

INTERNATIONAL STANDARD

ISO
2475

Third edition
1990-12-15

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

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



Reference number
ISO 2475:1990(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 2475 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 2475:1987). The main technical difference in this new edition of ISO 2475 in comparison with the second edition is the inclusion of a new clause giving the required format for a test report (clause 9).

© ISO 1990

All rights reserved. No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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

1 Scope

This International Standard specifies

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

General purpose chloroprene rubbers fall into three 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), either standard test formula 5.1 or 6.1 may be used.

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 37:1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 247:1990, *Rubber — Determination of ash.*

ISO 248:1979, *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 1795:1974, *Raw rubber in bales — Sampling.*

ISO 1796:1982, *Rubber, raw — Sample preparation.*

ISO 2393:1973, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417:1977, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

ISO 6502:1983, *Rubber — Measurement of vulcanization characteristics with rotorless curemeters.*

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 Preparation of the test portion shall be in accordance with ISO 1796.

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

Determine the volatile matter content by the oven method specified in ISO 248.

If the rubber is in a suitable form, which is not the case if it is in chip form, the hot-mill method specified in ISO 248 may also be used, but with a mill roll temperature of $50\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

4.3 Ash content

Determine the ash content in accordance with ISO 247.

5 Preparation of the test mix 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 NIST¹⁾ standard reference materials as indicated in table 1 or other, equivalent, national or international standard reference materials.

Table 1 — Standard test formula for evaluation of sulfur-modified chloroprene rubbers

Material	NIST standard reference material number	Number of parts by mass
Chloroprene rubber (CR), sulfur-modified	—	100,00
Stearic acid	372	0,50
Magnesium oxide ¹⁾	—	4,00
SRF black, low modulus	382	30,00
Zinc oxide	370	5,00
Total		139,50

1) The surface area of the magnesium oxide shall be greater than 125 m²/g.

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

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 in diameter. Mill the rubber for 6 mm, 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 $50\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ throughout the mixing.

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to mill openings may be necessary.

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

Total time 15,0

h) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If

^{*)} National Institute of Standards and Technology (formerly the National Bureau of Standards) of the USA.

the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for curemeter testing.

i) Sheet the batch to an approximate thickness of 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

j) 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 Preparation of the test mix for evaluation of mercaptan-modified chloroprene rubbers

6.1 Standard test formula

The standard test formula is given in table 2.

The materials shall be NIST¹⁾ standard reference materials as indicated in table 2 or other, equivalent, national or international standard reference materials.

Table 2 — Standard test formula for evaluation of mercaptan-modified chloroprene rubbers

Material	NIST standard reference material number	Number of parts by mass
Chloroprene rubber (CR), mercaptan-modified	—	100,00
Stearic acid	372	0,50
Magnesium oxide ¹⁾	—	4,00
SRF black, low modulus	382	30,00
Zinc oxide	370	5,00
ETU (as masterbatch)	—	0,50 + x ²⁾
Total		140,00 + x ²⁾

1) The surface area of the magnesium oxide shall be greater than 125 m²/g.

2) x is the number of parts by mass of the binder. The formulation shall contain 0,50 parts by mass of pure ETU (ethylene thiourea or 2-mercaptoimidazoline). Then x shall be adjusted according to the amount of binder reported by the supplier.

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

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

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

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to mill openings may be necessary.

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

i) Sheet the batch to an approximate thickness of 6 mm and check-weight 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 re-mix. Remove sufficient material for curemeter testing.

j) Sheet the batch to an approximate thickness of 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

k) 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.

7 Evaluation of vulcanization characteristics by a curemeter test

Measure the following standard test parameters:

M_L , M_H at defined time, t_{s1} , $t'_c(50)$ and $t'_c(90)$

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

selectivity: to be chosen to give at least 75 % of full scale deflection at M_H

NOTE 1 With some rubbers, 75 % may not be attainable.

die temperature: 160 °C ± 0,3 °C

pre-heat time: none

8 Evaluation of tensile stress-strain properties of vulcanized test mixes

Vulcanize sheets at 150 °C for three periods chosen from a cure series of 10 min, 20 min, 30 min, 40 min and 60 min. A vulcanization temperature of 160 °C may also be used.

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.

9 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 (mill or oven);
- the time used for measuring M_H in clause 7 ;
- the curemeter test used in clause 7 (ISO 3417 or ISO 6502);
- the vulcanization temperature and times used in clause 8;
- 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;
- the results and the units in which they have been expressed;
- the date of the test.