

INTERNATIONAL STANDARD

ISO
289-2

First edition
1994-07-01

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



Reference number
ISO 289-2:1994(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 289-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

It cancels and replaces the second edition of ISO 667 (ISO 667:1981), of which it constitutes a technical revision.

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*

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

Part 2:

Determination of pre-vulcanization characteristics

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.

NOTE 1 No method of test can be expected to correlate with all the different types of processing conditions such as are found in mixing, calendaring, extrusion and moulding. Therefore it is necessary to consider previous experience with a particular process when interpreting the results.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 289. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 289 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 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*.

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards*.

3 Definition

For the purposes of this part of ISO 289, the following definition applies.

3.1 pre-vulcanization time; scorch time: The time in minutes, including warm-up time, for the viscosity to increase by a specified amount from the minimum value. 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.

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 Preparation of test specimen

Prepare the two discs comprising the test specimen from a sheet of rubber compound, using the pro-

cedure for preparation of test pieces described in ISO 289-1.

7 Test temperature

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

8 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. A typical trace obtained with the large rotor is shown in figure 1.

9 Precision

9.1 General

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. Consult this for precision concepts and nomenclature. ISO 289-1:1994, annex A, gives guidance on the use of repeatability and reproducibility.

9.2 Programme details

An interlaboratory test programme (ITP) was organized in 1987. Duplicate test pieces of the following compounded rubbers were sent to all participating laboratories: chloroprene (CR), EPDM (highly loaded), fluororubber (FKM) and SBR 1500 + 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.

9.3 Precision results

9.3.1 The precision results are given in table 1.

9.3.2 The symbols used in table 1 are defined as follows:

- r = repeatability, in Mooney units
- (r) = repeatability, in percent (relative)
- R = reproducibility, in Mooney units
- (R) = reproducibility, in percent (relative)

10 Test report

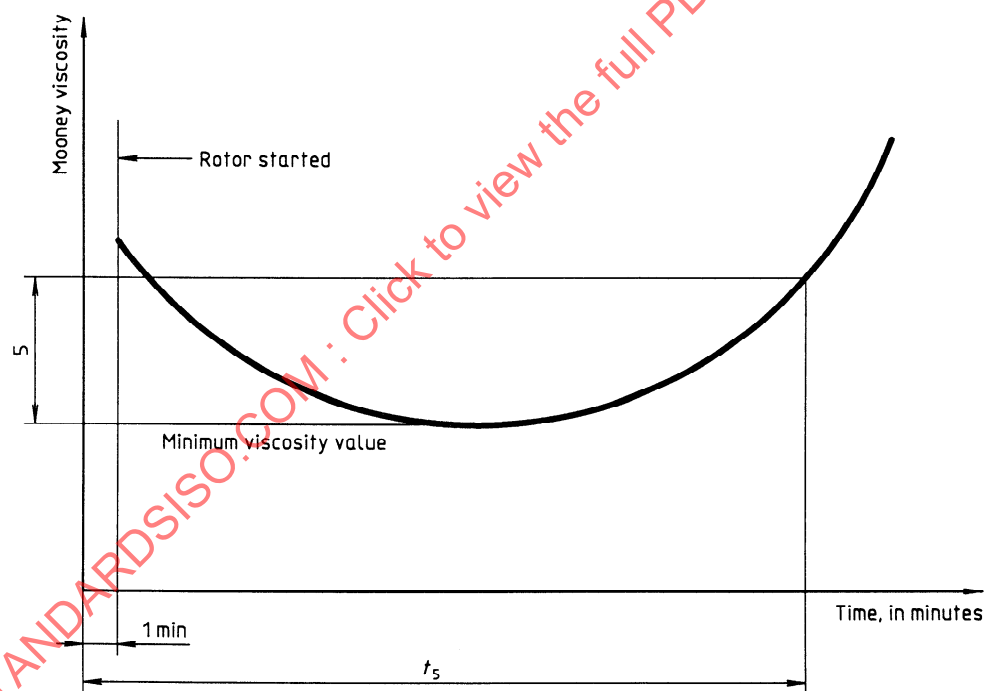
The test report shall include the following information:

- a) a full description and identification of the rubber compound tested, including its origin;
- b) a reference to this part of ISO 289;
- c) a description of the apparatus used, including
 - 1) the model used, and the manufacturer of the apparatus,
 - 2) the rotor size (large or small);
- d) the test temperature;
- e) the minimum viscosity, in Mooney units;
- f) the pre-vulcanization or scorch time (t_5 or t_3), in minutes;
- g) the date of the test.

1) Designation according to ASTM D 1765-89, *Standard classification system for carbon blacks used in rubber products*.

Table 1 — Precision of pre-vulcanization characteristic determinations

Rubber material	Average	Within lab		Between labs	
		r	(r)	R	(R)
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

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

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