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Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter

Caoutchouc — Détermination des caractéristiques de vulcanisation à l'aide du rhéomètre à disque oscillant

Reference number
ISO 3417:1991(E)

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.

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 3417 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 3417:1977), to which has been added an annex concerning the precision of the method specified.

Annex A of this International Standard is for information only.

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Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter

1 Scope

This International Standard specifies a method for determining selected vulcanization characteristics of a rubber compound by means of the oscillating disc curemeter.

2 Principle

2.1 A test piece of rubber is contained in a sealed test cavity under an initial positive pressure and maintained at an elevated temperature. A biconical disc is embedded in the test piece and is oscillated through a small rotary amplitude. This action exerts a shear strain on the test piece and the force (torque) required to oscillate the disc depends on the stiffness (shear modulus) of the rubber. The torque is recorded autographically as a function of time.

Direct proportionality between torque and stiffness cannot be expected under all test conditions because — particularly in higher torque ranges — an elastic deformation of disc shaft and driving device must be taken into account. Moreover, in cases of small amplitudes of deformation, the strain can be expected to have a considerable elastic component. For routine control purposes, corrections are not necessary.

2.2 The stiffness of the rubber test piece increases as vulcanization proceeds. The curve is complete when the recorded torque rises either to an equilibrium value or to a maximum value (see figure 1). The time required to obtain a vulcanization curve is a function of the test temperature and the characteristics of the rubber compound.

2.3 The following measurements can be taken from the recorded curve of torque as a function of time, i.e. $M = f(t)$ (see figure 1).

M_L: minimum torque;

t_{sx}: time to incipient cure (scorch time);

$t_c(y)$: time to a percentage of full cure;

M_{HF}: plateau torque;

M_{HR}: maximum torque (reverting curve); or

M_H: highest torque value attained in a curve where no plateau or maximum value is obtained after the specified time.

The minimum torque M_L depends on the stiffness and the viscosity at low shear rate of the unvulcanized compound.

The time to incipient cure t_{sx} is a measure of processing safety. The time to optimum cure $t_c(y)$ is measured at some percentage y of the highest torque and the highest torque is a measure of the stiffness of the fully cured vulcanizate at the test temperature. The cure rate index (see 8.4) is the average slope of the rising cure curve.

3 Apparatus

3.1 Curemeter

The curemeter consists of a biconical disc in a temperature-controlled die cavity. The shaft of the disc is secured in a drive shaft and oscillated through a small rotary amplitude.

The torque applied to the disc represents the resistance of the rubber test piece to deformation and is recorded autographically to yield a curve of torque versus time. The die-disc relationship is shown in figure 2.

3.2 Die cavity

3.2.1 The dies shall be manufactured from a non-deforming tool steel having a minimum Rockwell hardness of 50 HRC.

The geometry of the dies is shown in figure 3 and figure 4. Suitable means shall be employed by design of dies or otherwise to apply pressure on the test piece throughout the test in order to minimize slippage between the disc and the rubber. Holes shall be drilled in both the upper and lower die member according to the dimensions given in figure 3 and figure 4 to enable temperature sensors to be inserted. Surfaces of the die cavity shall contain rectangular shaped grooves located at 20° intervals to minimize slippage. The lower die dimensions are given in figure 3. The upper die member shall contain identically shaped grooves. The dimensions of the upper die are given in figure 4.

3.2.2 The lower die shall have a hole in the centre to allow for insertion of the disc stem. A suitable low constant friction seal shall be provided in this hole to prevent material from leaking from the cavity.

3.3 Die closure

The dies shall be closed and held closed during the test by a pneumatic cylinder with a force of 11,0 ± 0,5 kN.

3.4 Disc

The biconical disc shall be fabricated from a non-deforming tool steel having a minimum Rockwell hardness of 50 HRC. The disc is shown in figure 5 and the critical dimensions are given in table 1.

3.5 Disc oscillation

The frequency of the rotary oscillation of the disc shall be 1,7 Hz ± 0,1 Hz except for particular purposes when other frequencies in the range 0,05 Hz to 2 Hz may be used. The maximum angular displacement of the disc shall be 1,00° ± 0,02° about its central position (total amplitude 2°) when the die cavity is empty. If a torque is acting on the disc, the resulting decrease in the angle of oscillation with increasing torque should be a linear function having a slope within the limits of 0,05°/N·m ± 0,002°/N·m. Suitable devices shall be provided to verify both the initial amplitude of oscillation and the decrease in amplitude with applied torque.

Other amplitudes may be used when specified for particular purposes. With different frequencies or amplitudes, different results will be obtained.

NOTE 1 An initial amplitude of oscillation of 3° can be used in cases where danger of slippage between test piece and die cavity or disc can be excluded (first of all by regular cleaning of the rotor, see 7.2.3). A higher sensitivity in testing may be obtained at this amplitude, which may be useful in production control.

3.6 Torque-measuring system

3.6.1 Measurement

A device which produces a signal that is directly proportional to the torque required to turn the disc shall be used to measure the torque on the disc.

3.6.2 Recording

A recorder shall be used to record the signal from the torque-measuring device. The recorder shall have a speed of response for full-scale deflection on the torque scale of 1 s or less. The torque shall be recorded with an accuracy of ± 0,5 % of the torque range. Three torque ranges from 0 to 2,5 N·m, 0 to 5 N·m and 0 to 10 N·m shall be provided.

3.7 Temperature measurement

3.7.1 The temperature-measuring systems shall enable the temperature of the dies to be measured to within ± 0,1 °C. Calibrated thermocouples, or other suitable temperature sensors inserted in the dies, shall be used for periodically checking the die temperatures.

3.7.2 The dies shall be mounted in electrically heated aluminium platens. Temperature controllers shall be used for controlling the temperature of each platen to within ± 0,3 °C at steady state. If the temperature of the dies is set at 150 °C ± 0,3 °C, the heat conductance shall suffice to cause recovery of the initial temperature to within 1,0 °C within 2 min after a test piece at 23 °C ± 5 °C is placed in the die chamber.

4 Calibration of torque transducer and recorder

4.1 Provisions shall be made for electronic verification of the recorder and torque transducer. One way is a calibration by means of a resistor incorporated in the torque-measuring circuit and which simulates an applied torque of specified value.

4.2 The torque-measuring system shall be calibrated by means of masses or by a standard torque system such as a calibrated torsion spring.

4.3 In order to detect differences between curemeters or changes with use in a single curemeter, tests on reference compounds are useful. The reference compound shall have a shear modulus equal to or greater than the production compounds being tested, and it shall be homogeneous and stable for several weeks. Several tests shall be made on calibrated curemeter(s) in good condition and a median curve established for the reference compound. Small changes with use or small differences between curemeters may be compensated for by small adjustments in the torque span control to make subsequent tests of the reference compound agree with the established median curve. If large deviations from the median curve are observed, adjustment in the torque span control is not to be used for correction. The cause of the large deviation shall be determined and necessary repairs or maintenance performed.

5 Test piece

5.1 A test piece approximately 30 mm in diameter and 12,5 mm in thickness or of the equivalent volume shall be used for each test. Preferably, the test piece should be cut from a previously sheeted sample, which shall be as free from air as practical. A total volume of 8 cm^3 for the test piece is considered optimum.

NOTE 2 In practice, the optimum sized test piece will be obtained by taking that mass of compound which will occupy the optimum volume.

5.2 Proper test piece size is assured if a small amount of compound is extruded between all edges of the dies. Oversized test pieces cool the cavity excessively during the early part of the test cycle and invalidate the test.

6 Curing temperature

Recommended curing temperatures are from 100 °C to 200 °C. Other temperatures may be used if necessary. Tolerances for these temperatures shall be ± 0,3 °C.

7 Procedure

7.1 Preparation for test

Bring the temperature of both dies (see 3.2) to the curing temperature with the disc (3.4) in place and the die cavity in the closed position. With the disc in place and the dies closed, adjust the recorder pen to the zero torque line on the chart. Position the pen to the zero time position on the chart. Calibrate the recorder if needed (see 4.1) and select the correct torque range (see 3.6.2).

7.2 Loading the curemeter

7.2.1 Open the dies, place the test piece on top of the disc and close the dies within 5 s.

When testing sticky compounds, insert some suitable thin film material below the rotor and above the test piece to keep the compound from sticking to the dies.

7.2.2 Time shall be counted from the instant the dies are closed. The disc may be oscillating (see 3.5) at zero time or started not later than 1 min after the dies are closed.

7.2.3 A deposit of material from the rubber compounds under test may build up on the disc and dies. This may affect the final torque values. It is suggested that stable compounds be tested daily to detect this occurrence. If such contamination develops, it may be removed by very light blasting with a mild abrasive. Extreme care shall be used in this operation to retain sharpness of serrations and not change dimensions. Ultrasonic cleaning or cleaning with hot solvents or non-corrosive cleaning solutions may also remove the deposit. If solvent or solution cleaning is used, the first two sets of results after cleaning shall be rejected.

8 Expression of results

The following values, where applicable, shall be taken from the cure curve:

8.1 Torque values

M_L: minimum torque, in newton metres;

M_{HF}: plateau torque, in newton metres;

M_{HR}: maximum torque (reverting curve), in newton metres;

M_H: highest torque value attained in a curve where no plateau or maximum value is obtained, in newton metres, after the specified time.

8.2 Time values

t_{sx}: time, in minutes, to an increase of x tenths of a unit of torque above M_L (see 8.3 and 8.4);

$t_c(y)$: cure time, in minutes, to y % of full torque development (see 8.4);

$t'_c(y)$: cure time, in minutes, for torque to increase from the minimum torque M_L to $M_L + 0{,}01y(M_H - M_L)$ (see 8.3).

8.3 Times to different percentages of full cure

Unless otherwise specified, it is recommended that the following specific parameters be used:

t_{s1}: time, in minutes, for torque to increase to 0,1 N·m above M_L;

$t'_c(50)$: time, in minutes, for torque to reach $M_L + 0{,}5(M_H - M_L)$;

$t'_c(90)$: time, in minutes, for torque to reach $M_L + 0,9(M_H - M_L)$.

If an amplitude of 3° is used instead of the standard 1°, t_{s2} shall be used in place of t_{s1}; i.e. the time, in minutes, for the torque to increase to 0,2 N·m above M_L.

8.4 Cure rate index

$100/[t_c(y) - t_{sx}]$: parameter proportional to the average slope of the cure rate curve in the steep region.

9 Test report

The test report shall include the following information:

a) Sample details:

1) a full description of the sample and its origin;

2) compound details.

b) Test method and test details:

1) a reference to this International Standard;

2) details of the curemeter used;

3) the dimensions of the die cavity;

4) the nominal amplitude of oscillation, reported as half of total displacement, i.e. 1° for a total displacement of 2°;

5) the frequency of oscillation, in hertz, if this is not the preferred value (see 3.5);

6) the torque range selected, in newton metres;

7) the paper feed speed of the recorder, in millimetres per minute;

8) the heating-up time, in minutes;

9) the curing temperature, in degrees Celsius.

c) Test results read from the vulcanization curve:

M_L: minimum torque, in newton metres;

M_{HR}: maximum torque, in newton metres; or

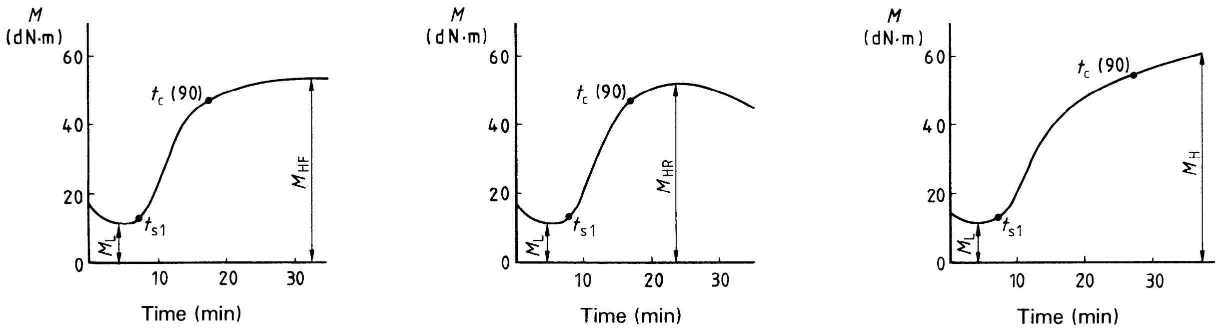
M_{HF}: plateau torque, in newton metres; or

M_H: highest torque value attained, in newton metres, in the case of a steady increase;

t_{sx}: scorch time, in minutes (time to an increase of x tenths of a unit of torque from M_L);

$t_c(y)$: cure time, in minutes.

d) The date of test.



a) Cure to equilibrium torque

b) Cure to a maximum torque with reversion

c) Cure to no equilibrium in maximum torque

Figure 1 — Types of vulcanization curve

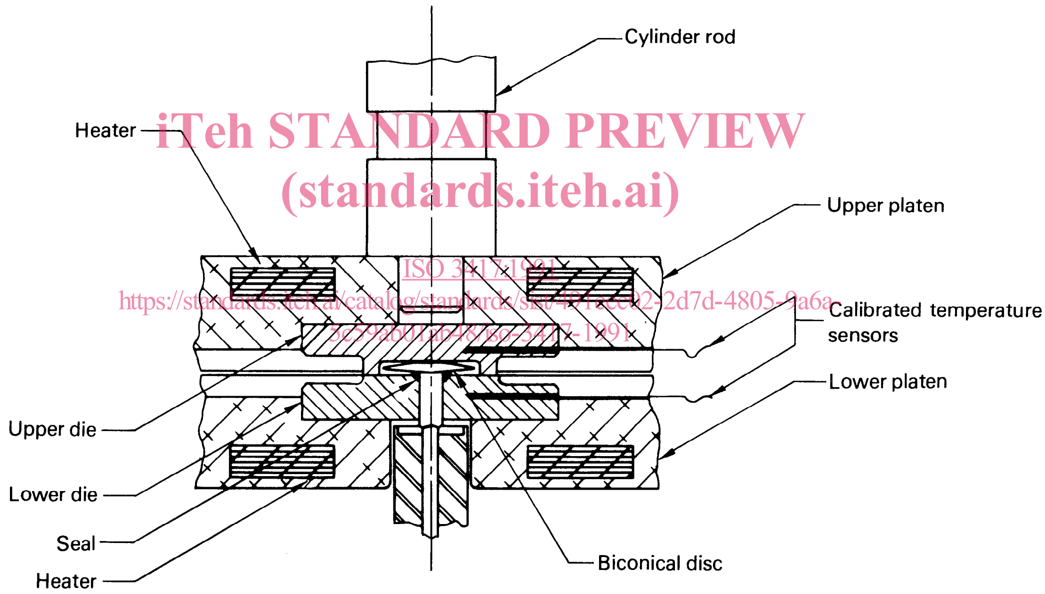


Figure 2 — Curemeter assembly

Dimensions in millimetres
(except where otherwise indicated)

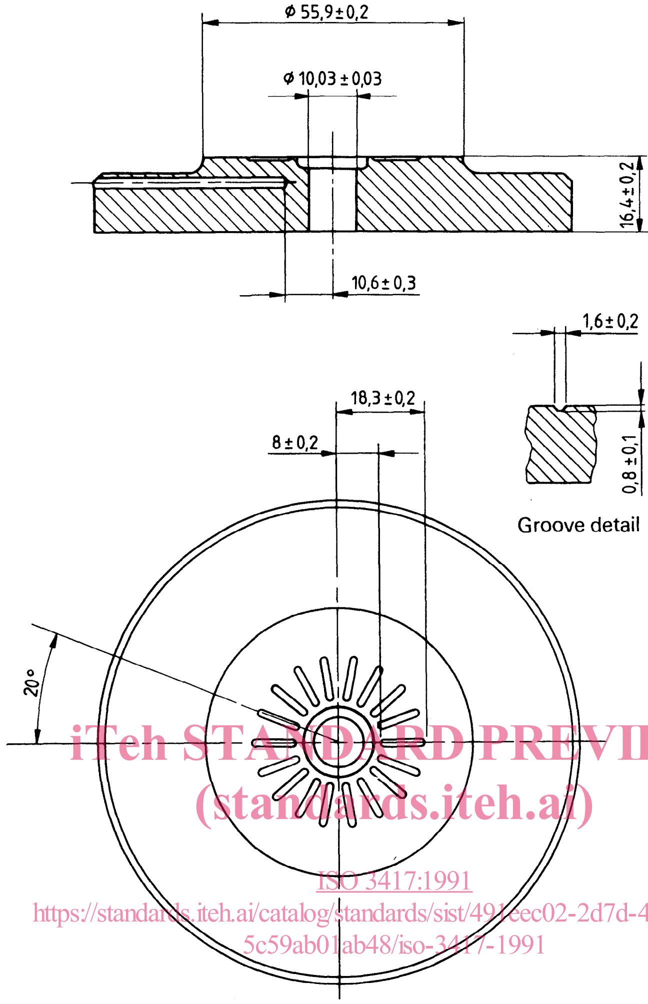


Figure 3 — Lower die

Dimensions in millimetres
(except where otherwise indicated)

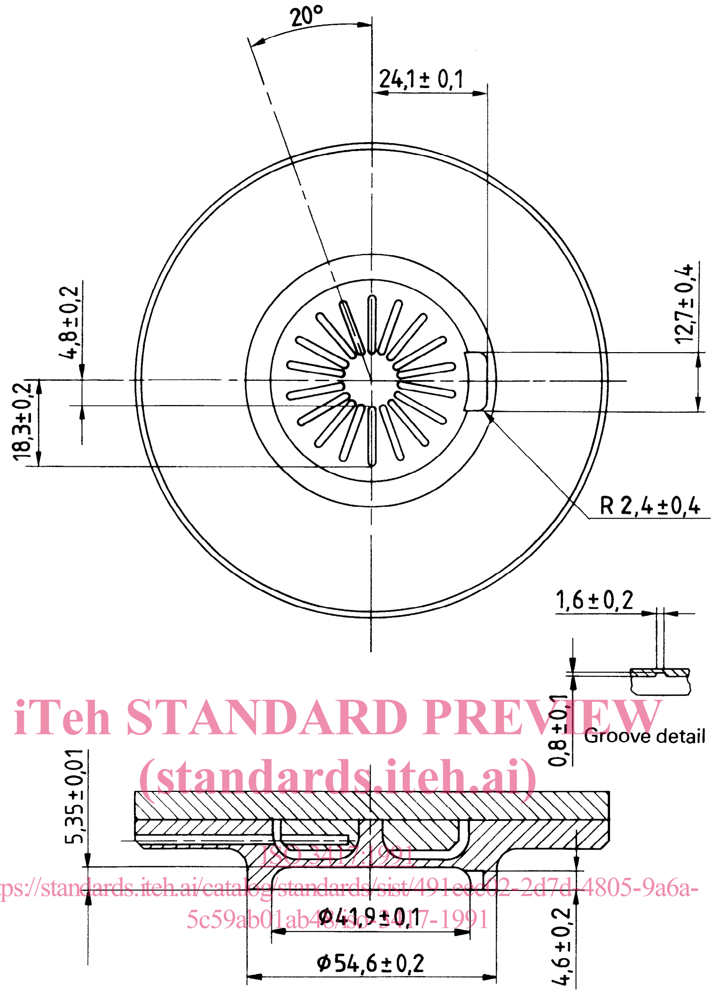


Figure 4 — Upper die