INTERNATIONAL STANDARD

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# Rubber — Measurement of vulcanization characteristics using curemeters —

## Part 1: Introduction

*Caoutchouc — Mesure des caractéristiques de vulcanisation à l'aide de rhéomètres —*

*Partie 1: Introduction*



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# Contents

Page

# Foreword

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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).

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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 6502-1 cancels and replaces the fourth edition of ISO 6502:2016, which has been technically revised to keep consistency within the ISO 6502 series.

A list of all parts in the ISO 6502 series can be found on the ISO website.



## Introduction

An International Standard specifying requirements for the use of oscillating disc curemeters was established in 1977 as ISO 3417, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter*. Later, when various rotorless curemeters were developed and became popular, an International standard for these instruments was produced as ISO 6502, *Rubber — Measurement of cure characteristics with rotorless curemeters*. However, because of the variety of available instruments that differed in geometry and construction, ISO 6502 was not able to specify such requirements in detail. In 1999, it became clear that a number of different rotorless curemeters were available and that significant developments had taken place and were continuing. Hence, it was concluded that, rather than specify individual rotorless instruments, possibly restricting future developments, a more general document was required. Accordingly, it was decided to provide guidance and assistance in the design and use of curemeters generally, and the title of ISO 6502 was changed to *Rubber — Guide to the use of curemeters*. As the use of rotorless curemeters has become more mature, it has now been decided to revise the Guide as Rubber — Measurement of vulcanization characteristics using curemeters — Part 1: Introduction, with subsequent parts for oscillating disc curemeters and rotorless curemeters.

# Rubber — Measurement of vulcanization characteristics using curemeters —

## Part 1: Introduction

**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 provides an introduction to the determination of vulcanization characteristics of rubber compounds by means of curemeters.



## 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 1382, *Rubber — Vocabulary*



## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1382 and the following 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**
**oscillating-disc curemeter**
**ODC**
curemeter consisting of a biconical disc oscillated within a temperature-controlled die cavity containing the test piece

Note 1 to entry: An oscillating-disc curemeter is also known as an oscillating disc rheometer (ODR).

**3.2**
**rotorless curemeter**
**RCM**
curemeter consisting of two dies forming a temperature-controlled cavity, one of which is moved relative to the other to apply a stress or strain to the test piece

Note 1 to entry: A rotorless curemeter is also known as a moving die rheometer (MDR).

**3.3**
**marching-modulus cure**
type of vulcanization during which the modulus does not reach a maximum value but, after a rapid rise, continues to rise slowly at the vulcanization temperature

**3.4**
**vulcanization characteristics**
characteristics which can be taken from a vulcanization curve

Note 1 to entry: See Figure 1.

Note 2 to entry: More explanations are given in Clause 4.

**3.5**
**stiffness**
measure of the resistance offered by rubber to deformation

Note 1 to entry: Force and torque have not been defined since they have a generally accepted scientific meaning.

## 4 Basic principles

The properties of a rubber compound change during the course of vulcanization, and the vulcanization characteristics can be determined by measuring properties as a function of time and temperature. Vulcanization characteristics are most commonly determined using instruments known as curemeters in which a cyclic stress or strain is applied to a test piece and the associated strain or force is measured. Normally, the test is carried out at a predetermined constant temperature and the measure of stiffness recorded continuously as a function of time.



The stiffness of the rubber increases as vulcanization proceeds. Vulcanization is complete when the recorded stiffness rises to a plateau value or to a maximum and then declines (see Figure 1). In the latter case, the decrease in stiffness is caused by reversion. In cases where the recorded stiffness continues to rise (marching-modulus cure), vulcanization is deemed to be complete after a specified time. The time required to obtain a vulcanization curve is a function of the test temperature and the characteristics of the rubber compound. Curves analogous to Figure 1 are obtained for a curemeter in which strain is measured.



Direct proportionality between torque and stiffness cannot be expected under all conditions and all instruments because, particularly at in high torque ranges, elastic deformation of the disc shaft and driving device has to be taken into account, Moreover, in cases of small amplitudes of deformation, the strain can be expected to have a considerable elastic component. However, for routine control purposes, corrections are not necessary.

The following vulcanization characteristics can be taken from the measure of stiffness against time curve (see Figure 1).

| | |
|---|---|
| Minimum force or torque | $F_L$ or $M_L$ |
| Force or torque at a specified time $t$ | $F_t$ or $M_t$ |
| Scorch time (time to incipient cure) | $t_{sx}$ |
| Time to a percentage $y$ of full cure from minimum force or torque | $t'_c(y)$ |
| Plateau force or torque | $F_{HF}$ or $M_{HF}$ |
| Maximum force or torque (reverting cure) | $F_{HR}$ or $M_{HR}$ |
| Force or torque value attained after a specified time (marching-modulus cure) | $F_H$ or $M_H$ |

The minimum force or torque $F_L$ or $M_L$ characterizes the stiffness of the unvulcanized compound at the curing temperature.

The scorch time (time to incipient cure) $t_{sx}$ is a measure of the processing safety of the compound.

The time $t'_c(y)$ and the corresponding forces or torques give information on the progress of cure. The optimum cure is often taken as $t'_c(90)$.

The highest force or torque is a measure of the stiffness of the vulcanized rubber at the curing temperature.

NOTE The term *F* denotes force and the term *M* denotes torque.

The scorch time $t_{sx}$ is the time required for the force or torque to increase by *x* units from $F_L$. It might be convenient to define the scorch as a given percentage, e.g. 2 % or 5 %, of the total cure.

The time to a percentage of full cure from minimum force, $t'_c(y)$, is the time taken for the force (or torque) to reach:

$$F_L + 0{,}01\,y\left(F_{HF} - F_L\right) \quad (1)$$

or

$$M_L + 0{,}01\,y\left(M_{HF} - M_L\right) \quad (2)$$

— $t'_c(10)$ is a measure of the early stages of cure.

— $t'_c(50)$ can be determined accurately providing the slope of the curve is greatest at this point.

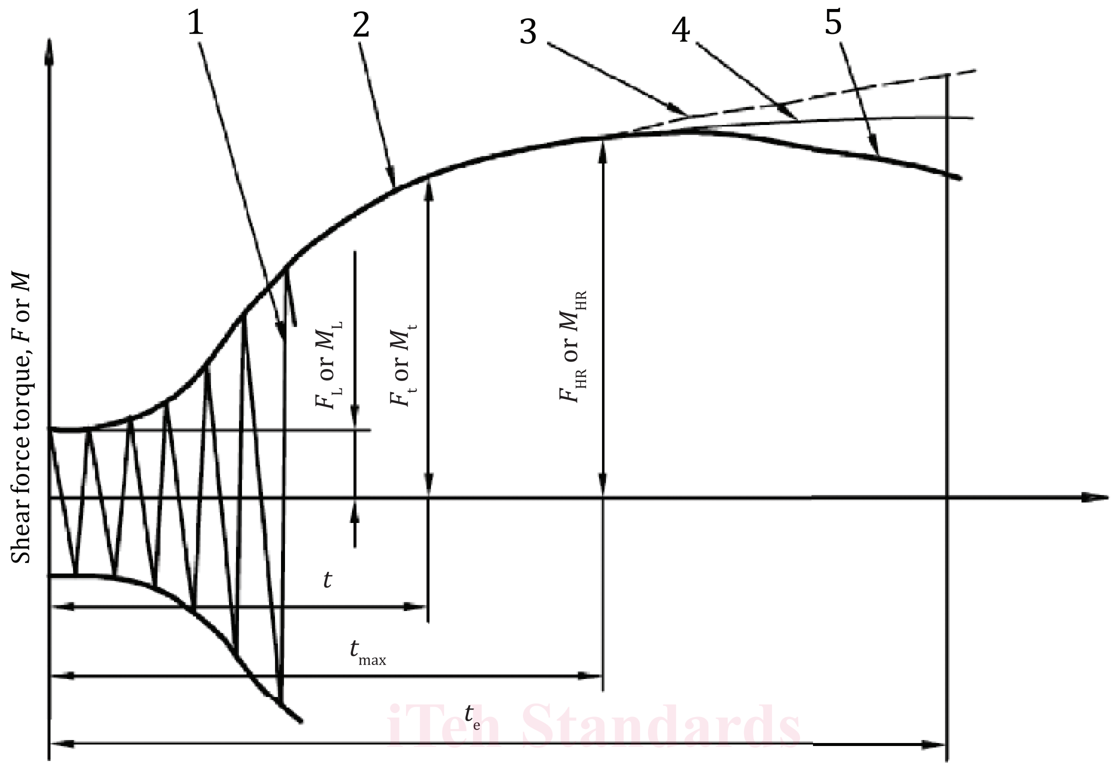
— $t'_c(90)$ is often used as an indicator of optimum press cure.

The cure rate index is the average slope of the rising curve and is given by:

$$100 / \left[t'_c\left(y\right) - t_{sx}\right] \quad (3)$$






**a) Vulcanization curve *F* or *M* = *f*(*t*)**