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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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Contents

	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Sampling and sample preparation	2
5 Physical and chemical tests on raw rubber	2
5.1 Mooney viscosity	2
5.2 Volatile matter	2
5.3 Ash	2
6 Sulfur-modified chloroprene rubbers — Preparation of the test mix for evaluation	2
6.1 Standard test formulation	2
6.2 Procedure	3
6.2.1 Equipment and procedure	3
6.2.2 Premastication	3
6.2.3 Mill mixing procedure	3
7 Mercaptan-modified chloroprene rubbers — Preparation of the test mix for evaluation	4
7.1 Standard test formulation	4
7.2 Procedure	5
7.2.1 Equipment and procedure	5
7.2.2 Premastication	5
7.2.3 Mill mixing procedure	5
8 Sulfur-modified or mercaptan-modified chloroprene with miniature internal mixer (MIM) — Preparation of the test mix for evaluation	6
8.1 Standard test formulations	6
8.2 Procedure	6
8.2.1 Equipment and procedure	6
8.2.2 Premastication	6
8.2.3 Mixing procedure	6
9 Evaluation of vulcanization characteristics by a curemeter test	7
9.1 Using an oscillating-disc curemeter	7
9.2 Using a rotorless curemeter	7
10 Evaluation of tensile stress-strain properties of vulcanized test mixes	8
11 Precision	8
12 Test report	8
Annex A (informative) Precision for carbon black formulations	9
Annex B (informative) Precision for carbon-black free formulations	11
Bibliography	13

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document 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 sixth edition cancels and replaces the fifth edition (ISO 2475:2011), which has been technically revised.

The main changes are as follows:

- normative references have been updated;
- one carbon-black free formulation for sulfur-modified CR has been added in [6.1](#);
- two carbon-black free formulations for mercaptan-modified CR have been added in [7.1](#);
- carbon-black free mill mixing procedures have been added for sulfur-modified CR in [6.2.3](#) and for mercaptan-modified CR in [7.2.3](#);
- precision data for three carbon-black free CR formulations have been added in [Annex B](#).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

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.

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

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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.

1 Scope

This document specifies:

- the physical and chemical tests on raw rubbers;
- the standard materials, the standard test formulations, the equipment and the processing methods to evaluate the vulcanization characteristics

for general-purpose chloroprene rubbers (CRs).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 247-1, *Rubber — Determination of ash — Part 1: Combustion method*

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

ISO 248-2, *Rubber, raw — Determination of volatile-matter content — Part 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit*

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 6502-2, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 2: Oscillating disc curemeter*

ISO 6502-3, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 3: Rotorless curemeter*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Sampling and sample preparation

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

4.2 Prepare the test portion in accordance with ISO 1795.

5 Physical and chemical tests on raw rubber

5.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1, on a test portion prepared as indicated in [4.2](#). Record the result as ML (1 + 4) at 100 °C.

5.2 Volatile matter

Determine the volatile-matter content in accordance with ISO 248-1 or ISO 248-2. The volatile-matter content is determined in accordance with ISO 248-1 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.

5.3 Ash

Determine the ash in accordance with ISO 247-1.

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

6.1 Standard test formulation

The standard test formulations are 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 formulations for evaluation of sulfur-modified chloroprene rubbers

Material	Formulation A parts by mass	Formulation B parts by mass
Chloroprene rubber (CR), sulfur-modified	100,00	100,00
Stearic acid (CAS 57-11-4) ^a	0,50	0,50
Magnesium oxide (CAS 1309-48-4) ^b	4,00	4,00
Carbon black (CAS 1333-86-4) ^c	25,00	—
Zinc oxide (CAS 1314-13-2) ^d	5,00	5,00
Total	134,50	109,50

^a See ISO 8312.

^b The surface area of the magnesium oxide shall be between 130 m²/g and 200 m²/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 9298:2017, Annex D).

6.2 Procedure

6.2.1 Equipment and procedure

The equipment and procedure for 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 $50\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

6.2.2.3 Band the rubber with a mill opening of 1,5 mm and 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 $50\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ 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 the mill opening can be necessary.

	Duration min	Formulation A	Formulation B
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	1,0	1,0
b) Add the stearic acid.	1,0	1,0	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	2,0	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	2,0	2,0
f) Make three 3/4 cuts from each side.	2,0	2,0	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	2,0	2,0
	Total time	15,0	10,0

- h) 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 % or -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 and ISO dumbbell specimens (as defined in ISO 37) or to the appropriate thickness for preparing ISO dumbbell or ring specimens in accordance with ISO 37.
- j) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, at the standard temperature and humidity as defined in ISO 23529 if possible.

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

7.1 Standard test formulation

The standard test formulations are 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

Material	Formulation C parts by mass	Formulation D parts by mass	Formulation E parts by mass	Formulation F parts by mass
Chloroprene rubber (CR), mercaptan-modified	100,00	100,00	100,00	100,00
Magnesium oxide (CAS 1309-48-4) ^a	4,00	4,00	4,00	4,00
Carbon black (CAS 1333-86-4) ^b	25,00	—	25,00	—
Zinc oxide (CAS 1314-13-2) ^c	5,00	5,00	5,00	5,00
MTT 80 in polymeric binder (curative) (CAS 1908-87-8) ^d	0,45	0,45	—	—
Sulfur (CAS 7704-34-9)	—	—	1,00	1,00
Di-ortho-tolyl guanidine (DOTG) (CAS 97-39-2) ^e	—	—	1,00	1,00
<i>N</i> -cyclohexyl-2-benzothiazyl sulfenamide (CBS) (CAS 95-33-0)	—	—	1,00	1,00
Total	134,45	109,45	137,00	112,00

^a The surface area of the magnesium oxide shall be between 130 m²/g and 200 m²/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.

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

^c Class B1a (see ISO 9298:2017, Annex D).

^d MTT 80 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

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

NOTE The CR test formulations contain 3-methylthiazolidinethione-2 (MTT) (in the case of Formulation C and Formulation D) or sulfur/DOTG/CBS (in the case of Formulation E and Formulation F) instead of ethylene thiourea, a suspected carcinogen.

7.2 Procedure

7.2.1 Equipment and procedure

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

7.2.2 Premastication

7.2.2.1 Weigh out 500 g of chloroprene rubber.

7.2.2.2 Adjust the mill-roll temperature to $50\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

7.2.2.3 Band the rubber with a mill opening of 1,5 mm and start the timer at the instant the rubber is banded.

7.2.2.4 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.

7.2.2.5 Remove the rubber from the mill and allow it to cool to room temperature prior to mixing.

7.2.3 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 $50\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ 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 the mill opening can be necessary.

		Duration min	Carbon black	Carbon- black free
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	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	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	2,0	
e)	If using Formulation C or Formulation D, add the MTT 80.	1,0	1,0	

NOTE MTT 80 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

If using Formulation E or Formulation F, add the sulfur, DOTG and CBS.	2,0	2,0
f) Make three 3/4 cuts from each side.	2,0	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	2,0

Total time:

Formulation C or Formulation D	15,0	10,0
Formulation E or Formulation F	16,0	11,0

- 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 and ISO dumbbell specimens or to the appropriate thickness for preparing ISO dumbbell or ring specimens in accordance with ISO 37.
- j) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, at the standard temperature and humidity as defined in ISO 23529 if possible.

8 Sulfur-modified or mercaptan-modified chloroprene with miniature internal mixer (MIM) — Preparation of the test mix for evaluation

8.1 Standard test formulations

See Formulation A given in [Table 1](#), Formulation C and Formulation E given in [Table 2](#).

8.2 Procedure

8.2.1 Equipment and procedure

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

NOTE The procedure applies to Formulation A, Formulation C and Formulation E ([Table 1](#) and [Table 2](#)).

Mix with the head temperature of the MIM maintained at $60\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ and the rotor speed at 6,3 rad/s to 6,6 rad/s (60 r/min to 63 r/min).

8.2.2 Premastication

8.2.2.1 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.

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

8.2.2.3 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.

8.2.3 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 $50\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

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 an approximate thickness of 2,2 mm for preparing test slabs and ISO dumbbell specimens or to the appropriate thickness for preparing ISO dumbbell or ring specimens in accordance with ISO 37.

i) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, at the standard temperature and humidity as defined in ISO 23529 if possible.

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

9 Evaluation of vulcanization characteristics by a curemeter test

9.1 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 6502-2, 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 at M_H ;

NOTE With some rubbers, 75 % can be unattainable.

- die temperature: $160\text{ }^{\circ}\text{C} \pm 0,3\text{ }^{\circ}\text{C}$;
- pre-heat time: none.

9.2 Using a rotorless 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 6502-3, 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 M_H ;
NOTE With some rubbers, 75 % can be unattainable.
- die temperature: 160 °C ± 0,3 °C;
- pre-heat time: none.

10 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 the middle cure time should be approximately $t'_c(90)$.

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

Measure the stress-strain properties using ISO dumbbell or ring specimens in accordance with ISO 37.

To compare properties between parties, the same conditions shall be used.

11 Precision

See [Annexes A](#) and [B](#).

12 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 2475:2024;
- b) all details necessary for the identification of the sample;
- c) the method used to determine the volatile-matter content (ISO 248-1 or ISO 248-2);
- d) the standard test formulation used;
- e) the reference materials used;
- f) the mixing procedure used;
- g) the conditioning environment used in [6.2.3 j](#)) or [7.2.2 j](#)) or [8.2.3 i](#)) and in [Clause 10](#);
- h) the curemeter method used in [Clause 9](#) (ISO 6502-2 or ISO 6502-3);
- i) the time used to measure M_H in [Clause 9](#);
- j) the vulcanization temperature and times used in [Clause 10](#);
- k) any unusual features noted during the determination;
- l) any operation not included in this document or in the International Standards to which reference is made, as well as any operation regarded as optional;
- m) the results and the units in which they have been expressed;
- n) the date of the test.

Annex A (informative)

Precision for carbon black formulations

A.1 General

The repeatability and reproducibility were calculated according to ISO/TR 9272:1986¹⁾.

A.2 Precision details

Type 2 interlaboratory precision programmes were conducted for Formulation C and Formulation E 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 (Formulation A);
- mercaptan-modified CR (Formulation C);
- mercaptan-modified CR (Formulation E).

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

The mercaptan-modified CR (Formulation E) 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 %.

1) Withdrawn.

Table A.1 — Precision results

Property	Unit	Mean	Intralaboratory comparison			Interlaboratory comparison		
			s_r	r	(r)	s_R	R	(R)
CR — Sulfur grade (Formulation A)^a								
M_L	dN·m	5,7	0,28	0,80	14,0	1,16	3,24	56,7
M_H	dN·m	53,9	1,03	2,87	5,3	2,97	8,32	15,4
t_{s1}	min	2,1	0,22	0,61	28,6	0,51	1,43	66,7
$t'_c(90)$	min	8,6	0,52	1,45	16,8	1,36	3,81	44,1
100 % modulus	MPa	3,0	0,10	0,27	9,0	0,17	0,48	16,1
300 % modulus	MPa	11,8	0,41	1,15	9,8	0,60	1,67	14,2
Tensile strength	MPa	26,1	0,77	2,15	8,3	1,66	4,65	17,8
Elongation	%	597	16,65	46,62	7,8	32,00	89,60	15,0
CR — Mercaptan grade (Formulation C)^a								
M_L	dN·m	7,6	0,27	0,77	10,1	1,02	2,87	37,9
M_H	dN·m	47,5	0,69	1,93	4,1	3,31	9,27	19,5
t_{s1}	min	2,2	0,10	0,28	12,9	0,32	0,89	41,1
$t'_c(90)$	min	10,7	0,87	2,43	24,6	2,47	6,91	69,8
100 % modulus	MPa	2,6	0,12	0,34	13,2	0,24	0,67	25,6
300 % modulus	MPa	14,5	0,69	1,94	13,4	1,18	3,31	22,8
Tensile strength	MPa	24,3	1,24	3,48	14,2	1,51	4,23	17,4
Elongation	%	441	23,58	66,03	15,0	34,17	95,67	21,7
CR — Mercaptan grade (Formulation E)^b								
M_L	dN·m	1,7	0,06	0,16	9,3	0,23	0,65	37,7
M_H	dN·m	27,8	0,40	1,14	4,1	2,55	7,23	26,0
t_{s1}	min	2,2	0,11	0,31	14,2	0,28	0,80	36,6
$t'_c(90)$	min	35,7	1,18	3,34	9,4	4,32	12,23	34,3
100 % modulus	MPa	3,5	0,26	0,75	21,4	0,50	1,42	40,8
300 % modulus	MPa	15,4	1,09	3,09	20,1	1,81	5,13	33,3
Tensile strength	MPa	26,5	0,98	2,76	10,4	1,74	4,93	18,6
Elongation	%	479	28,18	79,74	16,7	49,27	139,45	29,1
Key								
s_r = repeatability standard deviation, in measurement units.								
r = repeatability, in measurement units; this is the value below which the absolute difference between two intralaboratory test results can be expected to lie, with a specified probability.								
(r) = repeatability, in percent (relative).								
s_R = reproducibility standard deviation, in measurement units.								
R = reproducibility, in measurement units; this is the value below which the absolute difference between two interlaboratory test results can be expected to lie, with a specified probability.								
(R) = reproducibility, in percent (relative).								
^a Curing conditions: 160 °C for 15 min. The curemeter used was the oscillating-disc type.								
^b Curing conditions: 160 °C for 20 min. The curemeter used was the rotorless type.								