INTERNATIONAL STANDARD

ISO 2475

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Chloroprene rubber (CR) — General-purpose types — Evaluation procedure

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

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2475 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fifth edition cancels and replaces the fourth edition (ISO 2475:1999), in which:

— the normative references have been updated;

— the precision data (Clause 10) have been moved to informative Annex A.

It also incorporates the Amendment ISO 2475:1999/Amd.1:2005 and the Technical Corrigendum ISO 2475:1999/Cor.1:2001.

Chloroprene rubber (CR) — General-purpose types — Evaluation procedure

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies, for general-purpose chloroprene rubbers (CRs):

— physical and chemical tests on raw rubbers;

— standard materials, standard test formulations, equipment and processing methods for evaluating the vulcanization characteristics.

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), the procedure for either a) or b) may be followed.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 247, *Rubber — Determination of ash*

ISO 248-1:2011, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 289-1, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

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

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

ISO 6502, *Rubber — Guide to the use of curemeters*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Sampling and sample preparation

3.1 Take a sample of mass approximately 1,5 kg by the method described in ISO 1795.

3.2 Prepare the test portion in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1, 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 in accordance with ISO 248-1:2011, either by the oven method or, if the rubber is in a suitable form (which is not the case if it is in chip or powder form), the hot-mill method, but with a mill roll temperature of 50 °C ± 5 °C.

4.3 Ash

Determine the ash in accordance with ISO 247.

5 Sulfur-modified chloroprene rubbers — Preparation of the test mix for evaluation

5.1 Standard test formulation

The standard test formulation is given in Table 1.

The materials shall be national or international standard reference materials, unless no standard reference materials are available, in which case the materials to be used shall be agreed between the interested parties.

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

Material	Parts by mass
Chloroprene rubber (CR), sulfur-modified	100,00
Stearic acid[a]	0,50
Magnesium oxide[b]	4,00
Carbon black[c]	25,00
Zinc oxide[d]	5,00
Total	134,50

[a] See ISO 8312.

[b] The surface area of the magnesium oxide shall be between 130 m^2/g and 200 m^2/g. Magnesium oxide absorbs water and carbon dioxide when exposed to air and this can affect its activity in compounds. Store it in a dry environment.

[c] The current industry reference black (IRB), or an equivalent national or international standard reference material, shall be used.

[d] Class B1a (see ISO 9298:1995, Annex D).

5.2 Procedure

5.2.1 Equipment and procedure

The equipment and procedure for preparation, mixing and vulcanization 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 °C ± 5 °C.

5.2.2.3 Band the rubber with a mill opening of 1,5 mm and start the timer at 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 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 shall be based on four times the recipe mass in grams.

The surface temperature of the rolls shall be maintained at 50 °C ± 5 °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 the mill opening might 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 carbon black	2,0
d)	Add the carbon black. Open the nip at intervals to maintain a rolling bank. Sweep up and add any material which has fallen into the pan	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 the mass of the batch differs from the theoretical value by more than +0,5 %/–1,5 %, 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 in accordance with ISO 37.

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 23529.

6 Mercaptan-modified chloroprene rubbers — Preparation of the test mix for evaluation

6.1 Standard test formulation

The standard test formulation is given in Table 2.

The materials shall be national or international standard reference materials, unless no standard reference materials are available, in which case the materials to be used shall be agreed between the interested parties.

Table 2 — Standard test formulations for evaluation of mercaptan-modified chloroprene rubbers[a]

Material	Formulation A (parts by mass)	Formulation B (parts by mass)
Chloroprene rubber (CR), mercaptan-modified	100,00	100,00
Magnesium oxide[b]	4,00	4,00
Carbon black[c]	25,00	25,00
Zinc oxide[d]	5,00	5,00
MTT 80 in polymeric binder (curative)[e]	0,45	—
Sulfur	—	1,00
Di-ortho-tolyl guanidine (DOTG)[f]	—	1,00
N-cyclohexyl-2-benzothiazyl sulfenamide (CBS)	—	1,00
Total	134,45	137,00

[a] The CR test formulation contains 3-methylthiazolidinethione-2 (MTT) (in the case of formulation A) or sulfur/DOTG/CBS (in the case of formulation B) instead of ethylene thiourea, a suspected carcinogen.

[b] The surface area of the magnesium oxide shall be between 130 m^2/g and 200 m^2/g. Magnesium oxide absorbs water and carbon dioxide when exposed to air and this can affect its activity in compounds. Store it in a dry environment.

[c] The current industry reference black (IRB), or an equivalent national or international standard reference material, shall be used.

[d] Class B1a (see ISO 9298:1995, Annex D).

[e] MTT 80 can be obtained from Rhein Chemie Rheinau GmbH, Mülheimer Str. 24-28, D-68219 Mannheim 81, Germany. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

[f] Under some conditions, DOTG might generate *o*-toluidine emissions classified as carcinogenic by the International Agency for Research on Cancer. Avoid exposure to emissions during curing of mixes.

6.2 Procedure

6.2.1 Equipment and procedure

The equipment and procedure for the preparation, mixing and vulcanization 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 °C ± 5 °C.

6.2.2.3 Band the rubber with a mill opening of 1,5 mm and start the timer at 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 shall be based on four times the recipe mass in grams.

The surface temperature of the rolls shall be maintained at 50 °C ± 5 °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 the mill opening might 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 magnesium oxide slowly, spreading it evenly over the entire width of the band. Ensure complete incorporation before adding the carbon black	2,0
c)	Add the carbon black. Open the nip at intervals to maintain a rolling bank. Sweep up and add any material which has fallen into the pan	5,0
d)	Add the zinc oxide	2,0
e)	If using formulation A, add the MTT 80	1,0
	If using formulation B, add the sulfur, DOTG and CBS	2,0
f)	Make three 3/4 cuts from each side	2,0
g)	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: Formulation A	15,0
	Formulation B	16,0

h) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than +0,5 %/–1,5 %, 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 in accordance with ISO 37.

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 23529.

7 Preparation of the test mix for evaluation of sulfur-modified or mercaptan-modified chloroprene with miniature internal mixer (MIM)

7.1 Standard test formulations

See Table 1 and Table 2.

7.2 Procedure

7.2.1 Equipment and procedure

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

NOTE The procedure applies to both formulations (Table 1 and Table 2).

7.2.2 Mix with the head temperature of the MIM maintained at 60 °C ± 3 °C and the rotor speed at 6,3 rad/s to 6,6 rad/s (60 r/min to 63 r/min).

7.2.3 Premastication

7.2.3.1 Cut the rubber into small pieces, weigh the appropriate amount and load it into the mixing chamber. Lower the ram, start the timer and masticate the rubber for 6 min.

7.2.3.2 Turn off the rotors, raise the ram, remove the mixing chamber and discharge the rubber.

7.2.3.3 Allow to cool to room temperature and weigh prior to mixing.

The standard laboratory batch shall be based on 0,65 times the recipe mass in grams.

7.2.4 Mixing procedure

		Duration (min)
a)	Load the mixing chamber with the rubber, lower the ram and start the timer	0
b)	Masticate the rubber	2
c)	Raise the ram, add the pre-blended powders with the carbon black (and curative for Table 2), taking care to avoid losses. Sweep the orifice, lower the ram and allow the batch to mix	7
	Total time	9

d) Turn off the rotors, raise the ram, open the mixing chamber and discharge the batch.

e) Immediately pass the batch through a laboratory mill with the mill opening set at 0,8 mm and at a temperature of 50 °C ± 5 °C.

f) Pass the rolled batch endwise through the rolls six times.

g) Sheet the batch to approximately 6 mm thickness. Check-weigh 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.

h) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens in accordance with ISO 37.

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 23529.

NOTE Very high Mooney viscosity grades can give difficulties (crumbs) at the discharge of the batch.

8 Evaluation of vulcanization characteristics by a curemeter test

8.1 Using an oscillating-disc curemeter

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, using the following test conditions:

oscillation frequency: 1,7 Hz (100 cycles per minute)

amplitude of oscillation: 1° of arc

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

NOTE With some rubbers, 75 % might not be attainable.

die temperature: 160 °C ± 0,3 °C

pre-heat time: none

8.2 Using a rotorless curemeter

Measure the following standard test parameters:

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

in accordance with ISO 6502, using the following test conditions:

oscillation frequency: 1,7 Hz (100 cycles per minute)

amplitude of oscillation: 0,5° of arc

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

NOTE With some rubbers, 75 % might not be attainable.

die temperature: 160 °C ± 0,3 °C

pre-heat time: none

9 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, in which case it is recommended that the middle cure time be approximately $t'_c(90)$.

Condition the vulcanized sheets for 16 h to 96 h, if possible at standard temperature and humidity as defined in ISO 23529.

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

NOTE For comparison of properties between parties, it will be necessary to use the same conditions.

10 Precision

See Annex A.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

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

c) the procedure used to prepare the standard test formulation;

d) when evaluating mercaptan-modified chloroprene rubbers, whether formulation A or B was used;

e) the reference materials used;

f) the method used to determine the volatile-matter content (mill or oven);

g) the time used to measure M_H in Clause 8;

h) the curemeter method used in Clause 8 (ISO 3417 or ISO 6502);

i) the vulcanization temperature and times used in Clause 9;

j) any unusual features noted during the determination;

k) 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;

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

m) the date of the test.

Annex A
(informative)

Precision statement

A.1 General

The repeatability and reproducibility were calculated in accordance with ISO/TR 9272:1986.

A.2 Precision details

Type 2 interlaboratory precision programmes were conducted for formulation A and formulation B in Table 2, using the mill mixing procedure. Both repeatability and reproducibility are short term, a period of a few days separating replicate test results. A test result is a value, as specified by this test method, obtained for one determination (measurement) of the selected property.

Three different CR formulations were evaluated for precision:

— sulfur-modified CR;

— mercaptan-modified CR (formulation A);

— mercaptan-modified CR (formulation B).

The sulfur-modified CR and mercaptan-modified CR (formulation A) were tested in eight laboratories on two different days.

The mercaptan-modified CR (formulation B) was tested in five laboratories on four different days.

On each of the days, duplicate determinations were made. The estimates of the repeatability parameters therefore contain two undifferentiated sources of variation, i.e. replicates within days and between days.

A.3 Precision results

The final precision parameters are given in Table A.1.

For repeatability, the two test results are obtained with the same method on nominally identical test materials under the same conditions (same operator, apparatus and laboratory) and within a specified time period. Unless stated otherwise, the probability is 95 %.

For reproducibility, the two test results are obtained with the same method on nominally identical test materials under different conditions (different operators, apparatus and laboratories) and within a specified time period. Unless stated otherwise, the probability is 95 %.