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

Part 4:
**Determination of the Mooney
stress-relaxation rate**

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

Partie 4: Détermination du taux de relaxation de contrainte Mooney



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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.

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The main task of technical committees is to prepare International Standards. 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.

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.

ISO 289-4 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analyses*.

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*

In this corrected version of ISO 289-4:2003, the following changes have been incorporated:

- page iv: corrected spelling of the TC title;
- pages v, 1, 2, 3, 6, 7: modifications of bibliographic reference numbers;
- page 2: addition of the word “thickness” to NOTE 2;
- page 3: change of symbol for Mooney units – from M to T ;
- page 4: Figure 1, addition of symbol t , to the key for the abscissa;
- page 6: subclause A.2.1, new text;
- page 8: Table A.1, deletion of the second line of the table title and also of the word “NOTE” above the table footnotes;
- pages 9 and 10: Bibliography, change in order and numbering of references, deletion of former reference [6], detailed changes in former references [1] [9] [12] [15] [17] [18].

Introduction

Mooney viscosity, as defined in ISO 289-1, is one of the most widely accepted rubber characterization parameters. However, Mooney viscosity alone is usually insufficient to guarantee that other rheological properties are well controlled [1]. It does not give any information about the elasticity of raw and unvulcanized rubbers [2]. Viscosity and elasticity can change independently, therefore it is important to have test procedures available that are able to measure both properties independently.

Mooney viscosity is measured at one specific shear rate and rubbers exhibit shear rate-dependant viscosity. Sophisticated test equipment to measure the viscosity of a rubber as a function of the shear rate is available. Generally speaking, this type of equipment, its operation and the interpretation of the results is too complicated to be used as a standard quality control tool at present.

As described in the literature [3] the Mooney stress-relaxation is related to the elastic effects in the rheology of unvulcanized rubbers. It can be measured relatively easily and only takes a few seconds extra at the end of a standard Mooney viscosity measurement. The MSR parameter is independent from Mooney viscosity.

Mooney stress-relaxation, combined with the conventional Mooney viscosity, gives a better description of the visco-elastic behaviour of uncompounded as well as compounded, unvulcanized rubbers [14]. Mooney stress relaxation measurements have been proposed as quality control tool [4] [5].

The short interval method as described in this part of ISO 289 is a refinement of the evaluation procedures for Mooney stress relaxation measurements. Short interval evaluation leads to higher reproducibility compared to using an extended interval.

Using a short interval, a major parameter relevant to rubber rheology can be obtained from Mooney stress relaxation experiments viz. the Mooney stress-relaxation rate (MSR) i.e. the rate of decay of torque versus time [16] [17] [18] [19].

The Mooney stress-relaxation rate also has been referred to as "slope", where the latter is sometimes presented as a positive and sometimes as a negative value. As the method described in this part of ISO 289 uses a specific evaluation interval and the parameter is always referred to as a positive value, a new distinctive name has been chosen [6] [7] [8] [15].

Data are available to show that the described method distinguishes polymers (EPDM) with different high molecular weight fractions despite the short evaluation interval.

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

Part 4: Determination of the Mooney stress-relaxation rate

1 Scope

This part of ISO 289 specifies a method of use of a shearing-disc viscometer for measuring the Mooney stress-relaxation rate (MSR) of uncompounded or compounded unvulcanized rubbers, characterizing the elastic response of those materials next to the viscous response as measured by the Mooney viscosity.

2 Normative references

The following referenced documents are indispensable for the application 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 289-1:1994, *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.

3.1

Mooney stress-relaxation rate

MSR

absolute value of the slope of the linear regression line of the log(torque) versus log(time) plot over a specified time interval after stopping the rotor at the end of a Mooney viscosity measurement

NOTE The MSR measurement is a stress relaxation measurement which covers a broad spectrum of relaxation times and is sensitive to polymer structure at a specified relaxation time interval.

4 Principle

The test consists of determining the decay of the Mooney torque immediately after the determination of the Mooney viscosity. After abruptly stopping the rotor at the end of the Mooney viscosity measurement, the decrease in torque is recorded as a function of time. The rate of change of the torque is evaluated over a short time interval assuming power law validity, in accordance with theoretical predictions [9].