

Physical testing of rubber —

Part A60: Curemetering —

Section 60.2 Method for the determination of vulcanization characteristics using an oscillating disc curemeter

Committees responsible for this British Standard

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- British Railways Board
- British Rubber Manufacturers' Association
- GAMBICA (BEAMA Ltd.)
- Institution of Mechanical Engineers
- Institution of Water and Environmental Management
- Malaysian Rubber Producers' Research Association
- Ministry of Defence
- RAPRA Technology Ltd.
- SATRA Footwear Technology Centre
- Coopted member

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National foreword

This Section of BS 903 has been prepared under the direction of the Plastics and Rubber Standards Policy Committee. It is identical with ISO 3417 *“Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter”*, published by the International Organization for Standardization (ISO). This Section of BS 903-A60 supersedes BS 1673-10:1977, which is withdrawn.

The oscillating disc curemeter (ODC) is widely used in the rubber industry by raw material producers and manufacturers of rubber products.

Raw material producers use the information obtained from the cure curve (see Figure 1) to specify and demonstrate that their products will give the required performance.

Manufacturers of rubber products use the ODC to verify that the raw materials are satisfactory and to check the batch-to-batch consistency of mixed compounds. The ODC will give a measure of compound viscosity, scorch time, rate of cure and cross-link density.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 y 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 $11,0 \pm 0,5$ 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 $1,7 \text{ Hz} \pm 0,1 \text{ Hz}$ except for particular purposes when other frequencies in the range $0,05 \text{ Hz}$ to 2 Hz may be used. The maximum angular displacement of the disc shall be $1,00^\circ \pm 0,02^\circ$ 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 $0,05^\circ/\text{N m} \pm 0,002^\circ/\text{N m}$. 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 $\pm 0,5 \%$ of the torque range. Three torque ranges from 0 to $2,5 \text{ N m}$, 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 $\pm 0,1^\circ\text{C}$. 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 treated aluminium platens. Temperature controllers shall be used for controlling the temperature of each platen to within $\pm 0,3^\circ\text{C}$ at steady state. If the temperature of the dies is set at $150^\circ\text{C} \pm 0,3^\circ\text{C}$, the heat conductance shall suffice to cause recovery of the initial temperature to within $1,0^\circ\text{C}$ within 2 min after a test piece at $23^\circ\text{C} \pm 5^\circ\text{C}$ 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 $\pm 0,3^\circ\text{C}$.