

INTERNATIONAL STANDARD

ISO
2475

Second edition
1987-07-15



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

Rubber, chloroprene (CR) — General purpose types — Evaluation procedures

Caoutchouc chloroprène (CR) — Types à usage général — Méthodes d'évaluation

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Reference number
ISO 2475: 1987 (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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2475 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 2475 : 1975), of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Rubber, chloroprene (CR) — General purpose types — Evaluation procedures

1 Scope and field of application

This International Standard specifies

- physical and chemical tests on raw rubbers;
- standard materials, equipment and processing methods for evaluating vulcanization properties of raw general purpose chloroprene rubbers (CR).

The general purpose chloroprene rubbers fall into two broad classes based on the type of polymerization modifier used in their preparation :

- a) sulfur-modified types;
- b) mercaptan-modified types;
- c) types modified by other products. For class c) the use of either test recipe from clause 5 or clause 6 is possible.

2 References

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

ISO 247, *Rubber — Determination of ash.*

ISO 248, *Rubbers, raw — Determination of volatile matter content.*

ISO 289, *Rubber, unvulcanized — Determination of Mooney viscosity.*

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

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 1796, *Rubber, raw — Sample preparation.*

ISO 2058, *Raw styrene-butadiene rubber (SBR) — Determination of volatile matter.*

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 Sampling and sample preparation

3.1 A sample of mass approximately 1 500 g shall be taken by the method described in ISO 1795.

3.2 Sample preparation shall be in accordance with ISO 1796.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity on a portion from the original sample according to ISO 289, and record the result as ML (1 + 4) at 100 °C.

4.2 Volatile matter

Determine the volatile matter by the hot-mill method specified in clause 3 of ISO 2058, but using a mill roll temperature of 50 ± 5 °C.

The use of the oven method of ISO 248 is also permissible.

4.3 Ash

Determine the ash by an ashing procedure in accordance with ISO 247.

5 Test procedure for evaluation of sulfur-modified chloroprene rubbers

5.1 Standard test formula

The standard test formula is given in table 1.

The materials shall be NBS¹⁾ Standard reference materials as indicated in table 1, or shall be in accordance with equivalent national standards.

Table 1

Material	NBS Standard reference material number	Parts by mass
Chloroprene rubber (CR), sulfur modified	—	100,00
Stearic acid	372	0,50
Magnesium oxide	376	4,00
SRF black, low modulus	382	30,00
Zinc oxide	370	5,00
Total		139,50

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.

5.2.2 Premastication

5.2.2.1 Weigh out 500 g of chloroprene rubber.

5.2.2.2 Adjust the mill-roll temperature to 50 ± 5 °C.

5.2.2.3 Band the rubber with a mill opening of 1,5 mm and take the time from the instant the rubber is banded.

5.2.2.4 Adjust the nip to maintain a rolling bank of approximately 12 mm. Mill the rubber for 6 min, cutting as necessary to maintain a rolling bank and a tight band.

5.2.2.5 Remove the rubber from the mill and allow it to cool to room temperature prior to mixing.

5.2.3 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 50 ± 5 °C throughout the mixing.

NOTE — All mill openings shall be adjusted to maintain a good rolling bank at the nip of the rolls during mixing. If this is not obtained with the settings specified hereunder, small adjustments to mill openings may be necessary.

	Duration (min)
5.2.3.1 Band the premasticated rubber on the mill with a nip setting of 1,5 mm or a suitable setting to maintain a rolling bank	1
5.2.3.2 Add the stearic acid	1
5.2.3.3 Add the magnesium oxide slowly, spreading it evenly over the entire width of the band. Ensure complete incorporation before adding the SRF black	2
5.2.3.4 Add the SRF black. Open the nip at intervals to maintain a rolling bank	5
5.2.3.5 Add the zinc oxide	2
5.2.3.6 Make three 3/4 cuts from each side	3
5.2.3.7 Cut the batch from the mill. Set the nip at 0,8 mm and pass the rolled batch lengthways through the mill six times	2
Total time	16

5.2.3.8 Sheet the batch to approximately 6 mm and check weigh. Remove sufficient material for testing with the oscillating disc curemeter.

5.2.3.9 Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

5.2.3.10 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing, at a standard laboratory temperature defined in ISO 471.

6 Test procedure for mercaptan-modified chloroprene rubbers

6.1 Standard test formula

The standard test formula is given in table 2.

The materials shall be NBS¹⁾ Standard reference materials as indicated in table 2, or shall be in accordance with equivalent national standards.

1) National Bureau of Standards of the USA.

Table 2

Material	NBS Standard reference material number	Parts by mass
Chloroprene rubber (CR), mercaptan modified	—	100,00
Stearic acid	372	0,50
Magnesium oxide	376	4,00
SRF black, low modulus	38	30,00
Zinc oxide	370	5,00
ETU (as master batch)	—	0,50*
		Total **

* Parts by mass refer to pure ETU. Therefore, the parts by mass must be adjusted according to the amount of binder reported by the supplier.

The results may depend on the particular ETU batch used. Where interlaboratory tests are concerned, the ETU batch should be distributed to the participants, or the participants should agree to obtain it from a single supplier.

** The total will be indicated as 140 + x (x being the number of parts by mass of the binder).

6.2 Procedure

6.2.1 Equipment and procedure

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

6.2.2 Premastication

6.2.2.1 Weigh out 500 g of chloroprene rubber.

6.2.2.2 Adjust the mill-roll temperature to 50 ± 5 °C.

6.2.2.3 Band the rubber with a mill opening of 1,5 mm and take the time from the instant the rubber is banded.

6.2.2.4 Adjust the nip to maintain a rolling bank of approximately 12 mm. Mill the rubber for 6 min, cutting as necessary to maintain a rolling bank and a tight band.

6.2.2.5 Remove the rubber from the mill and allow it to cool to room temperature prior to mixing.

6.2.3 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 50 ± 5 °C throughout the mixing.

NOTE — All mill openings shall be adjusted to maintain a good rolling bank at the nip of the rolls during mixing. If this is not obtained with the settings specified hereunder, small adjustments to mill openings may be necessary.

	Duration (min)
6.2.3.1 Band the premasticated rubber on the mill with a nip setting of 1,5 mm or a suitable setting to maintain a rolling bank	1
6.2.3.2 Add the stearic acid	1
6.2.3.3 Add the magnesium oxide slowly, spreading it evenly over the entire width of the band. Ensure complete incorporation before adding the SRF black	2
6.2.3.4 Add the SRF black. Open the nip at intervals to maintain a rolling blank	5
6.2.3.5 Add the zinc oxide	2
6.2.3.6 Add the ETU master batch	1
6.2.3.7 Make three 3/4 cuts from each side	3
6.2.3.8 Cut the batch from the mill. Set the mill opening at 0,8 mm and pass the rolled batch lengthways through the mill 6 times	2
Total time	17

6.2.3.9 Sheet the batch to approximately 6 mm and check weigh. Remove sufficient material for testing with the oscillating disc curemeter.

6.2.3.10 Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

6.2.3.11 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing, at a standard laboratory temperature defined in ISO 471.

7 Evaluation of vulcanization characteristics

7.1 Stress-strain properties

Vulcanize sheets at 150 °C for three periods chosen from a cure series of 10; 20; 40 and 60 min.

NOTE — The three periods of cure shall be chosen to cover the undercure, optimum cure and overcure of the material under test.

Condition the vulcanized test slab for 16 to 96 h in a standard laboratory temperature and humidity defined in ISO 471.

Measure the stress-strain properties in accordance with ISO 37.

7.2 Evaluation according to oscillating disc
curemeter test

Measure the following standard test parameters:

M_L , M_H or M_{HR} , 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° arc

selectivity:

to be chosen to give at least 75 %
full scale deflection at M_H or
 M_{HR}

NOTE — With some polymers,
75 % may not be attainable.

die temperature:

160 °C

pre-heat time:

none

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