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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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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 document 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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This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 6502-1:2018), which has been technically revised.

The main changes are as follows:

— description of t’c (50) was corrected in Clause 4;

— descriptions of reciprocating types and top hat type dies were deleted.

A list of all parts in the ISO 6502 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.

Introduction

An International Standard specifying requirements for the use of oscillating disc curemeters was established in 1977 as ISO 3417.[1)]
Later, when various rotorless curemeters were developed and became popular, an International Standard for these instruments was produced as ISO 6502[2)]
. However, owing to the differences in geometry and construction of the various available instruments, 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 to do so. 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 general guidance and assistance in the design and use of curemeters.

The previous edition of this document (ISO 6502-1:2018) replaced ISO 6502, which was split into subsequent parts, covering oscillating disc curemeters (ISO 6502-2) and rotorless curemeters (ISO 6502-3). ISO 6502-1 covers the general principle required for the measurement of vulcanization characteristics using curemeters.

1) Withdrawn.

2) Withdrawn.

Rubber — Measurement of vulcanization characteristics using curemeters —

Part 1:
Introduction

WARNING — 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.

Certain procedures specified in this document can involve the use or generation of substances, or the generation of waste, that can 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. It describes the basic principles and general requirements for 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 terminology databases for use in standardization at the following addresses:

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

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

3.1
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

3.2
vulcanization characteristic
characteristic which can be taken from a vulcanization curve

3.3
stiffness
measure of the resistance offered by rubber to deformation

4 Basic principles

The properties of a rubber compound change during the course of vulcanization. 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 is 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 shall 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).

Table 1 — Vulcanization characteristics

Characteristic	Symbol
Force	F
Torque	M
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.

The scorch time, t_{sx}, is the time required for the force or torque to increase by x units from F_L. It can 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 and is given by Formula (1) or Formula (2):

$$t'c(y) = F_L + 0{,}01\, y (F_{HF} - F_L) \quad (1)$$

or

$$t'c(y) = M_L + 0{,}01\, y (M_{HF} - M_L) \quad (2)$$

where

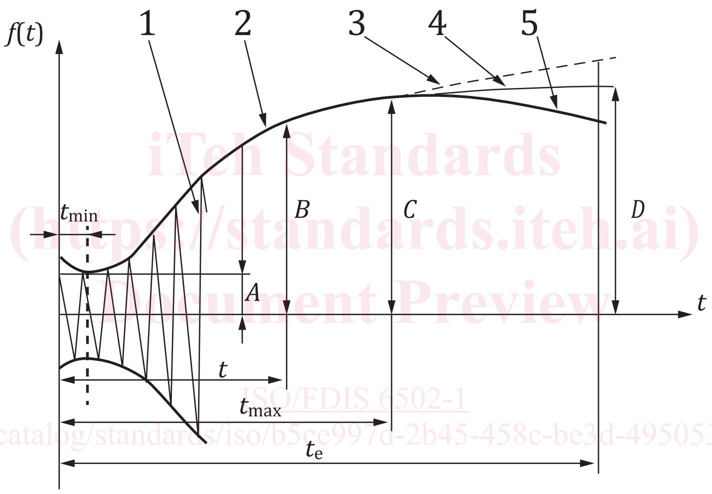
$t'_c(10)$ is a measurement time of the early stages of cure;

$t'_c(50)$ is a time to the midpoint of cure stage;

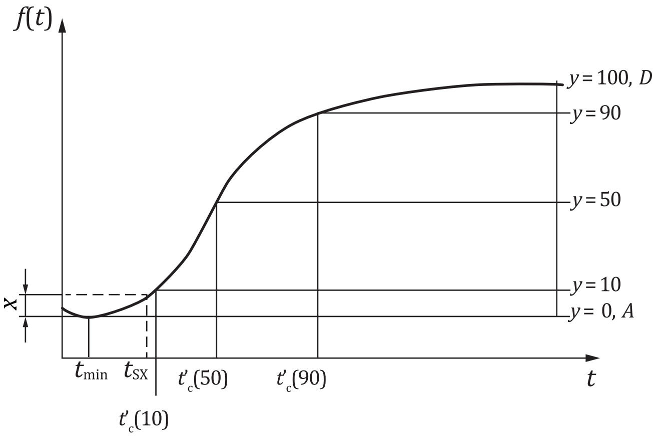
$t'_c(90)$ is often used as an indicator of optimum press cure time.

The cure rate index is the average slope of the rising curve and is given by Formula (3):

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



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



b) Method of evaluation

Key

t time

Y force F or torque M

A F_L or M_L

B F_t or M_t

C F_{HR} or M_{HR}

D F_{HF} or M_{HF}

1 sinusoidal curve

2 envelope curve

3 vulcanization curve with steady increase to F_{H} or M_{H} at time t_{e} at end of test (marching-modulus cure)

4 vulcanization curve with plateau at F_{HF} or M_{HF} (plateau cure)

5 vulcanization curve with maximum F_{HR} or M_{HR} at time t_{max} (reverting cure)

NOTE See Table 1 for the definition of the symbols in this figure.

Figure 1 — Typical vulcanization curve and method of evaluation

5 Types of curemeter

Three types of curemeters have found widespread use:

— oscillating-disc;

— reciprocating-paddle;

— rotorless.

Other geometries are possible, for example with a vibrating probe or needle.

NOTE The reciprocating-paddle type was previously popular but is now more or less obsolete and is thus not considered in this document.

The oscillating-disc curemeter consists of a biconical disc that oscillates in a closed cavity. For many years it was the most widely used type of instrument.

In rotorless curemeters, one half of a die enclosing the test piece oscillates or reciprocates, rather than a disc within the test piece. The rotorless type of curemeter has increased greatly in popularity, largely because of its advantages of the specified temperature being reached in a shorter time after insertion of the test piece into the die cavity and better temperature distribution in the test piece (see Annex A).