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Methods of test for
**Raw general purpose
chloroprene rubbers**

[ISO title: Rubber, chloroprene (CR) - General purpose types - Evaluation procedures]

Méthodes d'essai des caoutchoucs chloroprènes bruts à usage général

Prüfmethoden für rohe Chloroprenkautschuke zum allgemeinen Gebrauch

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Foreword

This British Standard has been prepared under the authority of the Rubber Industry Standards Committee. It is identical with ISO 2475 'Rubber, chloroprene (CR) – General purpose types – Evaluation procedures'.

For the purposes of this British Standard the text of ISO 2475 given in this publication should be modified as follows.

Terminology. The words 'British Standard' should replace 'International Standard' wherever they appear.

Cross references. The references to other International Standards should be replaced by references to British Standards as follows.

Reference to ISO publication	Appropriate British Standard
ISO/R 37	BS 903 Methods of testing vulcanized rubber Part A2 Determination of tensile stress-strain properties
ISO/R 247	BS 1673 Methods of testing raw rubber and unvulcanized compounded rubber Part 8 Chemical analysis of chloroprene rubber
ISO/R 289	BS 1673 Part 3 Methods of physical testing
ISO 667	BS 1673 Part 3
ISO 1795	BS 1673 Part 1 Sampling and further preparative procedures

ISO 1796	BS 1673 Part 1
ISO 2058	BS 1673 Part 8
ISO 2393	BS 1674 Equipment and procedures for mixing and vulcanizing rubber test mixes
ISO 3417	BS 1673 Part 10 Measurement of pre-vulcanizing and curing characteristics by means of curemeters*

The references in 5.1 and 6.1 to NBS Standard reference materials should similarly be replaced by a reference to BS 4398 'Compounding ingredients for rubber test mixes'.

Text changes

ISO reference	Original ISO text	Text to be substituted
5.2.3.11 and 6.2.3.12	'Condition the batch for 2 to 24 h after mixing...'	'Condition the batch for 2 to 24 h at 23 ± 2 °C after mixing.....'
6.1 (table) and 6.2.3.7	'(2-mercaptoimidazolene)'	'(2-mercaptoimidazoline)'

NOTE. ISO 2475 does not specify the temperature for conditioning the batch and 23 °C has been selected from the ISO preferred temperatures, given in ISO 2393, as appropriate for United Kingdom conditions.

NOTE. This corrects a misprint in ISO 2475.

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British Standard Methods of test for

Raw general purpose chloroprene rubbers

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 ± 5 °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 ± 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)
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 $50 \pm 5^\circ\text{C}$.

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 $50 \pm 5^\circ\text{C}$ throughout the mixing.

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

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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