INTERNATIONAL STANDARD

ISO
8013

First edition
1988-11-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Rubber, vulcanized — Determination of creep in compression or shear

Caoutchouc vulcanisé — Détermination du fluage en compression ou en cisaillement

Reference number
ISO 8013 : 1988 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8013 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

Annex A of this International Standard is for information only.

Introduction

When a constant stress is applied to rubber, the deformation is not constant but increases gradually with time; this behaviour is called "creep". Conversely, when rubber is subjected to a constant strain, a decrease in the stress in the material takes place; this behaviour is called "stress relaxation".

The creep test is of particular interest where vulcanized rubbers are used to support a constant load, such as in bearings or mountings.

The processes responsible for creep may be physical or chemical in nature, and under all normal conditions both processes will occur simultaneously. However, at normal or low temperatures and/or short times, creep is dominated by physical processes, whilst at high temperatures and/or long times, chemical processes are dominant. In general, physical creep is found to be directly proportional to logarithmic time, and chemical creep to linear time; but great care must be taken in extrapolating time/creep curves in order to predict creep after periods considerably longer than those covered by the test, and in using tests at higher temperatures as accelerated tests to give information on creep at lower temperatures.

In addition to the need to specify the temperature- and time-intervals in a creep test, it is also necessary to specify the initial strain and the previous mechanical history of the test piece, since these may also influence the measured creep, particularly in rubbers containing filler.

This page intentionally left blank

Rubber, vulcanized — Determination of creep in compression or shear

1 Scope

This International Standard specifies a method for the determination of creep in rubber continuously subject to compressive or to shear forces. The standard cannot be used for intermittent deformation of rubber.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 471 : 1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 815 : 1972, *Vulcanized rubbers — Determination of compression set under constant deflection at normal and high temperatures.*

ISO 1747 : 1976, *Rubber, vulcanized — Determination of adhesion to rigid plates in shear — Quadruple shear method.*

ISO 1826 : 1981, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

ISO 1827 : 1976, *Rubber, vulcanized — Determination of modulus in shear — Quadruple shear method.*

ISO 3383 : 1985, *Rubber — General directions for achieving elevated or subnormal temperatures for test purposes.*

ISO 4648 : 1978, *Rubber, vulcanized — Determination of dimensions of test pieces and products for test purposes.*

ISO 4661-1 : 1986, *Rubber, vulcanized — Preparation of samples and test pieces — Part 1: Physical tests.*

ISO 4664 : 1987, *Rubber — Determination of dynamic properties of vulcanizates for classification purposes (by forced sinusoidal shear strain).*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 creep increment: The increase in strain which occurs in a specified time-interval under constant force and at constant temperature. It is expressed as the ratio of the increase in deformation over the time-interval to the initial, unstrained thickness.

3.2 creep index: The relative increase in strain which occurs in a specified time-interval under constant force and at constant temperature. It is expressed as the ratio of the increase in the strain over the time-interval to the strain at the beginning of the interval.

3.3 compliance increment: The ratio of the increase in strain which occurs in a specified time-interval under constant force and at constant temperature to the constant stress applied to the test piece.

4 Apparatus

4.1 Thickness-measuring device.

Thickness measurements shall be made using a device complying with either ISO 4648 or the test apparatus described in 4.2. It shall be capable of measuring the test piece thickness to the nearest 0,1 mm. It shall have plates of diameter at least 30 mm. The dial gauge shall be fitted with a flat contact perpendicular to the plunger and parallel to the base plate and shall operate with a foot pressure of 22 kPa ± 5 kPa.

4.2 Compression device for measurement in compressions.

The apparatus shall consist of two parallel, flat steel plates, between which the prepared test piece is compressed. In the case of unbonded test pieces, the plates shall be highly polished with a surface finished not worse than 0,2 µm arithmetic mean deviation from the mean line of the profile. It is recommended to lubricate the operating surfaces of the plates. The test pieces shall be free from mould lubricants or dusting powder. The plates shall be sufficiently rigid to withstand the force without bending and of sufficient size to ensure that the whole of the compressed test piece is within the area of the plates.

NOTE — For most purposes, a silicone or fluorosilicone liquid having a kinematic viscosity of 0,01 m^2/s at standard temperature is a suitable lubricant.

One of the plates shall be rigidly mounted so that it does not move in any direction under the action of the compressive force. The other plate shall be able to move in a friction-free manner in one direction only, i.e. in direction coincident with the axis of the test piece (see figure 1).

The apparatus shall be capable of applying the full force with negligible overshoot and maintaining it constant to within 0,1 %. The mechanism for applying the force should be such that the line of action of the applied force remains coincident with the axis of the test piece as it creeps.

Suitable equipment should be connected to the compression device so that the deformation of the test piece may be determined, to an accuracy of ±0,1 % of the initial test piece thickness, at different times after the force has been fully applied.

Many types of apparatus have been used, with mechanical, electronic or optical measurement of deformation. Figure 4 shows a typical example using a micrometer dial gauge, for the determination of creep in compression. The measuring device shall not exert a pressure of more than 22 kPa on the test piece in the beginning.

The test piece and the flat plates of the compression device shall be inside a temperature-controlled room or an oven (see 4.4).

4.3 Shear device for measurements in shear.

The apparatus shall be capable of measuring the shear deflection in the test piece due to the application of a constant shear force.

The apparatus shall be capable of applying the full force with negligible overshoot and maintaining it constant to within 0,1 %.

The force shall be applied either to the central metal plate, with the outer plates rigidly mounted, or to the outer metal plates with the central plate rigidly mounted. The line of action of the applied force shall be in the plane of the central plate, and pass through its centre in a direction perpendicular to the undeformed rubber test pieces. This line of action shall be maintained as the test piece creeps (see figure 2).

The movement of the central plate relative to the outer plates shall be in a friction-free manner and only in the direction of the line of action of the applied force.

Suitable equipment shall be connected to the test piece, so that relative movement of the centre plate with respect to the outside plates can be determined with an accuracy of ±0,01 mm at different times after the force has been fully applied.

The test piece and the flat plates to which it is bonded shall be inside a temperature-controlled room or an oven (see 4.4).

4.4 Temperature-controlled cabinet.

An oven in accordance with ISO 3383, provided with temperature control to maintain the specified air temperature within the tolerances given in clause 8, shall be used. Satisfactory circulation of the air shall be achieved by means of a fan. Care shall be taken to minimize change in temperature of the test piece by conduction through metal parts which are connected with the outside of the oven or by direct radiation from heaters within the oven.

4.5 Timer, reading in seconds and minutes.

5 Test piece

5.1 Test piece for measurements in compression

5.1.1 The test piece shall be a cylindrical disc; two sizes may be used having the following dimensions:

— Type A shall have a diameter of 29,0 mm ± 0,5 mm and a thickness of 12,5 mm ± 0,5 mm.

— Type B shall have a diameter of 13,0 mm ± 0,5 mm and a thickness of 6,3 mm ± 0,3 mm.

NOTE — These sizes correspond to Type A and B test pieces in ISO 815.

5.1.2 The test pieces shall be prepared in accordance with ISO 4661-1 by either moulding or cutting. They shall be free from any fabric or other reinforcing support.

Cutting shall be carried out by means of a sharp rotating circular die or revolving knife, lubricated with soapy water and brought carefully into contact with the rubber. Alternatively, the die or knife may be kept stationary and the rubber rotated against it.

NOTE — When cupping is a problem, the test piece shape can be improved by cutting the test piece in two stages: first cut an oversized test piece and then trim it to the exact dimensions with a second cutter.

If bonded test pieces are required, the plane surfaces of the rubber disc shall be bonded to rigid end pieces (see figure 1). Bonding to the end pieces shall be carried out either during moulding or subsequently, using suitable adhesive that does not flow under the test conditions and avoiding the use of excessive amounts of adhesive. The thickness of the end pieces shall be determined prior to bonding.

NOTES

1 Different results will be obtained in compression with bonded and non-bonded test pieces.

2 Since rigidity is required only in a radial direction, the end pieces may be of thin metal sheet, having a minimum thickness of 0,25 mm.

5.2 Test piece for measurements in shear

The double shear test piece shall be of either circular or square cross-section and shall be bonded to rigid end plates and a rigid centre plate (see figures 2 and 3).

To avoid significant bending, the diameter (or side in the case of square test pieces) shall be at least four times the thickness. This will ensure that the deformation is essentially simple shear of the calculated magnitude and that the apparent shear modulus differs by less than 3 % from the true value.

Because of the difficulties of ensuring uniform vulcanization in thick pieces, the thickness of the vulcanized pieces should not be more than 12 mm.

Preferably, the test pieces should have a circular cross-section of 25 mm diameter and a thickness of 6,3 mm. Alternatively, the double shear test piece specified in ISO 4664, ISO 1827 or ISO 1747 may be used.

The test pieces should preferably be prepared by moulding directly on to steel plates. The thickness of the steel plates used shall be determined prior to moulding or bonding.

5.3 Number

At least three test pieces shall be used.

6 Time-lapse between vulcanization and testing

For all test purposes, the minimum time between vulcanization and testing or mechanical conditioning shall be 16 h.

For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks and, for evaluations intended to be comparable, the test, as far as possible, shall be carried out after the same time interval.

For product tests, whenever possible, the time between vulcanization and testing shall not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt of the product (see ISO 1826).

Samples and test pieces shall be protected from light and excessive heat as completely as possible during the interval between vulcanization and testing.

7 Mechanical conditioning

It is known that results are affected by the strain history of the sample and that reproducibility of the results is improved by mechanical conditioning. It is therefore preferable that the following conditioning procedure be carried out, at standard temperature:

a) strain the test piece by about 25 % ± 2 % in the same direction at a rate of 25 mm/min as in the test (see 4.2 or 4.3) and then return it to approximately zero deflection;

b) repeat step a) to give a total of five deformations.

A minimum of not less than 16 h and maximum of not more than 48 h at standard laboratory temperature shall occur between mechanical conditioning and testing.

8 Test temperature

The temperature of test will be chosen for technical reasons, but it is recommended that one of the following be used, in accordance with ISO 471, with the following tolerances:

Standard temperature, 55 °C ± 1 °C, 70 °C ± 1 °C, 85 °C ± 1 °C, 100 °C ± 1 °C, 125 °C ± 2 °C, 150 °C ± 2 °C, 175 °C ± 2 °C, 200 °C ± 2 °C, 225 °C ± 2 °C, 250 °C ± 2 °C.

Other temperatures, including sub-normal, may also be used.

9 Procedure

9.1 Testing

Bring the testing device to the test temperature for an adequate time for all parts to reach thermal equilibrium.

Keep the test piece at the specified test temperature (see clause 8) for a minimum of 30 min to reach equilibrium (see ISO 3383), then determine the initial cross-sectional area A_0.

WARNING — Testing staff should note that the testing temperatures specified present potential burn hazards.

Measure the initial thickness δ_0 of the compression test piece at the selected test temperature with an apparatus meeting the requirements of 4.1 or 4.2.

Measure the total sandwich thickness of the shear test piece at the selected test temperature and determine the thickness of rubber by subtracting the thickness of the end plates and central plate.

Mount the test piece in the testing device. Take the initial reading of the measuring device, or alternatively set the indicator to zero, depending upon the type of device used.

Apply the force to the test piece such that the full force is reached in not more than 6 s (0,1 min) and without significant overshoot.

For tests in compression and shear, the force shall be chosen such that the initial strain is 20 % ± 2 %.

NOTES

1 A trial run on a separate test piece may be necessary to determine the required force.

2 Guidance on the values of forces required for rubbers of differing hardnesses is given in annex A.

Measure the deformation of the test piece at different times after the application of the full force, this force being held constant throughout the total test time. The height δ_1 in compression or the deformation l_1 in shear shall be measured after 10 min ± 0,2 min and the height δ_2 or deformation l_2 after the times recommended in 9.2.

9.2 Duration of test

It is recommended that the measurements of δ_2 and l_2 be made on an approximately logarithmic time-scale, for example 100, 1 000 and 10 000 min, or 1, 2, 4, 7, . . . days.

10 Calculation of results

10.1 Creep increment

10.1.1 In compression

The creep increment in compression $\Delta\epsilon$ is given by the equation

$$\Delta\epsilon = \epsilon_2 - \epsilon_1$$

$$= \frac{\delta_1 - \delta_2}{\delta_0}$$

with

$$\epsilon_1 = \frac{\delta_0 - \delta_1}{\delta_0}$$

$$\epsilon_2 = \frac{\delta_0 - \delta_2}{\delta_0}$$

where

ϵ_1 is the compression strain of the compression test piece 10 min after application of the force;

ϵ_2 is the compression strain of the compression test piece after the specified test duration;

δ_0 is the initial thickness, in millimetres, of the test piece at the test temperature (see figure 1);

δ_1 is the thickness, in millimetres, of the compression test piece compressed under constant force 10 min after application of the force;

δ_2 is the thickness, in millimetres, of the compression test piece after the specified test duration.

10.1.2 In shear

The creep increment in shear $\Delta\gamma$ is given by the equation

$$\Delta\gamma = \gamma_2 - \gamma_1$$

$$= \frac{l_2 - l_1}{\delta_0}$$

with

$$\gamma_1 = \frac{l_1}{\delta_0}$$

$$\gamma_2 = \frac{l_2}{\delta_0}$$

where

γ_1 is the shear strain of the double shear test piece 10 min after application of the constant shear force;

γ_2 is the shear strain of the double shear test piece after the specified test duration;

l_1 is the shear displacement, in millimetres, of the double shear test piece under constant shear force 10 min after application of the force;

l_2 is the shear displacement, in millimetres, of the double shear test piece after the specified test duration;

δ_0 has the same meaning as in 10.1.1.

10.2 Creep index

10.2.1 In compression

The creep index in compression $\frac{\Delta\epsilon}{\epsilon_1}$ is given by the equation

$$\frac{\Delta\epsilon}{\epsilon_1} = \frac{\delta_1 - \delta_2}{\delta_0 - \delta_1}$$

where $\Delta\epsilon$, ϵ_1, δ_0, δ_1 et δ_2 have the same meaning as in 10.1.1.

10.2.2 In shear

The creep index in shear $\frac{\Delta\gamma}{\gamma_1}$ is given by equation

$$\frac{\Delta\gamma}{\gamma_1} = \frac{l_2 - l_1}{l_1}$$

where $\Delta\gamma$, γ_1, l_1 and l_2 have the same meaning as in 10.1.2.

10.3 Compliance increment

10.3.1 In compression

The compliance increment in compression I_ϵ, expressed in square millimetres per newton, is given by the equation

$$I_\epsilon = \frac{\Delta\epsilon}{\sigma_0}$$

$$= \frac{\delta_1 - \delta_2}{\sigma_0\delta_0}$$

with

$$\sigma_0 = \frac{F_c}{A_0}$$

where

σ_0 is the compressive stress, in megapascals, acting on the compression test piece;

F_c is the constant compressive force, in newtons, acting on the compression test piece;

A_0 is the initial cross-section, in square millimetres, of the test piece;

$\Delta\epsilon$, δ_0, δ_1 and δ_2 have the same meaning as in 10.1.1.

10.3.2 In shear

The compliance increment in shear I_γ, expressed in square millimetres per newton, is given by the equation

$$I_\gamma = \frac{\Delta\gamma}{\tau_0}$$

$$= \frac{l_2 - l_1}{\tau_0\delta_0}$$

with

$$\tau_0 = \frac{F_s}{A_0}$$

where

τ_0 is the shear stress, in megapascals, acting on the double shear test piece;

F_s is half the constant shear force, in newtons, acting on the double shear test piece;

A_0 has the same meaning as in 10.3.1;

$\Delta\gamma$, l_1 and l_2 have the same meaning as in 10.1.2;

δ_0 has the same meaning as in 10.1.1.

11 Expression of results

Report the median from the individual results obtained of the creep increment, creep index and compliance increment (as required).

If required, report the range or the individual results.

If an individual result differs from the median by more than 10 %, test three further test pieces and report the median of all the results obtained (and range or individual results, if required).

NOTE — If the deformation shows a linear dependence when plotted against the logarithm of time, it is common practice to quote the results as the slope of this line, i.e. creep index rate (see 3.2).

12 Test report

The test report shall include the following particulars:

a) sample details:

1) full description of the sample and its origins,

2) compound details and curing conditions, if known,

3) preparation of test pieces, for example whether moulded or cut,

4) test piece bonded or not bonded to metal plates,

5) any relevant fact about the pre-test history of test pieces,

6) test piece dimensions;

b) test method and test details:

1) reference number of this International Standard,

2) type of apparatus used and whether shear or compression,

3) test temperature,

4) time and temperature of conditioning and details of mechanical conditioning used,

5) standard temperature (23 °C or 27 °C);

6) any non-standardized procedures adopted;

c) test results:

1) number of test pieces used,

2) calculated median values of creep increment, creep index and compliance increment,

3) range or individual test results, if required;

d) date of test.