INTERNATIONAL STANDARD

ISO 289-2

Third edition
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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 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 289-2:2016), which has been technically revised.

The main change compared to the previous edition is that the precision statement in Annex B has been updated after a new ITP was performed.

A list of all parts in the ISO 289 series can be found on the ISO website.

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.

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

Part 2:
Determination of pre-vulcanization characteristics

WARNING 1 — 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 and to determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document 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 document 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 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 289-1, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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: It is 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

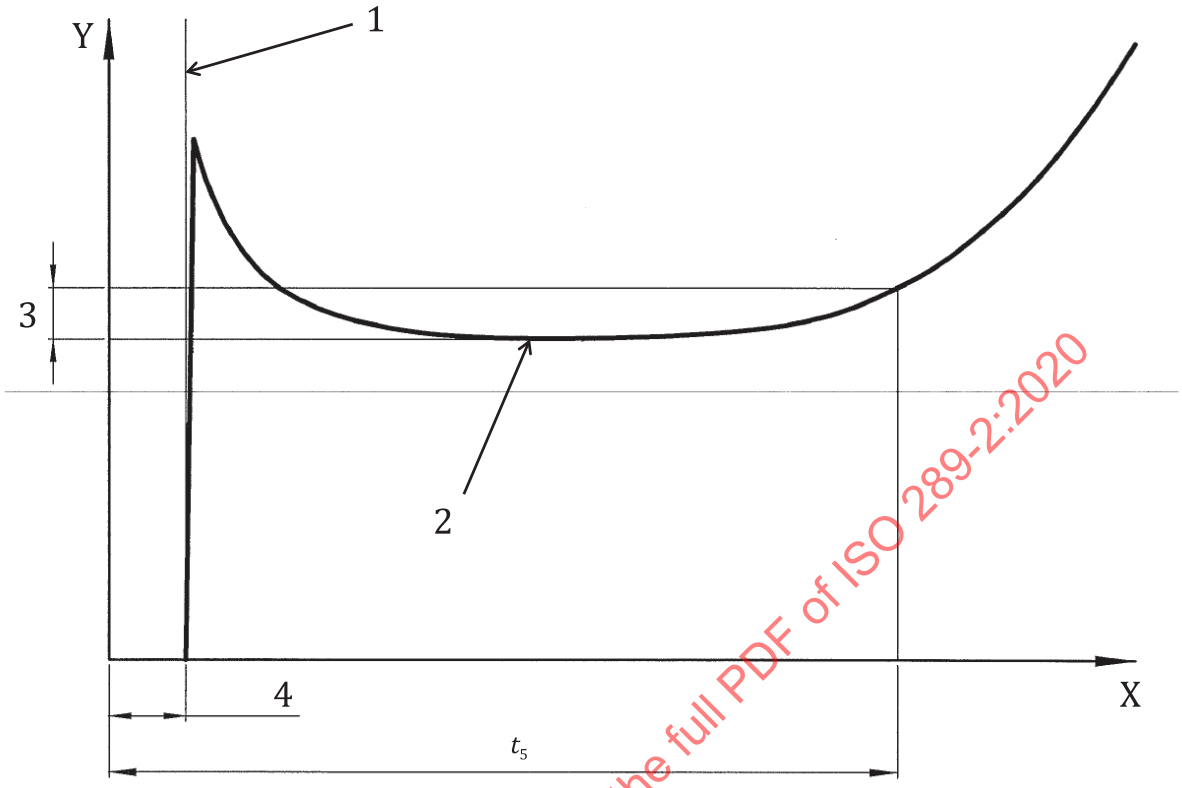
Prepare the two discs comprising the test specimen from a sheet of rubber compound, using the procedure for preparation of test pieces described in ISO 289-1.

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

X time, in minutes
Y Mooney viscosity, in Mooney units
1 rotor started
2 minimum viscosity value
3 5 units
4 1 min

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 document, i.e. ISO 289-2:2020;

d) a description of the apparatus used, including the model used, the manufacturer of the apparatus and the rotor size (large or small),

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 document;

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
(informative)

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 document. 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

B.1 General

The following interlaboratory test programme (ITP) was carried out in 2018.

All calculations to provide repeatability, day-to-day repeatability and reproducibility values were performed in accordance with ISO 19983. Precision concepts and nomenclature are also given in ISO 19983.

B.2 Precision results from the ITP

B.2.1 Programme details

The ITP was organized and conducted by Hari Shankar Singhania Elastomer and Tyre Research Institute (HASETRI) in 2018. Compounds were prepared by HASETRI and sent to all 16 participating laboratories.

A total of three compounds were used in the test, i.e. SBR 1502, NBR and EPDM. The samples were designated as sample A, sample B and sample C. For the details of the compounding of the materials, see Table B.1.

Table B.1 — Compounding

Ingredient	Number of parts by mass		
	Sample A	Sample B	Sample C
SBR (1502)	100		
NBR (ACN content 31 % – 35 %)		100	
EPDM			100
HAF carbon black (N 330)	62		
FEF carbon black (N 550)		40	70
Zinc oxide_ white seal		3	3
Zinc oxide_ rubber grade	4		
Stearic acid	2	1	1
Antioxidant (6PPD)[a]	2		
Paraffinic oil			7
Naphthenic oil		4	
Accelerator (MBTS)[b]			0,5
Accelerator (TMTD)[c]			1,5

[a] N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine.

[b] Dibenzothiazole disulfide.

[c] Tetramethylthiuram disulfide.

[d] N-cyclohexyl-2-benzothiazole sulfenamide.

[e] N- tert-2-butyl-benzothiazole sulfenamide.

Table B.1 *(continued)*

Ingredient	Number of parts by mass		
	Sample A	Sample B	Sample C
Accelerator (CBS)[d]		2	
Accelerator (TBBS)[e]	1		
Sulfur (soluble)		1,5	1,5
Sulfur (insoluble)	1,75		
Total	172,75	151,5	184,5

[a] N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine.

[b] Dibenzothiazole disulfide.

[c] Tetramethylthiuram disulfide.

[d] N-cyclohexyl-2-benzothiazole sulfenamide.

[e] N- tert-2-butyl-benzothiazole sulfenamide.

The test pieces were provided as discs with a diameter of 50,0 mm and thickness of 6 mm. Conditioning and the measurement of the test samples were in accordance with this document. Two test pieces for each sample material for week 1 and 2 were used. An overview of the test program is found in Table B.2.

Table B.2 — Overview of the test programme

Test method	Compound	Test condition °C/min
ISO 289-2	Sample A	MS135 Test 1: 135 °C/60 min
		MS115 Test 2: 115 °C/90 min
	Sample B	MS135 Test 1: 135 °C/60 min
		MS115 Test 2: 115 °C/90 min
	Sample C	MS135 Test 1: 135 °C/60 min
		MS115 Test 2: 115 °C/90 min

The number of laboratories on which precision data for each property is based is given in the tables of precision results (Tables B.3 and B.4). The number of participating laboratories as noted in these tables is the final number after identifying certain laboratory values as outliers.

The ITP testing was conducted over a period of two sequential weeks. On a specified day in each of these two weeks, two individual measurements were performed on all three materials. The test result of each week is the two individual measurements. All analysis was conducted on the basis of these test results.

The participating laboratories were encouraged to use single competent operator for this ITP: The aim of the use of different test pieces and the repetition over two test weeks was to include such normal variation sources in the final or pooled combined database. Thus, the precision values represent more reliable and realistic values compared to the usual ITP results which constitute a "single point in time" estimate of precision.