INTERNATIONAL STANDARD

ISO 6502

Third edition
1999-12-01

Rubber — Guide to the use of curemeters

Caoutchouc — Guide pour l'emploi des rhéomètres

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4e-02b3-4479-8ac2-bb1a2296cc14/iso-6502-1999

Reference number
ISO 6502:1999(E)

Contents

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/...
bb1a2296cc14/iso-6502-1999

© ISO 1999
All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch
Printed in Switzerland

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

International Standard ISO 6502 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests.*

This third edition cancels and replaces the second edition (ISO 6502:1991), which has been technically revised.

Annexes A, B and C of this International Standard are for information only.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4e-02b3-4479-8ac2-bb1a2296cc14/iso-6502-1999

Introduction

Whilst reviewing the previous edition of this International Standard, it became clear that a number of different curemeters were available and that significant developments had taken place, especially with the rotorless types. Rather than specify individual rotorless instruments, possibly restricting future developments, it was felt that a more general document was required. Accordingly, it was decided to provide guidance and assistance in the design and use of curemeters generally.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4e-02b3-4479-8ac2-bb1a2296cc14/iso-6502-1999

Rubber — Guide to the use of curemeters

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 ensure compliance with any national regulatory conditions.

1 Scope

This International Standard provides guidance on the determination of vulcanization characteristics of rubber compounds by means of curemeters.

2 Normative references

iTeh STANDARD PREVIEW
(standards.iteh.ai)

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4c-0285-4479-8ac2-601a2296c014/iso-6502-1999

ISO 1382:1996, *Rubber — Vocabulary.*

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 1382 apply, plus the following:

3.1
ocscillating-disc curemeter
ODC
a curemeter consisting of a biconical disc oscillated within a temperature-controlled die cavity containing the test piece

3.2
rotorless curemeter
RCM
a 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 Types of rotorless curemeter are listed in clause 5 and illustrated in Figures 3 to 7.

3.3
marching-modulus cure

a 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 may be taken from a vulcanization curve (see Figure 1).

NOTE More explanations are given in clause 4.

3.5
stiffness

a general term used for modulus or resistance to stress

NOTE 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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/ ... iso-6502-1999

The following vulcanization characteristics can be taken from the measure of stiffness against time curve (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

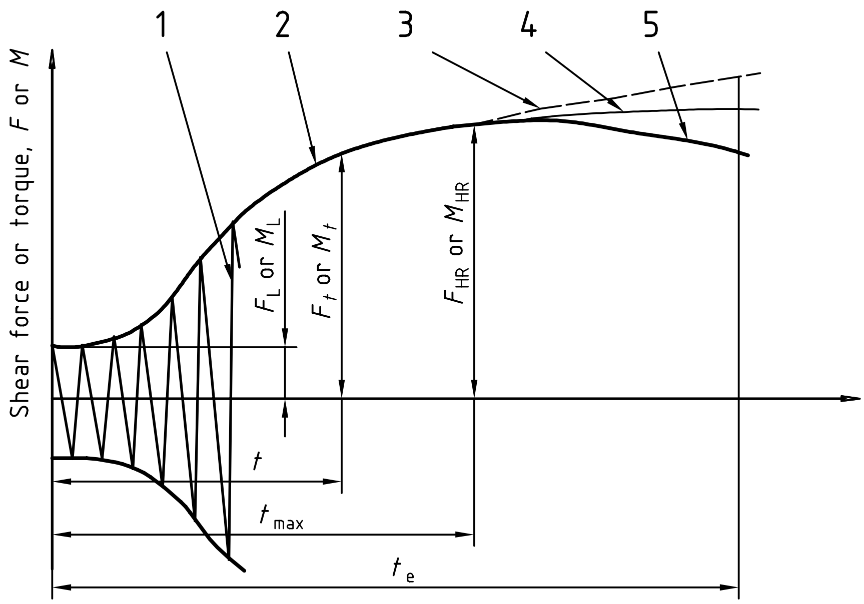
The minimum force or torque F_L or M_L characterizes the stiffness of the unvulcanized compound at the curing temperature.

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.



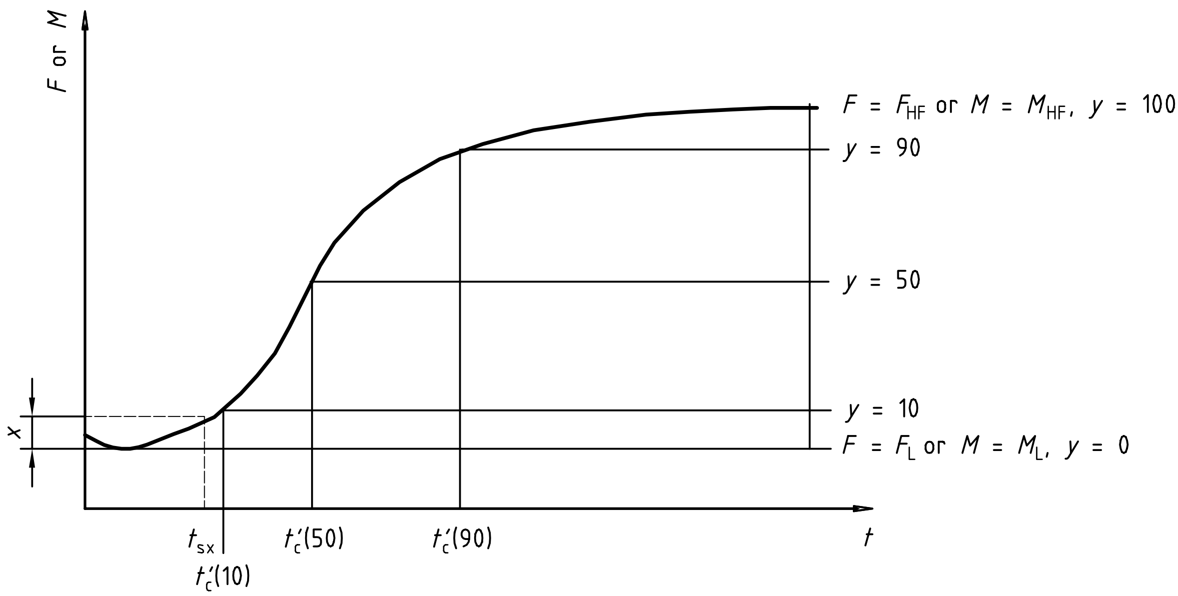
Key

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)

iTeh STANDARD PREVIEW
(standards.iteh.ai)

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

ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4e-02b3-4479-8ac2-bb1a2296cc14/iso-6502-1999



b) Method of evaluation

Figure 1 — Typical vulcanization curve and method of evaluation

5 Types of curemeter

Three types of curemeter have found widespread use:

— Oscillating-disc;

— Reciprocating-paddle;

— Rotorless.

The reciprocating-paddle type was popular, but is now much less used and is not considered further in this guide.

Rotorless curemeters can be subdivided into three forms:

— Reciprocating (linear strain);

— Oscillating (torsion) unsealed cavity;

— Oscillating (torsion) sealed cavity.

Oscillating types may have a biconical die, a flat-plate die or be of top-hat section.

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

The oscillating-disc curemeter has for many years been the most widely used type of instrument, but more recently the rotorless type of curemeter has increased greatly in popularity. The principal advantages of the rotorless type are that the specified temperature is reached in a shorter time after insertion of the test piece into the die cavity and there is better temperature distribution in the test piece (see annex A).

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4e-02b3-4479-8ac2-bb1a2296cc14/iso-6502-1999

6 Apparatus

6.1 General

A curemeter consists of two heated dies with means of closing them under a specified force to form a die cavity containing the test piece, a means of oscillating a rotor within the cavity, or alternatively oscillating or reciprocating one of the dies relative to the other, and a means of measuring and recording the force or torque required to produce the relative movement, or the movement produced by a given applied force or torque. In addition, with sealed rotorless torsion systems, reaction torque on the stationary die opposite the moving die may be measured.

The general arrangements for oscillating-disc and rotorless curemeters are shown in Figures 2 to 7.

6.2 Die cavity

The dies should be manufactured from a non-deforming material. The surface of the dies should minimize the effect of contamination and be hard so as to prevent wear. A minimum Rockwell hardness of 50 HRC, or equivalent, is recommended. The tolerances necessary on the dimensions of the dies will depend on the particular design, but as a general guide the dimensions of the cavity should be controlled to ±0,2 %.

The top and bottom surfaces of the cavity should have a pattern of grooves of dimensions sufficient to prevent slippage of the rubber test piece.

Holes should be provided in both the upper and lower dies to accommodate temperature sensors. The positions of the sensors relative to the cavity should be controlled to ensure reproducible response.

In the case of oscillating-disc instruments, one die requires a central hole to allow insertion of the die stem. A seal of suitable low, constant friction should be provided in this hole to prevent material leaking from the cavity.

Suitable means should be employed by design of dies or otherwise to apply pressure to the test piece throughout the test to minimize slippage between the disc and the rubber. A positive pressure is also important to exclude air which may affect the cure of, for example, peroxide-cured rubbers and to prevent any tendency for the rubber to become porous.

The dimensions of the die cavity may be checked by measuring the dimensions of the vulcanized test piece. For biconical-die rotorless curemeters, particular attention should be paid to the thin central portion, the thickness of which depends on the die gap. For oscillating-disc curemeters, the vulcanized test piece should be cut in half and checked to see that it is symmetrical. Any asymmetry indicates that the rotor height has been set incorrectly.

The dimensions of the cavity and of the vulcanized test piece will not be identical because of the effect of mould shrinkage.

6.3 Die closure

The dies are closed and held closed during the test by, for example, a pneumatic cylinder.

A force of 11 kN ± 0,5 kN is recommended for oscillating-disc instruments with a mating-surface area between the dies of approximately 1 400 mm^2.

In unsealed rotorless instruments, the dies are not completely closed but a small clearance is left which should be between 0,05 mm and 0,2 mm. For sealed cavities, no gap should exist at the edges of the die cavity. The minimum closing force required depends on the clearance area. As a general guide, a minimum of 7 kN to 8 kN is recommended.

6.4 Moving member

iTeh STANDARD PREVIEW
(standards.iteh.ai)

The disc in an oscillating-disc instrument should be manufactured from a non-deforming material having a minimum hardness of 50 HRC. Both the top and bottom surfaces should have a pattern of grooves to prevent slippage of the rubber test piece.

ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4e-02b3-4479-8ac2-bb1a2296ee14/iso-6502-1999

The disc should be biconical in shape to give an approximately uniform shear rate, and its diameter should be controlled to ± 0,03 % and the cone angle to ± 1,3 %.

The moving member in a rotorless instrument is one of the dies. The shape of the die cavity should be a plane disc for reciprocating types and either biconical, flat plate or “top hat” in the oscillating type to produce a substantially uniform shear rate.

The drive linkage should be sufficiently stiff to prevent significant deformation.

6.5 Movement

The frequency of oscillation or reciprocation should be between 0,05 Hz and 2 Hz, and tests may be made at two or more frequencies. If a single frequency is selected, 1,7 Hz ± 0,1 Hz is recommended.

Generally, greater sensitivity can be obtained with larger amplitudes, but the amplitude that can be used in practice is restricted by the possibility of slippage between the test pieces and the die surface or rotor.

For oscillating-disc curemeters, an amplitude of ± 1° is recommended, but ± 3° may be possible and advantageous in some circumstances.

For rotorless curemeters, the range may be between ± 0,1° and ± 2° or, for reciprocating types, between ± 0,01 mm and ± 0,1 mm.

The tolerance on amplitude should be ± 2 % apart from the instruments considered in ISO 3417, and the drive should be sufficiently powerful and stiff to substantially maintain the amplitude under load.

6.6 Stiffness measurement

The means of measuring force or torque should be rigidly coupled to a die or rotor and be capable of measuring the resultant force or torque to an accuracy of ± 1 % of the force or torque range. This tolerance should include any errors due to deformation of the measuring device and its coupling and of the output device.

The recorder to continuously monitor force or torque should have a response time for full-scale deflection of 1 s or less.

6.7 Heating and temperature control

The heating and temperature control system should be capable of producing a reproducible and evenly distributed temperature in the dies and permit rapid and reproducible temperature recovery after insertion of the test piece. Close control of these parameters is necessary for the precise measurement of vulcanization characteristics.

The temperature-measuring system should enable temperature to be measured to a resolution of ± 0,1 °C over the range 100 °C to 200 °C. The temperature controllers should enable the dies to be controlled to an accuracy of ± 0,3 °C at the steady state. The temperature of the dies should recover after insertion of a test piece at 23 °C ± 5 °C to within 0,3 °C within 3 min for biconical-die rotorless instruments. For flat-plate-die rotorless instruments, the recovery range should be ± 1 °C within 1,5 min at the test temperature of 150 °C.

The effect of temperature distribution on measured cure rate is discussed in annex A.

6.8 Calibration

iTeh STANDARD PREVIEW
(standards.iteh.ai)

Calibration of curemeters should be carried out in accordance with the manufacturer's instructions. The force or torque should be determined at several points over the range(s) used but, additionally, it may be useful to have provision for making in-use checks.

Stable standard rubber compounds may also be tested periodically to check for consistent performance.

ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4e-02b3-4479-8ac2-bb1a2296cc14/iso-6502-1999

7 Test piece

The test piece should be homogeneous and as far as possible free from trapped air. The test piece volume should be slightly larger than the die cavity volume such that a small amount of material is extruded between all edges of the dies when they are closed. The optimum volume should be determined by preliminary tests, and test pieces of equal volume should be used to obtain reproducible results. Oversize test pieces may cool the cavity excessively during the early part of the test cycle.

The test piece should be punched from sheeted material by an appropriate device which ensures the production of test pieces of constant volume.

Normally one test piece is taken from each rubber sample, but if this may not be representative of the batch then further test pieces should be taken.

8 Vulcanization temperature

The vulcanization temperature is chosen as that appropriate for the rubber compound being tested and intended processing. The range of 100 °C to 200 °C is recommended.

9 Conditioning

The rubber sample should be conditioned at 23 °C ± 5 °C for a minimum of 3 h before testing.

10 Test procedure

10.1 Preparation for the test

The temperature of both dies should be brought to the test temperature with the cavity closed and, in the case of oscillating-disc curemeters, with the disc in place, and allowed to stabilize.

Any necessary zeroing and selection of range of the force- or torque-measuring device should be made before loading the test piece.

10.2 Loading the curemeter

The loading of the test piece and the closure of the dies should be carried out as quickly as possible. The dies should be closed immediately after insertion of the test piece. The whole cycle, from opening to closure, should not exceed 20 s.

The vulcanization time should be recorded from the instant the dies are fully closed. Oscillation of the movable die or disc should be started before or at the instant of die closure.

After removal of the cured test piece, a further sample may be inserted immediately if the temperature of the dies has remained within $\pm 0{,}3$ °C of the set value. If not, the dies should be closed and the temperature allowed to recover to the test value.

A deposit of material from the rubber compound may build up on the dies (and disc) which may affect the final torque values. The use of a standard compound may be used to detect this occurrence. If such contamination develops, it may be removed by very light blasting with a mild abrasive, ultrasonic cleaning or non-corrosive cleaning fluids. Great care should be taken with cleaning, and the manufacturer's advice followed. If fluids are used, the first two tests after cleaning should be rejected. Running a natural-rubber gum compound can be used to remove debris. In some cases, a protective film may be used to prevent contamination. For biconical-die rotorless curemeters of the sealed-cavity type, the use of protective film (polyester of thickness $< 0{,}03$ mm) is strongly recommended.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 6502:1999
https://standards.iteh.ai/catalog/standards/sist/c09c1d4e-02b3-4479-8ac2-bb1a2296cc14/iso-6502-1999

11 Expression of results

All or some of the cure characteristics given in clause 4 should be taken from the cure curve. Times should be given in minutes, force in newtons and torque in newton metres.

The scorch time t_{sx} is the time required for the force or torque to increase by x units from F_L. It may 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

$$F_L + 0{,}01y\,(F_{HF} - F_L)$$

or

$$M_L + 0{,}01y\,(M_{HF} - M_L)$$

$t'_c(10)$ 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

$$100/[t'_c(y) - t_{sx}]$$