INTERNATIONAL STANDARD

ISO 8013

Third edition 2012-10-01

Rubber, vulcanized — Determination of creep in compression or shear

Caoutchouc vulcanisé — Détermination du fluage en compression ou en cisaillement



Reference number ISO 8013:2012(E)

ISO 8013:2012(E)



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ISO 8013:2012(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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8013 was prepared by Technical Committee ISO/TC 45, Rubber and rubber products, Subcommittee SC 2, Testing and analysis.

This third edition cancels and replaces the second edition (ISO 8013:2006), which has been revised to include a calibration schedule for the apparatus used (see Annex B). In addition, the maximum thickness of test pieces for measurement in shear (see 6.2) has been increased from 12 mm to 13 mm.

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 can 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 has to 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 intervals 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 might also influence the measured creep, particularly in rubbers containing filler.

Rubber, vulcanized — Determination of creep in compression or shear

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

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1827, Rubber, vulcanized or thermoplastic — Determination of shear modulus and adhesion to rigid plates — Quadruple shear methods

ISO 4664-1, Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance

ISO 18899:2004, Rubber — Guide to the calibration of test equipment

ISO 23529, Rubber — General procedures for preparing and conditioning test pieces for physical test methods

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

creep increment

increase in strain which occurs in a specified time interval under constant force and at constant temperature

NOTE It is expressed as the ratio of the increase in deformation over the time interval to the initial, unstrained thickness.

3.2

creep index

relative increase in strain which occurs in a specified time interval under constant force and at constant temperature

NOTE 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

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

Apparatus

Thickness-measuring device 4.1

Thickness measurements shall be made using a device complying with either ISO 23529 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 \pm 5) kPa.

Compression device for measurement in compression

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 to not worse than 0,2 µm arithmetic mean deviation from the mean line of the profile. It is recommended that the operating surfaces of the plates be lubricated. 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 m²/s at standard laboratory 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 a direction coincident with the axis of the test piece (see Figures 1 and 2).

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 shall 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 shall be connected to the compression device so that the deformation of the test piece can 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 2 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 before the test load is applied.

If the tests are carried out at an elevated temperature, the test piece and the flat plates of the compression device shall be inside a temperature-controlled chamber (see 4.4).

Shear device for measurements in shear 4.3

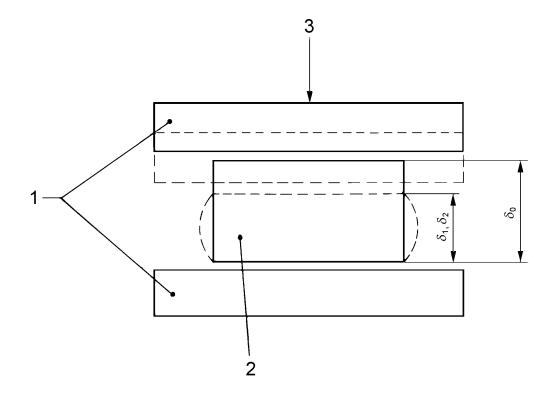
The apparatus shall be capable of measuring the shear deflection in the test piece due to the application of a constant shear force.

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 3).

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 central plate with respect to the outside plates can be determined with an accuracy of \pm 0,01 mm at different times after the force has been fully applied.



- 1 steel plates
- 2 test piece
- 3 line of action of compressive force
- δ_0 initial thickness

Figure 1 — Test piece in compression

If the tests are carried out at an elevated temperature, the test piece and the flat plates to which it is bonded shall be inside a temperature-controlled chamber (see 4.4).

Figure 4 shows a typical shear test fixture.

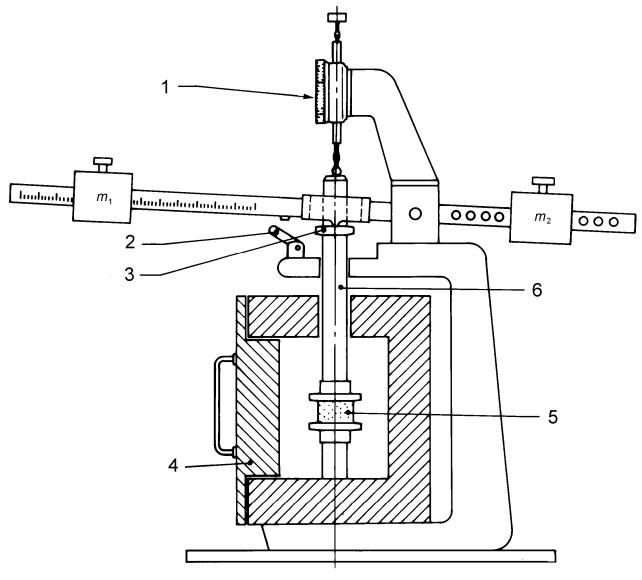
4.4 Temperature-controlled chamber

If the tests are to be carried out at an elevated temperature, a test chamber shall be used, constructed in accordance with ISO 23529, and provided with temperature control to maintain the specified air temperature within the tolerances given in Clause 9. 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 chamber or by direct radiation from heaters within the chamber.

4.5 Timer

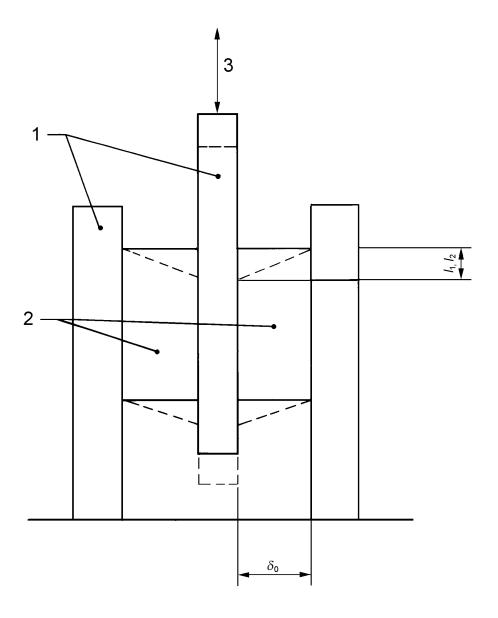
Use a timer reading in seconds and minutes.

3



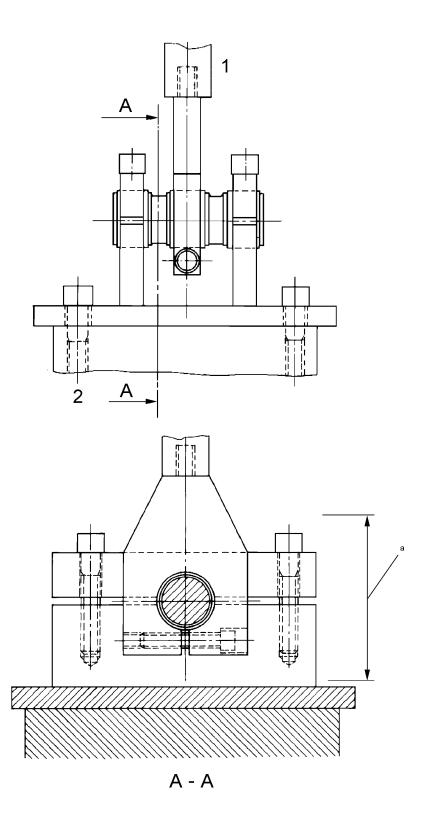
- dial gauge 1
- 2 locking lever
- 3 setting ring
- temperature-controlled chamber 4
- 5 test piece
- ceramic bar 6

Figure 2 — Example of test arrangement for creep in compression with temperature-controlled chamber



- 1 steel plates
- 2 bonded test pieces
- 3 line of action of shear force
- δ_0 initial thickness

Figure 3 — Test piece in shear



- actuator
- load cell
- Shall be in test chamber.

Figure 4 — Typical test fixture for double-shear test piece

5 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in Annex B.

6 Test piece

6.1 Test piece for measurements in compression

- **6.1.1** 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 \pm 0,5) mm and a thickness of (12,5 \pm 0,5) mm.
- Type B shall have a diameter of (13,0 \pm 0,5) mm and a thickness of (6,3 \pm 0,3) mm.

NOTE These sizes correspond to type A and B test pieces in ISO 815-1^[1].

6.1.2 The test pieces shall be prepared in accordance with ISO 23529 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. Bonding to the end pieces shall be carried out either during moulding or subsequently, using a 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.

The test pieces shall be free from mould lubricants and dusting powder.

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

NOTE 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.

6.2 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 3 and 4).

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 shall not be more than 13 mm.

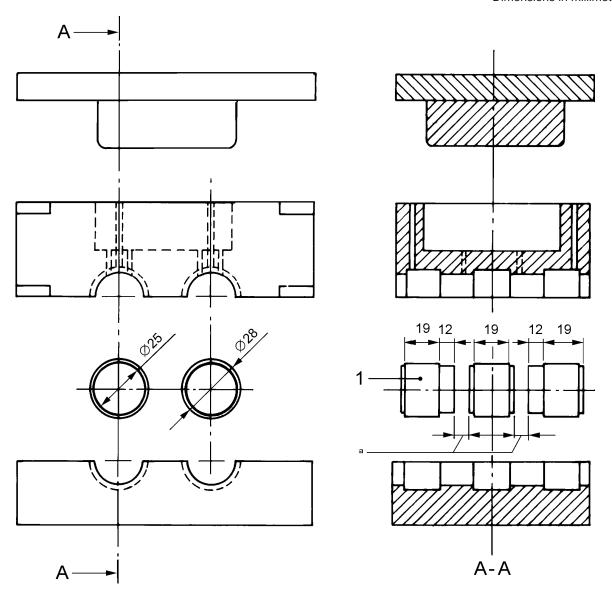
Preferably, the test pieces shall have a circular cross-section of 25 mm diameter and a thickness of 6,3 mm. Alternatively, the double-shear test piece specified in ISO 4664-1 or ISO 1827 may be used.

The test pieces shall preferably be prepared by moulding (see Figure 5) directly on to steel plates. The thickness of the steel plates used shall be determined prior to moulding or bonding.

6.3 Number

At least three test pieces shall be used.

Dimensions in millimetres



Key

- metal parts
- Thickness of rubber block.

Figure 5 — Mould for double-shear test piece

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.

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

8 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 a standard laboratory temperature:

- a) strain the test piece by about (25 ± 2) % at a rate of 25 mm/min in the same direction 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 be allowed to elapse between mechanical conditioning and testing.

9 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 23529, with the following tolerances:

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

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

10 Procedure

10.1 Testing

Bring the test 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 9) for a minimum of 30 min to reach equilibrium (see ISO 23529), then determine the initial cross-sectional area A_0 .

WARNING — Operators should note that the test 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 laboratory temperature or at the selected test temperature and determine the thickness of rubber by subtracting the thicknesses of the end plates and central plate.

Mount the test piece in the test 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) %.

- NOTE 1 A trial run on a separate test piece might be necessary to determine the required force.
- NOTE 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 \pm 0,2) min and the height δ_2 or deformation l_2 after the times recommended in 10.2.

10.2 Duration of test

It is recommended that the measurements of δ_2 and δ_1 be made on an approximately logarithmic time-scale, for example 100 min, 1 000 min and 10 000 min, or 1 day, 2 days, 4 days, 7 days, ... days.

11 Calculation of results

11.1 Creep increment

11.1.1 In compression

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

$$\Delta \varepsilon = \varepsilon_2 - \varepsilon_1 = \frac{\delta_1 - \delta_2}{\delta_0}$$

with

$$\varepsilon_1 = \frac{\delta_0 - \delta_1}{\delta_0}$$

$$\varepsilon_2 = \frac{\delta_0 - \delta_2}{\delta_0}$$

where

- is the compression strain of the compression test piece 10 min after application of the force;
- is the compression strain of the compression test piece after the specified test duration; *E*2
- is the initial thickness, in millimetres, of the test piece at the test temperature (see Figure 1); δ_0
- is the thickness, in millimetres, of the compression test piece compressed under constant force 10 min after application of the force;
- is the thickness, in millimetres, of the compression test piece after the specified test duration.

11.1.2 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

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;
- l₁ is the shear displacement, in millimetres, of the double-shear test piece under constant shear force 10 min after application of the force;
- l2 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 11.1.1.

11.2 Creep index

11.2.1 In compression

The creep index in compression $\frac{\Delta \varepsilon}{\varepsilon_1}$ is given by the equation

$$\frac{\Delta \varepsilon}{\varepsilon_1} = \frac{\delta_1 - \delta_2}{\delta_0 - \delta_1}$$

where $\Delta \varepsilon$, ε_1 , δ_0 , δ_1 and δ_2 have the same meanings as in 11.1.1.

11.2.2 In shear

The creep index in shear $\frac{\Delta \gamma}{\gamma_1}$ is given by the 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 meanings as in 11.1.2.

11.3 Compliance increment

11.3.1 In compression

The compliance increment in compression I_{ε} , expressed in square millimetres per newton, is given by the equation

$$I_{\varepsilon} = \frac{\Delta \varepsilon}{\sigma_0} = \frac{\delta_1 - \delta_2}{\sigma_0 \delta_0}$$

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;
- $\Delta\varepsilon$, δ_0 , δ_1 and δ_2 have the same meanings as in 11.1.1.

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11.3.2 In shear

The compliance increment in compression 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

is the shear stress, in megapascals, acting on the double-shear test piece; τ \cap

is half the constant shear force, in megapascals, acting on the double-shear test piece; F_{S}

has the same meaning as in 11.3.1; A_0

 Δ_{γ} , l_1 and l_2 have the same meanings as in 11.1.2;

has the same meaning as in 11.1.1.

12 Expression of results

Report the median of the individual results obtained for 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).

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

13 Test report

The test report shall include the following particulars:

- sample details:
 - a full description of the sample and its origin,
 - the compound details and curing conditions, if known,
 - 3) details of preparation of the test pieces, for example whether moulded or cut,
 - whether the test piece was bonded or not bonded to metal plates, 4)
 - 5) any relevant fact about the pre-test history of the test pieces,
 - the test piece dimensions; 6)
- test method: b)
 - the reference number of this International Standard,

- 2) the type of apparatus used and whether it was a shear or compression apparatus;
- c) test details:
 - 1) the test temperature,
 - 2) the time and temperature of conditioning and details of any mechanical conditioning carried out,
 - 3) the standard laboratory temperature used (23 °C or 27 °C),
 - 4) details of any procedure not specified in this International Standard;
 - 5) the number of test pieces tested;
- d) test results:
 - 1) the range of the test results or the individual test results, if required;
 - 2) the calculated median values of the creep increment, creep index and compliance increment;
- e) the date of testing.

Annex A

(informative)

Values of forces required for rubbers of different hardnesses

A.1 In compression

The forces in Table A.1 give a compression of approximately 20 %.

Table A.1 — Compression force on test piece

Hardness	Compression force on test piece		
	Type A	Type B	
IRHD	N	N	
35 to 44	430	80	
45 to 54	540	100	
55 to 64	750	140	
65 to 75	1 200	220	
76 to 85	3 300	600	
86 to 95	7 300	1 300	

A.2 In shear

In the case of a shear strain $\gamma = 0.2$ and a double-shear test piece with a diameter of 25 mm, the forces given in Table A.2 should be suitable.

Table A.2 — Shear force

Hardness	Shear force		
IRHD	N		
35 to 44	90		
45 to 54	130		
55 to 64	210		
65 to 75	340		
76 to 85	950		
86 to 95	2 000		

Annex B

(normative)

Calibration schedule

B.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded on any calibration report or certificate. It shall be reported whether calibration is carried out in the "as-received" condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

B.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table B.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code-letter. The code-letters used in the calibration schedule are:

- C requirement to be confirmed, but no measurement;
- N initial verification only;
- S standard interval as given in ISO 18899;
- U in use.

Table B.1 (continued)

Parameter	Requirement	Subclause in ISO 18899:2004	Verification frequency guide	Notes	
Time during which test piece kept at test temperature prior to loading	≥ 30 min, see ISO 23529	С	U		
Environment	Temperature control within tolerance given in ISO 23529	18	S		
Change in temperature of test piece	To be a minimum (protect from radiation, conduction, etc.)	С	U		
	Air circulation with fan	С	N		
Test temperature	As per ISO 23529			See Clause 9	
	Below 125 °C: tolerance ±1 °C	18	S		
	125 °C and above: tolerance ±2 °C	18	S		
Materials	Lubricant, e.g. silicone or fluorosilicone oil of kinematic viscosity 0,01 m ² /s at standard laboratory temperature.				
	Adhesive for bonding that does not flow under test conditions.				

In addition to the items listed in Table B.1, use of the following is implied, all of which need calibrating in accordance with ISO 18899:

- timer;
- thermometers for monitoring the conditioning and test temperatures;
- instruments for determining the dimensions of the test piece.

Bibliography

[1] ISO 815-1, Rubber, vulcanized or thermoplastic — Determination of compression set — Part 1: At ambient or elevated temperatures

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