless curemeters*	Nil	

In the case of ISO 3417 : 1991 *and* ISO 6502 : 1991, *the Technical Committee responsible for the* preparation of this standard has reviewed their contents and has decided that they are acceptable for use in conjunction with this standard.

For tropical countries like India, the standard temperature and the relative humidity shall be taken as 27 ± 2°C and 65 ± 5 percent respectively.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 14 (1873).

Amendments Issued Since Publication

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