
Rubber — Guide to the use of curemeters

Caoutchouc — Guide pour l'emploi des rhéomètres

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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).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 6502:1999), of which it constitutes a minor revision. The references have been updated.

Introduction

In 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.

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Rubber — Guide to the use of curemeters

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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.

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).

Note 2 to entry: Types of rotorless curemeter are listed in [Clause 5](#) and illustrated in [Figure 3](#) to [Figure 7](#).

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 may 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

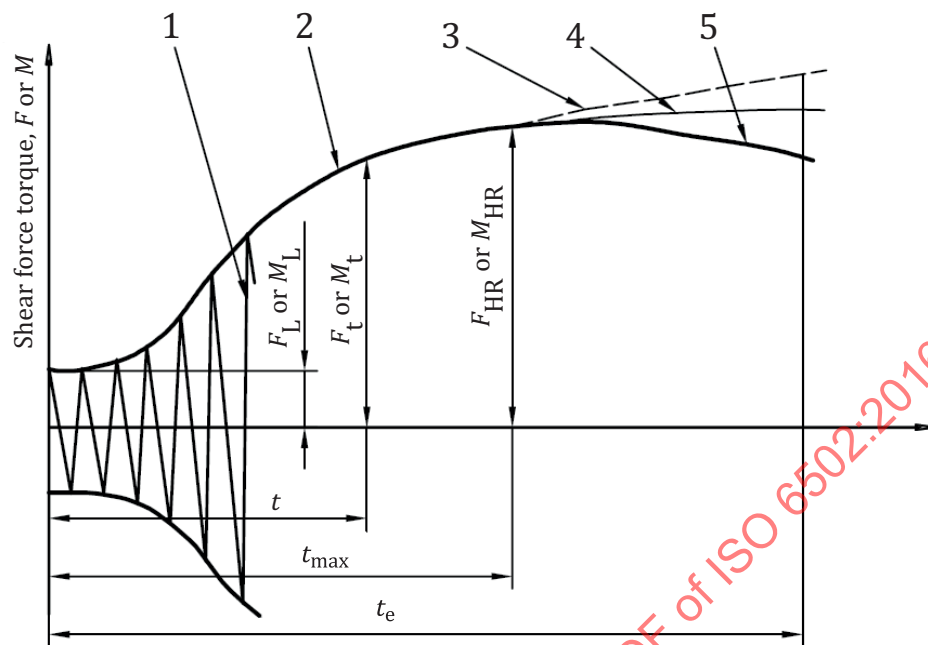
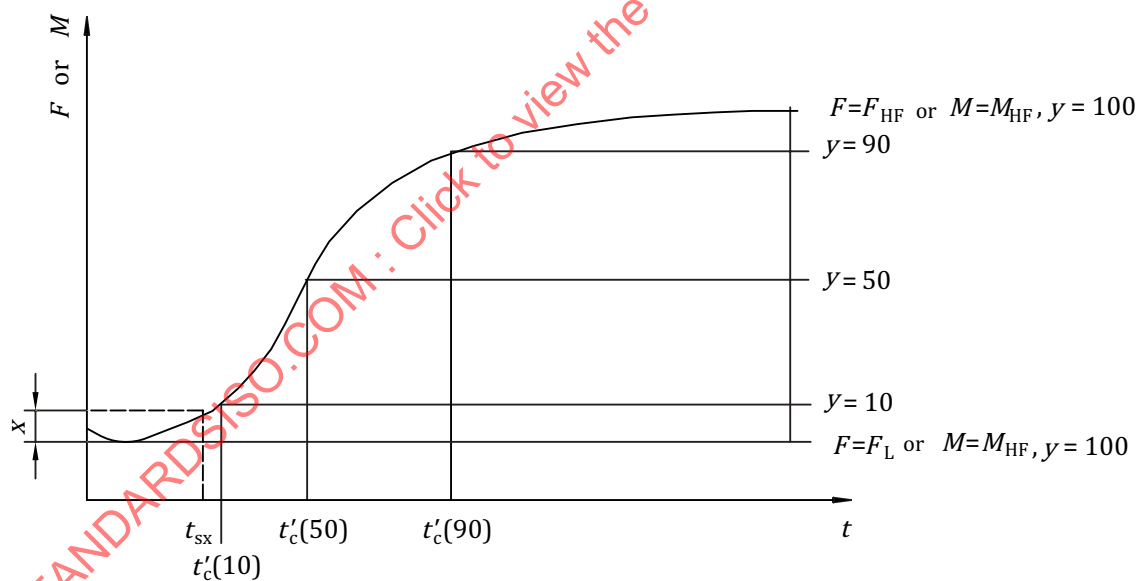
The properties of a rubber compound change during the course of vulcanization, and the vulcanization characteristics can be determined by measuring properties as a function of time and temperature. Vulcanization characteristics are most commonly determined using instruments known as curemeters in which 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.

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.

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

b) Method of evaluation

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)

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.

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

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, 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).

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 [Figure 2](#) to [Figure 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 $\pm 0,2\%$.

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 might 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 $11 \text{ kN} \pm 0,5 \text{ kN}$ is recommended for oscillating-disc instruments with a mating-surface area between the dies of approximately $1\,400 \text{ mm}^2$.

In unsealed rotorless instruments, the dies are not completely closed but a small clearance is left which should be between $0,05 \text{ mm}$ and $0,2 \text{ mm}$. 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

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.

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

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 $0,05 \text{ Hz}$ and 2 Hz , and tests may be made at two or more frequencies. If a single frequency is selected, $1,7 \text{ Hz} \pm 0,1 \text{ Hz}$ 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 $\pm 1^\circ$ is recommended but $\pm 3^\circ$ may be possible and advantageous in some circumstances.

For rotorless curemeters, the range may be between $\pm 0,1^\circ$ and $\pm 2^\circ$ or, for reciprocating types, between $\pm 0,01 \text{ mm}$ and $\pm 0,1 \text{ mm}$.

The tolerance on amplitude should be $\pm 2 \%$ 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 $\pm 1\%$ 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 $\pm 0,1\text{ }^{\circ}\text{C}$ over the range $100\text{ }^{\circ}\text{C}$ to $200\text{ }^{\circ}\text{C}$. The temperature controllers should enable the dies to be controlled to an accuracy of $\pm 0,3\text{ }^{\circ}\text{C}$ at the steady-state. The temperature of the dies should recover after insertion of a test piece at $23\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ to within $0,3\text{ }^{\circ}\text{C}$ within 3 min for biconical-die rotorless instruments. For flat-plate-die rotorless instruments, the recovery range should be $\pm 1\text{ }^{\circ}\text{C}$ within 1,5 min at the test temperature of $150\text{ }^{\circ}\text{C}$.

The effect of temperature distribution on measured cure rate is discussed in [Annex A](#).

6.8 Calibration

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.

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 $100\text{ }^{\circ}\text{C}$ to $200\text{ }^{\circ}\text{C}$ is recommended.

9 Conditioning

The rubber sample should be conditioned at $23\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ 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.

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

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

or

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

- $t'_c(10)$ 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

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

12 Test report

The test report shall include the following information:

- a) a reference to the International Standard, i.e. ISO 6502;
- b) sample details:
 - 1) a full description of the sample and its origin;
 - 2) the method of preparation of the test pieces;
- c) test details:
 - 1) type and identification of curemeter used;
 - 2) the amplitude of oscillation;
 - 3) the frequency of oscillation;
 - 4) the force or torque range used;
 - 5) the heating-up time;
 - 6) the curing temperature;
- d) test results:
 - 1) the recorded vulcanization curve with force and time scales indicated, if required;
 - 2) the values of the parameters obtained from the vulcanization curve, as required;
- e) date of the test.

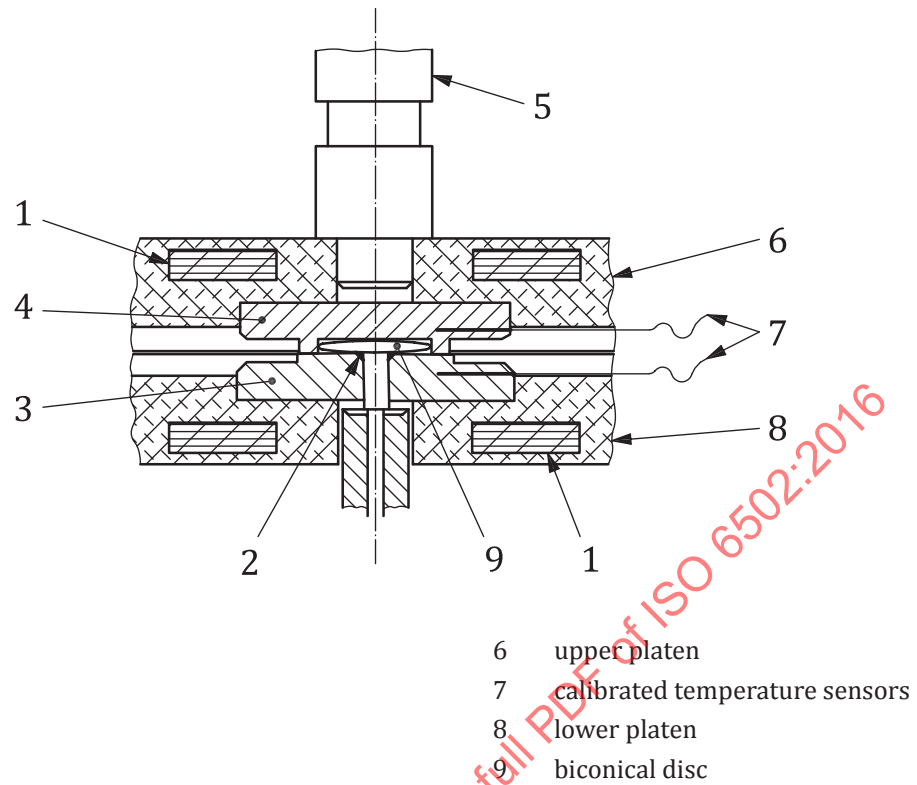
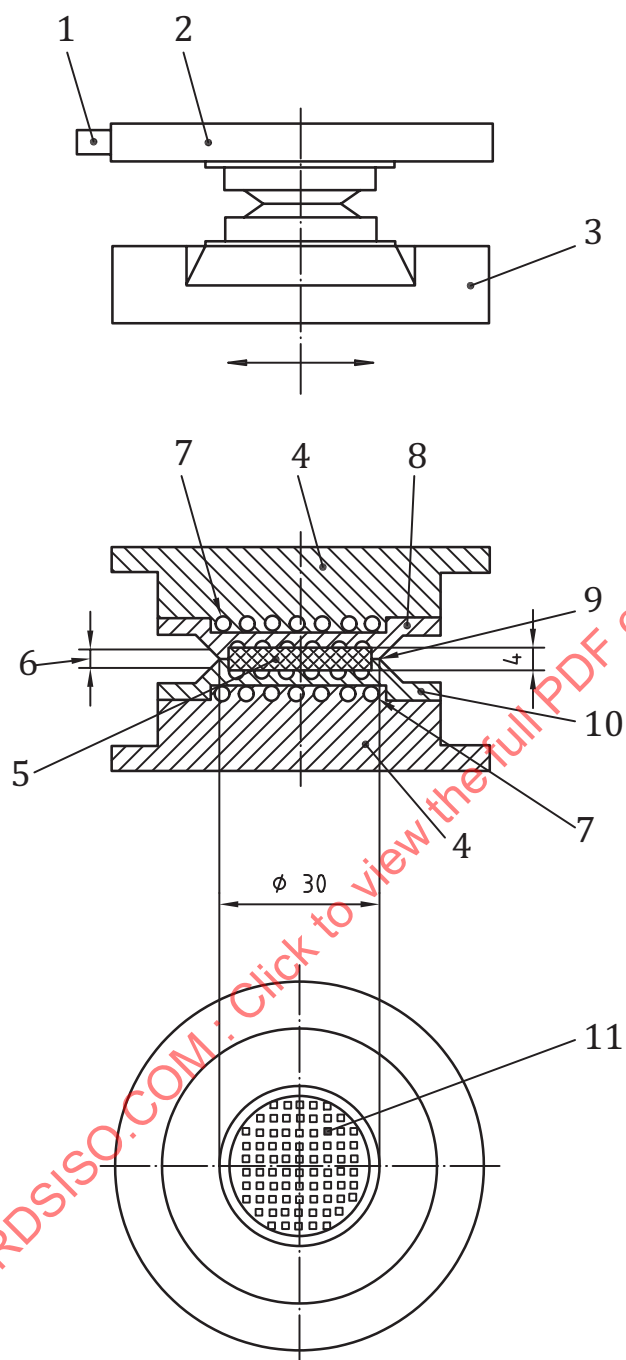


Figure 2 — Typical oscillating-disc curemeter

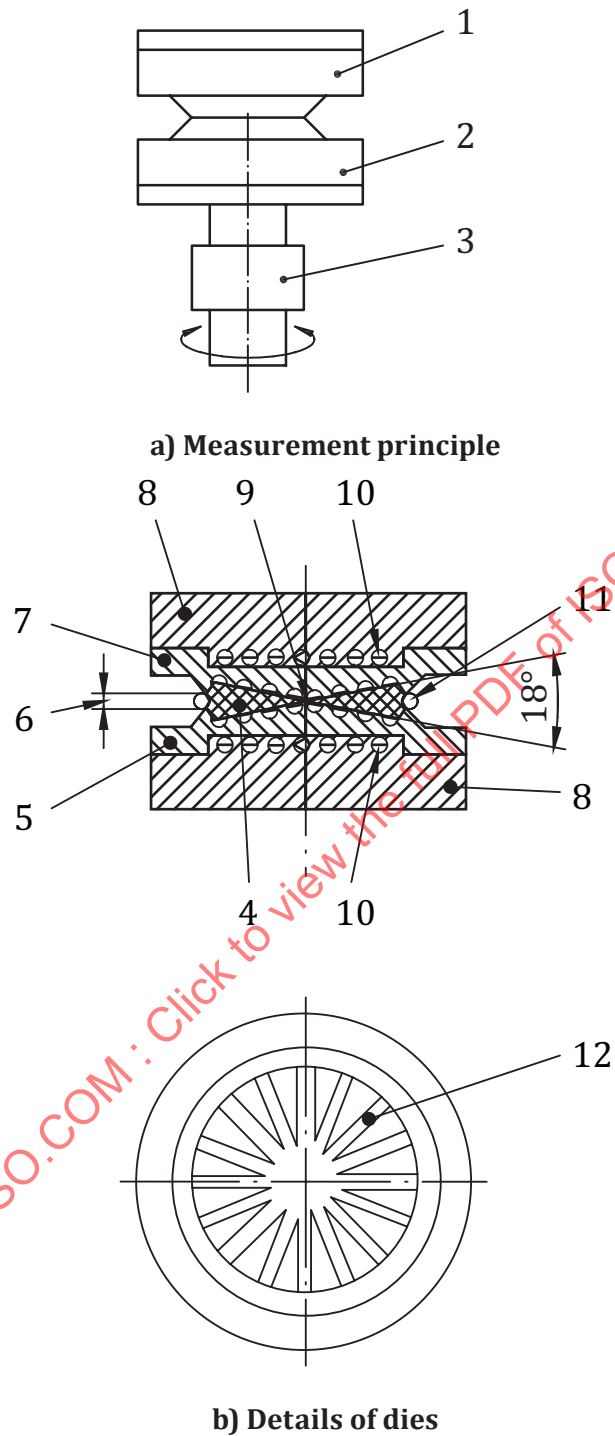
Dimensions in millimetres

**Key**

- 1 force-measuring system
- 2 fixed die
- 3 oscillating die
- 4 temperature sensor
- 5 test piece
- 6 die gap

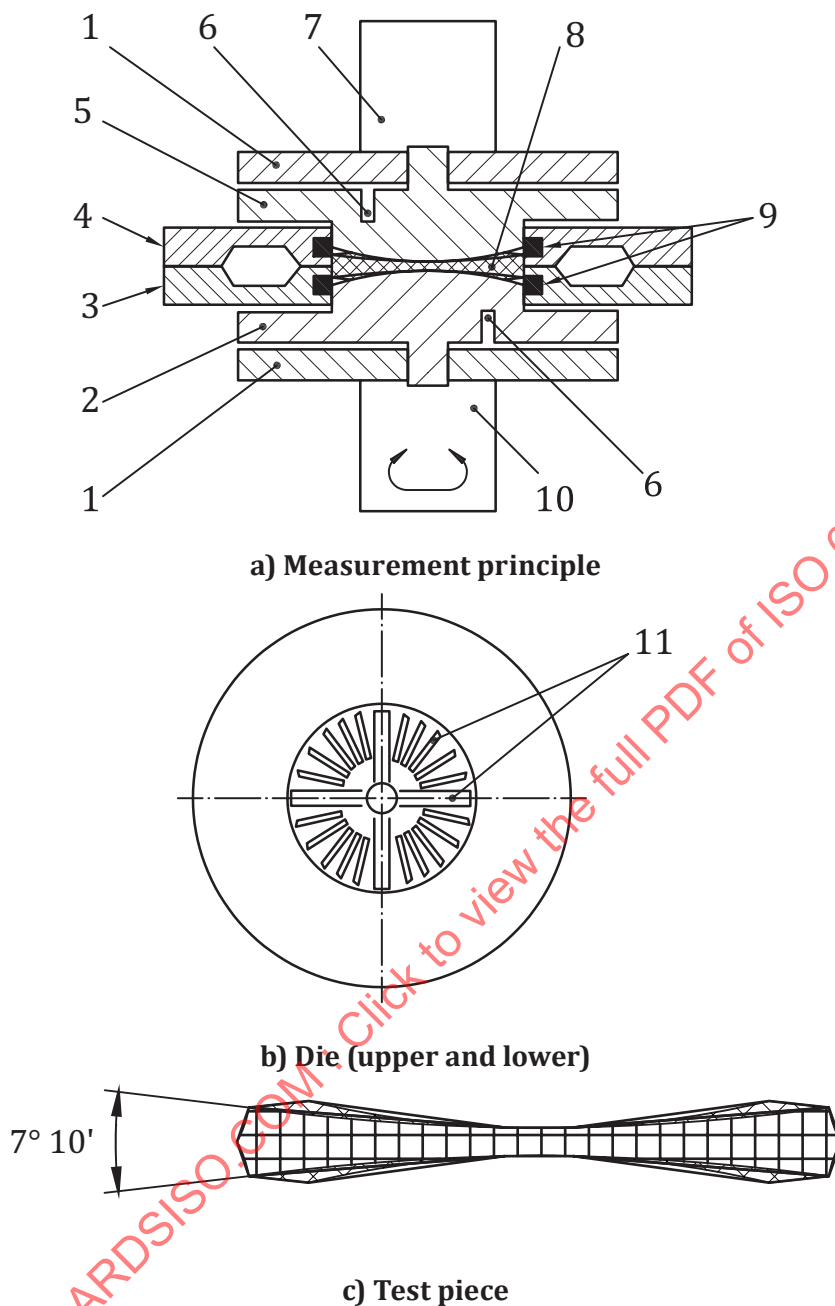
- 7 heater
- 8 upper die
- 9 spew
- 10 lower die
- 11 grooves

Figure 3 — Typical linear-shear curemeter

**Key**

- | | | | |
|---|-------------------------|----|--------------------|
| 1 | fixed die | 7 | upper die |
| 2 | oscillating die | 8 | temperature sensor |
| 3 | torque-measuring system | 9 | die gap >0,5 mm |
| 4 | test piece | 10 | heater |
| 5 | lower die | 11 | spew |
| 6 | die gap | 12 | grooves |

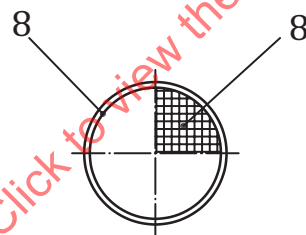
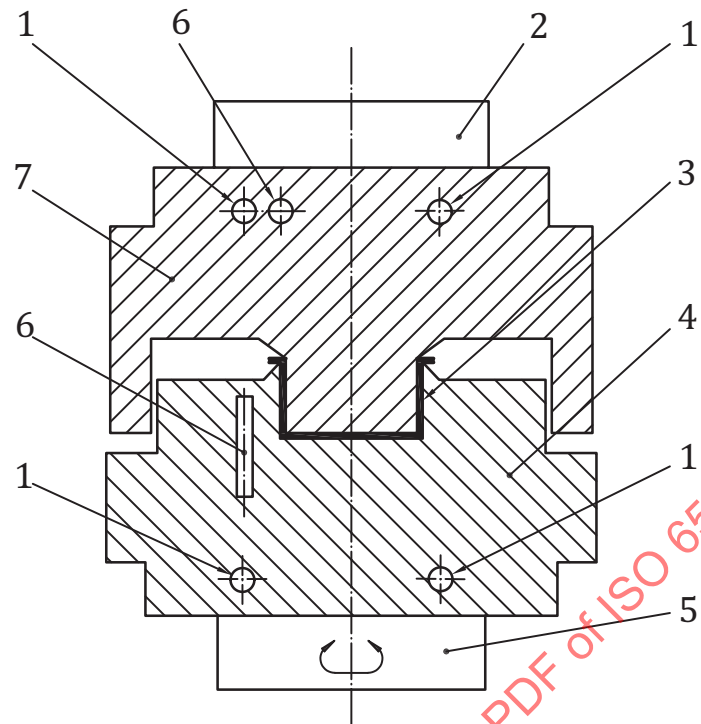
Figure 4 — Typical unsealed torsion-shear curemeter



Key

- | | | | |
|---|--------------------|----|--------------------------|
| 1 | heater | 7 | torque-measuring system |
| 2 | lower die | 8 | test piece |
| 3 | lower seal plate | 9 | seals |
| 4 | upper seal plate | 10 | oscillating-drive system |
| 5 | upper die | 11 | grooves |
| 6 | temperature sensor | | |

Figure 5 — Typical sealed torsion-shear rotorless curemeter with biconical-die structure



a) Details of dies



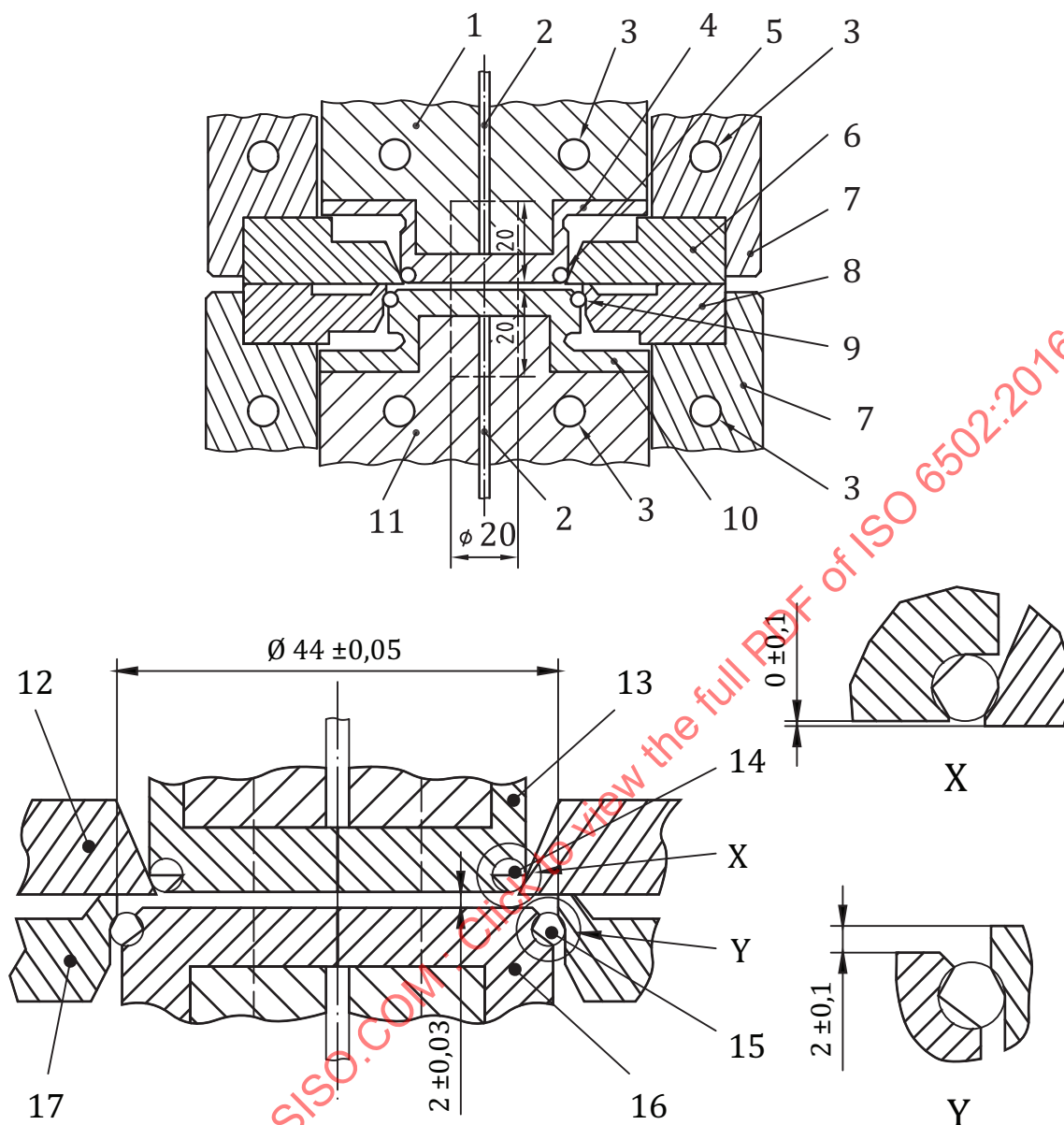
b) Test piece

Key

- | | | | |
|---|-------------------------|---|--------------------------|
| 1 | heater | 5 | oscillating-drive system |
| 2 | torque-measuring system | 6 | temperature sensor |
| 3 | test piece | 7 | upper die |
| 4 | lower die | 8 | grooves |

Figure 6 — Typical top-hat-section rotorless curemeter

Dimensions in millimetres



Key

1	shaft or torque-measuring system	10	lower die
2	temperature sensor	11	drive shaft
3	heater	12	upper seal plate
4	upper die	13	upper die
5	upper seal	14	upper seal
6	upper seal plate	15	lower seal
7	platen	16	lower die
8	lower seal plate	17	lower seal plate
9	lower seal		

Figure 7 — (continued)

Dimensions in millimetres

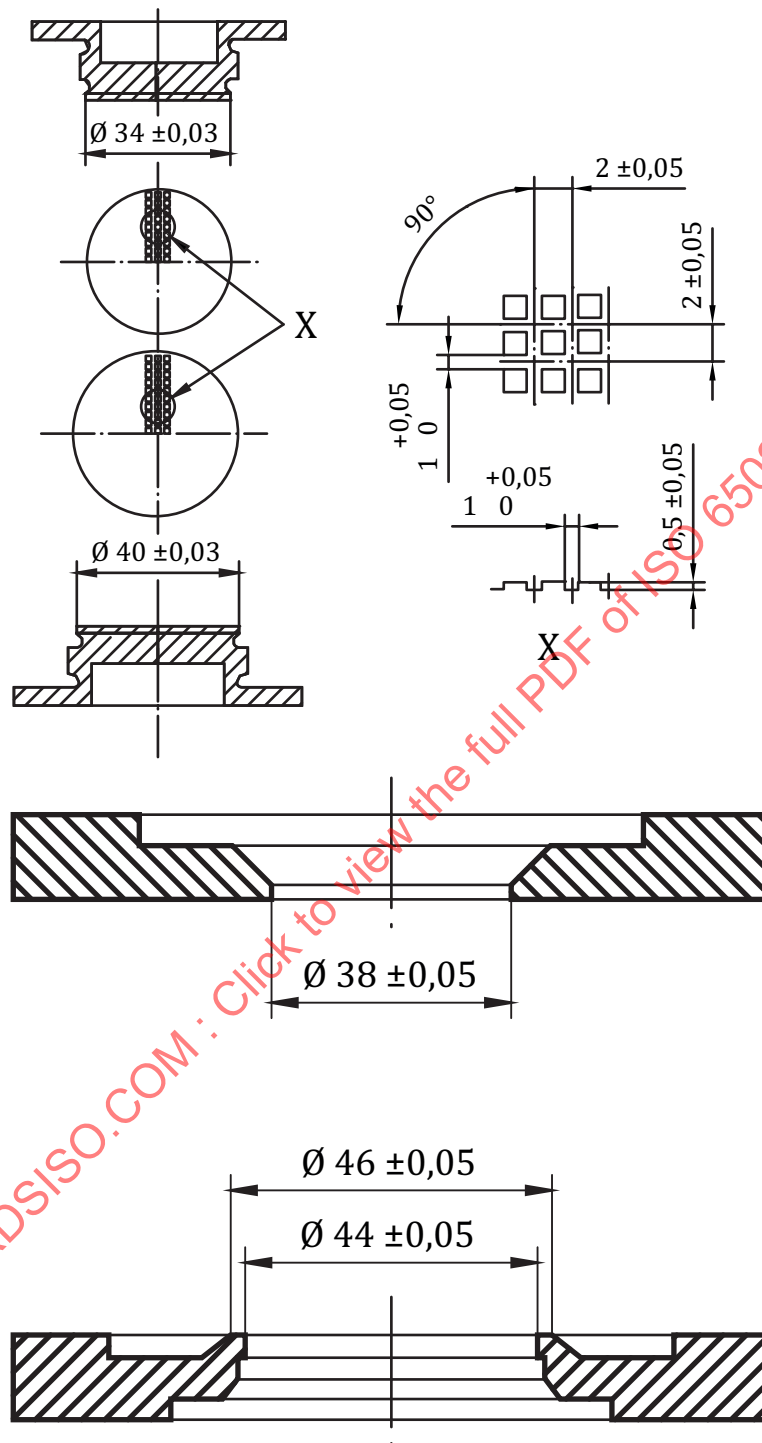


Figure 7 — Typical unsealed torsion-shear curemeter

Annex A (informative)

Effect of thermal parameters on measured cure properties

A.1 General

Curing (vulcanization) is the basic product-forming process in the rubber industry. In order to calculate the cure profile in an article, and hence the optimum cure schedule, it is necessary to know the relationship between cure, time and temperature for the rubber compound under isothermal conditions. In current industrial practice, the basic cure parameters for rubber are determined from measurements made on curemeters. These instruments measure a property, which can be called stiffness, which is approximately proportional to the hot-shear modulus. The sample has a stiffness before any crosslinking takes place, and stiffness increases from this minimum value to a maximum during the course of the curing reaction, giving rise to the familiar S-shaped curve. The cure time at a given temperature is the time taken to reach 90 %, say, of this stiffness change. The material should be characterized by making measurements at different temperatures.

A.2 Deviation from isothermal conditions

The majority of curemeters do not operate under isothermal conditions. In any instrument, this deviation is a result of the finite time required for the sample to reach thermal equilibrium. During this heating-up period, the stiffness decreases [see [Figure 1 b](#)]. As a rule of thumb, a 1 mm-thick sheet heated from both surfaces would take about 6 s to heat up. Time increases as the square of the thickness, and a 6 mm-thick sheet would take about 4 min. The heating-up time for a particular curemeter thus depends on the thickness of the test piece. It becomes more important at higher temperatures because the time needed to heat the sample is a larger fraction of the cure time.

There is an additional problem with curemeters with a rotor. Heat is lost continuously by conduction from the rotor down the drive shaft, causing the rotor to be cooler than the platens. The average temperature of the test piece is, therefore, less than the set temperature, and the curing process is slower.

The gap between the platens in a rotorless curemeter should be set so that the test piece remains under a positive pressure during the measurement. This is not only to eliminate problems with porosity, but also because it has been reported that lack of pressure gives cure times which are too long.^[1] Pressure may have a direct effect on the curing-reaction rate, or it may affect the heat transfer between the platen surfaces and the test piece.

If the curemeters are only being used for quality control, the above considerations may not matter, unless results from different types of instrument are being compared. To accommodate this, the following action may be carried out. To obtain vulcanization characteristics comparable with those obtained with the oscillating-disc curemeter described in ISO 3417, the heater of the rotorless curemeter should be adjusted so as to heat the test piece to the specified temperature in 6 min. This has given comparable results with a number of compounds of medium filler content of the most important rubber types. This procedure should only be adopted with extreme caution.

If, on the other hand, curemeter results are to be used for calculating cure times, operators should be aware of the size and effect of any deviations from isothermal conditions.^[2] For example, at 150 °C an oscillating-disc curemeter can overestimate the cure time by a factor of about two.^{[3][4]} The factor increases at higher temperatures. The relationship is not linear and should be determined experimentally.