



BSI Standards Publication

Rubber, unvulcanized — Determinations using a shearing-disc viscometer

Part 4: Determination of the Mooney stress-relaxation rate

National foreword

This Published Document is the UK implementation of ISO/TS 289-4:2017. It supersedes BS ISO 289-4:2003, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/22, Testing and analysis of rubber.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Part 4: Determination of the Mooney stress- relaxation rate

*Caoutchouc non vulcanisé — Déterminations 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.

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 on 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 the following URL: 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 first edition of ISO/TS 289-4 cancels and replaces ISO 289-4:2003, which has been technically revised.

The main changes are:

- publication as an ISO Technical Specification instead of an International Standard;
- the addition of [Annex B](#).

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

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[2]. It does not give any information about the elasticity of raw and unvulcanized rubbers[3]. 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 are too complicated to be used as a standard quality control tool at present.

As described in the literature[4], 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 Mooney stress-relaxation rate (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[5]. Mooney stress-relaxation measurements have been proposed as a quality control tool[6][7].

The short interval method as described in this document 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, i.e. the rate of decay of torque versus time[8][9][10][11].

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 document uses a specific evaluation interval and the parameter is always referred to as a positive value, a new distinctive name has been chosen[12][13][14][15].

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

The decision to publish this document as a Technical Specification instead of an International Standard is based on the fact that the method in this document was new and not widely enough practised at the time of development.

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

Part 4: Determination of the Mooney stress-relaxation rate

1 Scope

This document 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. The intended use of this document is on quality control measurements.

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 289-1:2015, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

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:

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

3.1

Mooney stress-relaxation rate

MSR

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

Note 1 to entry: 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^[16].