
**Rubber, unvulcanized —
Determinations using a shearing-
disc viscometer —**

**Part 2:
Determination of pre-vulcanization
characteristics**

*Caoutchouc non vulcanisé — Déterminations utilisant un
consistomètre à disque de cisaillement —*

Partie 2: Détermination des caractéristiques de prévulcanisation



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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.

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 documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 289-2:1994), which has been technically revised. The following changes have been incorporated:

- a calibration schedule has been added in [Annex A](#);
- a precision clause has been moved to [Annex B](#);
- the introductory statements concerning safety and environmental caution have been added;
- the layout of the test report clause has been changed in accordance with ISO/TC 45/SC 2 internal agreements.

ISO 289 consists of the following parts, under the general title *Rubber, unvulcanized — Determinations using a shearing-disc viscometer*:

- *Part 1: Determination of Mooney viscosity*
- *Part 2: Determination of pre-vulcanization characteristics*
- *Part 3: Determination of the Delta Mooney value for non-pigmented, oil-extended emulsion-polymerized SBR*
- *Part 4: Determination of the Mooney stress-relaxation rate*

Rubber, unvulcanized — Determinations using a shearing-disc viscometer —

Part 2:

Determination of pre-vulcanization characteristics

WARNING 1 — Persons using this part of ISO 289 should be familiar with normal laboratory practice. This part of ISO 289 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.

WARNING 2 — Certain procedures specified in this part of ISO 289 might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This part of ISO 289 specifies a method for determining the pre-vulcanization characteristics of compounded rubber.

The pre-vulcanization characteristics determined by this method provide a means of estimating how long compounded rubber can be maintained at high temperatures and remain processable.

2 Normative references

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

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

ISO 18899, *Rubber — Guide to the calibration of test equipment*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

pre-vulcanization time scorch time

time including warm-up time, for the viscosity to increase by a specified amount from the minimum value

Note 1 to entry: Expressed in minutes.

4 Principle

The test consists of determining how the Mooney viscosity of the rubber compound changes with running time at a specified temperature relevant to the process for which the compound is to be used. The time at which the Mooney viscosity has increased by a specified number of units is recorded.

5 Apparatus

The apparatus specified in ISO 289-1 shall be used. It is permissible to use the small rotor for high-viscosity compounds.

6 Calibration schedule

See [Annex A](#).

7 Preparation of test specimen

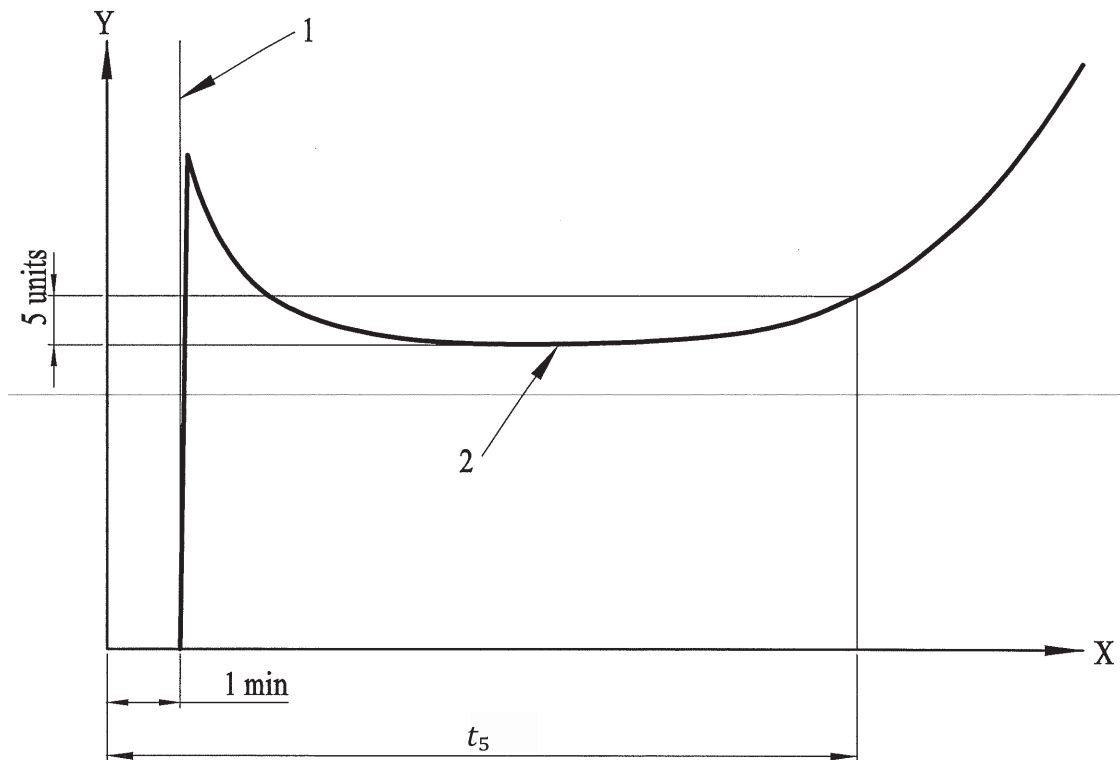
Prepare the two discs comprising the test specimen from a sheet of rubber compound, using the procedure for preparation of test pieces described in ISO 289-1.

8 Test temperature

Choose a test temperature relevant to the process for which the compound is to be used.

9 Procedure

Use the procedure described in ISO 289-1. The pre-heating time shall be 1 min, and the test shall be continued until the viscosity reaches the specified number of units above the minimum. When a large rotor is used, the increase is specified as five units and when a small rotor is used, the increase is specified as three units. The corresponding pre-vulcanization times are designated t_5 and t_3 , respectively. A typical trace obtained with the large rotor is shown in [Figure 1](#).

**Key**

- 1 rotor started
- 2 minimum viscosity value
- X time (in minutes)
- Y Mooney viscosity (in Mooney units)

**Figure 1 — Determination of the pre-vulcanization or scorch time using the large rotor
(increase in viscosity = 5 units)**

10 Precision

See [Annex B](#).

11 Test report

The test report shall include the following information:

- a) sample details:
 - 1) full description of the sample and its origin;
 - 2) details of compounded rubbers, if applicable;
- b) details of the preparation of the test pieces,
- c) a reference to this part of ISO 289, i.e. ISO 289-2;
- d) a description of the apparatus used, including the model used, the manufacturer of the apparatus and the rotor size (large or small),

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- e) test details:
 - 1) the test temperature;
 - 2) the die-closing force, if other than 11,5 kN;
 - 3) details of any procedures not specified in this part of ISO 289;
- f) test results:
 - 1) the minimum viscosity, in Mooney units;
 - 2) the pre-vulcanization or scorch time (t_5 or t_3), in minutes;
- g) date(s) of test.

Annex A **(normative)**

Calibration schedule

A.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration was carried out in the “as-received” condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

A.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. However, the frequency of calibration and procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule is given in ISO 289-1.

Annex B (informative)

Precision results from an interlaboratory test programme

B.1 General

The following interlaboratory test programme (ITP) was initially carried out in 1987.

All calculations to provide repeatability and reproducibility values were performed in accordance with ISO/TR 9272^[1]. Precision concepts and nomenclature are also given in ISO/TR 9272.

B.2 Programme details

Duplicate test pieces of the following compounded rubbers were sent to all participating laboratories: chloroprene (CR), EPDM (highly loaded), fluororubber (FKM) and SBR 1500 including 50 parts per hundred (mass of rubber) N550 black¹⁾.

Pre-vulcanization characteristic determinations (single measurement) were made on two separate days (one week apart), one determination on each day. The test conditions were as follows: for CR and EPDM compounds 120 °C and the small rotor; for FKM compound 150 °C and the large rotor; for SBR compound 170 °C and the small rotor. A total of 16 laboratories participated.

This ITP corresponds to a type 1 precision evaluation, with no preparation or processing steps in the participating laboratories.

B.3 Precision results

B.3.1 The precision results are given in [Table B.1](#).

1) Designation according to ASTM D1765-89, *Standard classification System for carbon blacks used in rubber products*.

Table B.1 — Precision of pre-vulcanization characteristic determinations

Rubber material	Average	Within lab		Between lab	
		<i>r</i>	(<i>r</i>)	<i>R</i>	(<i>R</i>)
Minimum viscosity (Mooney torque units)					
SBR	22,0	1,03	4,70	3,06	13,9
CR	22,3	1,28	5,75	4,96	22,2
FKM	46,1	2,81	6,11	7,20	15,6
EPDM	60,3	1,94	3,23	11,10	18,4
Pooled values	37,7	1,88	4,99	7,23	19,2
Pre-vulcanization time (min)					
SBR	5,23	0,34	6,41	2,55	48,8
CR	14,80	1,82	12,30	7,55	50,9
FKM	8,97	1,27	14,20	3,88	43,3
EPDM	20,80	5,32	25,50	11,60	55,5
Pooled values	12,50	2,89	23,10	7,28	58,1

B.3.2 The symbols used in [Table B.1](#) are defined as follows:

- r* repeatability (in Mooney units);
- (*r*) repeatability (in percent of mean level);
- R* reproducibility (in Mooney units);
- (*R*) reproducibility (in percent of mean level).

Bibliography

- [1] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

