
INTERNATIONAL STANDARD



2475

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

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

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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 2475 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*, and circulated to the Member Bodies in March 1974.

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

Australia	France	Spain
Belgium	Germany	Sweden
Brazil	Hungary	Thailand
Bulgaria	Italy	Turkey
Canada	Netherlands	United Kingdom
Chile	Poland	U.S.A.
Czechoslovakia	Romania	Yugoslavia

No Member Body expressed disapproval of the document.

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

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies

- a) physical and chemical tests on raw polymer;
- b) 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) sulphur-modified types;
- b) mercaptan-modified types.

2 REFERENCES

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

ISO/R 247, *Determination of ash in raw natural rubber.*

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

ISO 667, *Compounded rubber – Determination of the rate of cure using the shearing disk viscometer.*

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

ISO 1796, *Raw rubber – 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, *Raw rubber – Determination of curing characteristics with the oscillating disc rheometer.*¹⁾

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.

NOTE – No sample preparation is required for most types of chloroprene rubber.

4 PHYSICAL AND CHEMICAL TESTS ON RAW POLYMER

4.1 Mooney viscosity

Determine the Mooney viscosity on a portion from the original sample according to ISO/R 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.

4.3 Ash content

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

1) At present at the stage of draft.

5 TEST RECIPE FOR EVALUATION OF SULPHUR-MODIFIED CHLOROPRENE RUBBERS

5.1 Standard test formula

The standard test formula is given in the following table.

The materials shall be NBS¹⁾ 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
Chloroprene rubber (CR), sulphur modified	—	100,00
Stearic acid	372	0,50
Magnesium oxide	376	4,00
Phenyl β -naphthylamine	377	2,00
SRF black, low modulus	382	30,00
Zinc oxide	370	5,00
		Total 141,50

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 $50 \pm 5^\circ\text{C}$.
- 5.2.2.3 Band the rubber with a mill opening of 1,5 mm and reckon 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 $50 \pm 5^\circ\text{C}$ throughout the mixing.

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

	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 phenyl β -naphthylamine	2
5.2.3.4 Add the phenyl β -naphthylamine	1
5.2.3.5 Add the SRF black. Open the nip at intervals to maintain a rolling bank	5
5.2.3.6 Add the zinc oxide.	2
5.2.3.7 Make three 3/4 cuts from each side.	3
5.2.3.8 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	17
5.2.3.9 Sheet the batch to approximately 6 mm and check weigh. Remove sufficient material for viscometer testing.	
5.2.3.10 Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.	
5.2.3.11 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing.	

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6 TEST RECIPE FOR MERCAPTAN-MODIFIED CHLOROPRENE RUBBERS

6.1 Standard test formula

The standard test formula is given in the following table.

The materials shall be NBS¹⁾ 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
Chloroprene rubber (CR), mercaptan modified	—	100,00
Stearic acid	372	0,50
Magnesium oxide	376	4,00
Phenyl β-naphthylamine	377	2,00
SRF black, low modulus	382	30,00
Zinc oxide	370	5,00
Ethylene thiourea (2-mercaptoimidazolene)	—	0,50
		Total 142,00

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 the mill opening of 1,5 mm and reckon 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.

	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 phenyl β-naphthylamine	2
6.2.3.4 Add the phenyl β-naphthylamine	1
6.2.3.5 Add the SRF black. Open the nip at intervals to maintain a rolling bank	5
6.2.3.6 Add the zinc oxide.	2
6.2.3.7 Add the ethylene thiourea (2-mercaptoimidazolene)	1
6.2.3.8 Make three 3/4 cuts from each side.	3
6.2.3.9 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	18

6.2.3.10 Sheet the batch to approximately 6 mm and check weigh. Remove sufficient material for viscometer testing.

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

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6.2.3.12 Condition the batch for 2 to 24 h after mixing and prior to vulcanizing.

Condition the vulcanized test slab for 16 to 72 h.

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

7 EVALUATION OF VULCANIZATION CHARACTERISTICS

7.2 Viscometer cure

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.

Measure the times t_5 and t_{35} at 120 °C in accordance with ISO/R 667.

The determination shall be made not less than 1 h and not more than 2 h after mixing.

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

NOTE — Curemeter testing in accordance with ISO 3417 may be considered as an alternative method for measuring the vulcanization characteristics.

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