Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

IS 15076 (2002): Chloroprene Rubber (CR) - General Purpose Types - Evaluation Procedure [PCD 13: Rubber and Rubber Products]
Indian Standard

CHLOROPRENE RUBBER (CR) — GENERAL PURPOSE TYPES — EVALUATION PROCEDURE

ICS 83.060
NATIONAL FOREWORD

This Indian Standard which is identical with ISO 2475 : 1999 'Chloroprene rubber (CR) — General-purpose types — Evaluation procedure' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Rubber Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been proposed to be approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated:

<table>
<thead>
<tr>
<th>International Standard</th>
<th>Corresponding Indian Standard</th>
<th>Degree of Equivalence</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO 471 : 1995 Rubber — Temperatures, humidities and times for conditioning and testing</td>
<td>IS 13867 : 1993 Rubber standard temperatures, humidities and times for the conditioning and time interval between vulcanization and testing of test pieces</td>
<td>do</td>
</tr>
</tbody>
</table>

(Continued on third cover)
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.

NOTE For class c), the procedure for either a) or b) may be followed.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.


ISO 471:1995, Rubber — Temperatures, humidities and times for conditioning and testing.
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.

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 °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.

1) To be published. (Revision of ISO 1795:1992)
Table 1 — Standard test formulation for evaluation of sulfur-modified chloroprene rubbers

<table>
<thead>
<tr>
<th>Material</th>
<th>Parts by mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloroprene rubber (CR), sulfur-modified</td>
<td>100,00</td>
</tr>
<tr>
<td>Stearic acid (^a)</td>
<td>0,50</td>
</tr>
<tr>
<td>Magnesium oxide (^b)</td>
<td>4,00</td>
</tr>
<tr>
<td>Carbon black (^c)</td>
<td>25,00</td>
</tr>
<tr>
<td>Zinc oxide (^d)</td>
<td>5,00</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>134,50</strong></td>
</tr>
</tbody>
</table>

\(^a\) See ISO 8312.

\(^b\) The surface area of the magnesium oxide shall be lower than 125 m\(^2\)/g.

\(^c\) 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 may be necessary.
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 formulation for evaluation of mercaptan-modified chloroprene rubbers

<table>
<thead>
<tr>
<th>Material</th>
<th>Parts by mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloroprene rubber (CR), mercaptan-modified</td>
<td>100,00</td>
</tr>
<tr>
<td>Magnesium oxide b</td>
<td>4,00</td>
</tr>
<tr>
<td>Carbon black c</td>
<td>25,00</td>
</tr>
<tr>
<td>Zinc oxide d</td>
<td>5,00</td>
</tr>
<tr>
<td>MTT 80 in polymeric binder (curative) e</td>
<td>0,45</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>134,45</strong></td>
</tr>
</tbody>
</table>

a This CR test formulation contains 3-methylthiazolidine-2 (MTT) instead of ethylene thiourea, a suspected carcinogen.

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

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 may be obtained from Rhein Chemie Rheinau GmbH, Mülheimer Str. 24-28, D-68219 Mannheim 81, Germany.

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 may be necessary.
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

<table>
<thead>
<tr>
<th>Duration (min)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>a) Load the mixing chamber with the rubber, lower the ram and start the timer</td>
<td>0</td>
</tr>
<tr>
<td>b) Masticate the rubber</td>
<td>2</td>
</tr>
<tr>
<td>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</td>
<td>7</td>
</tr>
<tr>
<td>d) Turn off the rotors, raise the ram, open the mixing chamber and discharge the batch.</td>
<td></td>
</tr>
<tr>
<td>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.</td>
<td></td>
</tr>
<tr>
<td>f) Pass the rolled batch endwise through the rolls six times.</td>
<td></td>
</tr>
<tr>
<td>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.</td>
<td></td>
</tr>
<tr>
<td>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.</td>
<td></td>
</tr>
<tr>
<td>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 471.</td>
<td></td>
</tr>
</tbody>
</table>

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, \( t_{51} \), \( t_{90} \), \( r_{50}' \) and \( r_{90}' \)

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 % may 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:

\[ F_L, F_H \text{ at defined time, } \tau_5, \tau'_5(50) \text{ and } \tau'_5(90) \]

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

- oscillation frequency: \(1.7 \text{ Hz (100 cycles per minute)}\)
- amplitude of oscillation: \(0.5^\circ \text{ of arc}\)
- selectivity: to be chosen to give at least 75% of full-scale deflection at \(F_H\)
- die temperature: \(160^\circ C \pm 0.3^\circ C\)
- pre-heat time: none

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

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 \(\tau'_5(90)\).

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.

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

10 Precision

10.1 General

The precision results have been taken from ASTM D 3190:1995(3). The repeatability and reproducibility were calculated in accordance with ISO/TR 9272.

10.2 Precision details

A type 2 interlaboratory precision programme was conducted using the mill mix 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. Two different types of CR were evaluated for precision: sulfur-modified CR and mercaptan-modified CR. Each CR was tested in eight laboratories on two different days. On each of the two 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.
10.3 Precision results

The final precision parameters are given in Table 3.

The symbols used in the table are defined as follows:

\[ r = \text{repeatability, in measurement units. This is the value below which the absolute difference between two} \]
\[ \text{"within-laboratory" test results may be expected to lie, with a specified probability.} \]

\[ (r) = \text{repeatability, in per cent (relative).} \]

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%.

\[ R = \text{reproducibility, in measurement units. This is the value below which the absolute difference between two} \]
\[ \text{"between-laboratory" test results may be expected to lie, with a specified probability.} \]

\[ (R) = \text{reproducibility, in per cent (relative).} \]

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%.

Table 3 — Type 2 precision for vulcanization parameters and stress/strain properties \(^a\) of CR

<table>
<thead>
<tr>
<th>Property</th>
<th>Unit</th>
<th>Mean</th>
<th>Within laboratory</th>
<th>Between laboratories</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>( s_r ) ( r ) ( (r) )</td>
<td>( s_R ) ( R ) ( (R) )</td>
</tr>
<tr>
<td>CR — Sulfur grade</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ML</td>
<td>dN-m</td>
<td>5,7</td>
<td>0,28</td>
<td>0,80</td>
</tr>
<tr>
<td>MH</td>
<td>dN-m</td>
<td>53,9</td>
<td>1,03</td>
<td>2,87</td>
</tr>
<tr>
<td>( t_{51} )</td>
<td>min</td>
<td>2,1</td>
<td>0,22</td>
<td>0,61</td>
</tr>
<tr>
<td>( t_{90}(90) )</td>
<td>min</td>
<td>8,6</td>
<td>0,52</td>
<td>1,45</td>
</tr>
<tr>
<td>100 % modulus MPa</td>
<td>MPa</td>
<td>3,0</td>
<td>0,10</td>
<td>0,27</td>
</tr>
<tr>
<td>300 % modulus MPa</td>
<td>MPa</td>
<td>11,8</td>
<td>0,41</td>
<td>1,15</td>
</tr>
<tr>
<td>Tensile strength MPa</td>
<td>MPa</td>
<td>28,1</td>
<td>0,77</td>
<td>2,15</td>
</tr>
<tr>
<td>Elongation %</td>
<td></td>
<td>597</td>
<td>16,65</td>
<td>46,62</td>
</tr>
<tr>
<td>CR — Mercaptan grade</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ML</td>
<td>dN-m</td>
<td>7,6</td>
<td>0,27</td>
<td>0,77</td>
</tr>
<tr>
<td>MH</td>
<td>dN-m</td>
<td>47,5</td>
<td>0,69</td>
<td>1,93</td>
</tr>
<tr>
<td>( t_{51} )</td>
<td>min</td>
<td>2,2</td>
<td>0,10</td>
<td>0,26</td>
</tr>
<tr>
<td>( t_{90}(90) )</td>
<td>min</td>
<td>10,7</td>
<td>0,87</td>
<td>2,43</td>
</tr>
<tr>
<td>100 % modulus MPa</td>
<td>MPa</td>
<td>2,6</td>
<td>0,12</td>
<td>0,34</td>
</tr>
<tr>
<td>300 % modulus MPa</td>
<td>MPa</td>
<td>14,5</td>
<td>0,69</td>
<td>1,94</td>
</tr>
<tr>
<td>Tensile strength MPa</td>
<td>MPa</td>
<td>24,3</td>
<td>1,24</td>
<td>3,48</td>
</tr>
<tr>
<td>Elongation %</td>
<td></td>
<td>441</td>
<td>23,58</td>
<td>66,03</td>
</tr>
</tbody>
</table>

\(^a\) Curing conditions: 160 °C for 15 min.

NOTE: The curemeters used were the oscillating-disc type.
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) the reference materials used;
e) the method used to determine the volatile-matter content (mill or oven);
f) the time used to measure $M_p$ in clause 8;
g) the curemeter method used in clause 8 (ISO 3417 or ISO 6502);
h) the vulcanization temperature and times used in clause 9;
i) any unusual features noted during the determination;
j) 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;
k) the results and the units in which they have been expressed;
l) the date of the test.
Bibliography


(Continued from second cover)

<table>
<thead>
<tr>
<th>International Standard</th>
<th>Corresponding Indian Standard</th>
<th>Degree of Equivalence</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO 3417 : 1991 Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter</td>
<td>Nil</td>
<td>Technically equivalent with minor deviations</td>
</tr>
<tr>
<td>ISO 6502 : 1999 Rubber — Guide to the use of curemeters</td>
<td>Nil</td>
<td>do</td>
</tr>
<tr>
<td>ISO 8313 : 1999 Rubber compounding ingredient — Stearic acid — Definition and test methods</td>
<td>Nil</td>
<td>do</td>
</tr>
<tr>
<td>ISO/TR 9272 : 1986 Rubber and rubber products — Determination of precision for test method standards</td>
<td>Nil</td>
<td>do</td>
</tr>
</tbody>
</table>

In the case of ISO 3417 : 1991 and ISO 6502 : 1999, ISO 8313 : 1999 and ISO/TR 9272 : 1986, the Technical Committee responsible for the preparation of this standard has reviewed their contents and has decided that they are acceptable for use in conjunction with this standard.

For tropical countries like India, the standard temperature and the relative humidity shall be taken as 27 ± 2°C and 65 ± 5 percent respectively.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (revised)’. 
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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of ‘BIS Handbook’ and ‘Standards: Monthly Additions’.

This Indian Standard has been developed from Doc: No. PCD 14(1433).

Amendments Issued Since Publication

<table>
<thead>
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<th>Amend No.</th>
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</thead>
</table>

BUREAU OF INDIAN STANDARDS

Headquarters:
Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 323 01 31, 323 3375, 323 94 02

Regional Offices:
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
           NEW DELHI 110002
           323 76 17, 323 38 41

Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi
          CALCUTTA 700054
          337 84 99, 337 85 61

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022
           337 86 26, 337 91 20

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113
           60 38 43

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
          MUMBAI 400093
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