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Rubber, vulcanized or thermoplastic — Determination of tension set under constant elongation, and of tension set, elongation and creep under constant tensile load

Caoutchouc vulcanisé ou thermoplastique — Détermination de la déformation rémanente sous allongement constant et de la déformation rémanente, de l'allongement et du fluage sous charge constante de traction

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Foreword

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This eighth edition cancels and replaces the seventh edition (ISO 2285:2013), of which it constitutes a minor revision. A few editorial changes have been made including updating the publication dates of normative references in Clause 2.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber, vulcanized or thermoplastic — Determination of tension set under constant elongation, and of tension set, elongation and creep under constant tensile load

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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 determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies a number of methods of determining the dimensional changes in test pieces of vulcanized or thermoplastic rubber during and after tensile loading for relatively short periods under constant elongation or constant loading.

The constant-elongation test is intended to measure the ability of rubbers to retain their elastic properties after extension, at a standard laboratory temperature, to a specified strain which is maintained for a specified time at the same or at a specified higher temperature and then released at the test temperature or at the standard laboratory temperature.

The constant-load test specifies a method for the determination of elongation, creep and tension set of rubbers subjected to a constant load at standard laboratory temperature.

The test methods are intended to measure the elastic properties of rubber in the hardness range 20 IRHD to 94 IRHD.

The creep measurement is not intended for product design or the evaluation of low-creep materials. For these, ISO 8013 applies, and there is no agreement between the results of this test and those of ISO 8013.

NOTE The constant-load test is primarily intended for the measurement of state of cure and the quality control of thin-walled products. An increase in the state of cure or degree of crosslinking is usually reflected in a decrease in set, creep or elongation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 188, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

ISO 8013, *Rubber, vulcanized — Determination of creep in compression or shear*

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

ISO 23529:2016, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8013 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
tension set at constant load
elongation remaining in a test piece in the unloaded condition after it has been subjected to a constant load during a specified time, expressed as a percentage of the original length in the unloaded condition

3.2
tension set at constant elongation
elongation remaining in a test piece in the relaxed condition after it has been subjected to a constant elongation during a specified time, expressed as a percentage of the elongation

3.3
creep
increase in elongation of a test piece at constant load during a specified time, expressed as a percentage of the elongation at the beginning of that time

4 Apparatus

4.1 Constant-elongation measurements

4.1.1 Straining device, consisting of a metal rod or other suitable guide fitted with pairs of holders, one fixed and one moveable, for the ends of the test piece. The holders shall be in the form of self-tightening clamps for strip test pieces, in the form of jaws to hold tab (enlarged) ends, and in the form of flat pulleys of about 5 mm width and 10 mm diameter for ring test pieces.

If so desired, a means of operating the moving holder other than by hand may be provided, for example a threaded rod. Suitable stops or graduations may also be provided to avoid over-extension in the initial straining of the test piece.

The straining device shall be so designed that, when used at high temperatures in an oven, it can be placed with the reference length of the test piece perpendicular to the direction of the air flow. It shall also be of minimum mass in order to avoid excessive lag in the attainment of temperature equilibrium after its introduction into the oven.

A multiple-unit straining device may be used, provided that the foregoing requirements are met.

4.1.2 Oven, conforming to the requirements of ISO 188 (if the test is to be carried out at a temperature above the standard laboratory temperature). For short heating times, controlled air flow is not necessary.

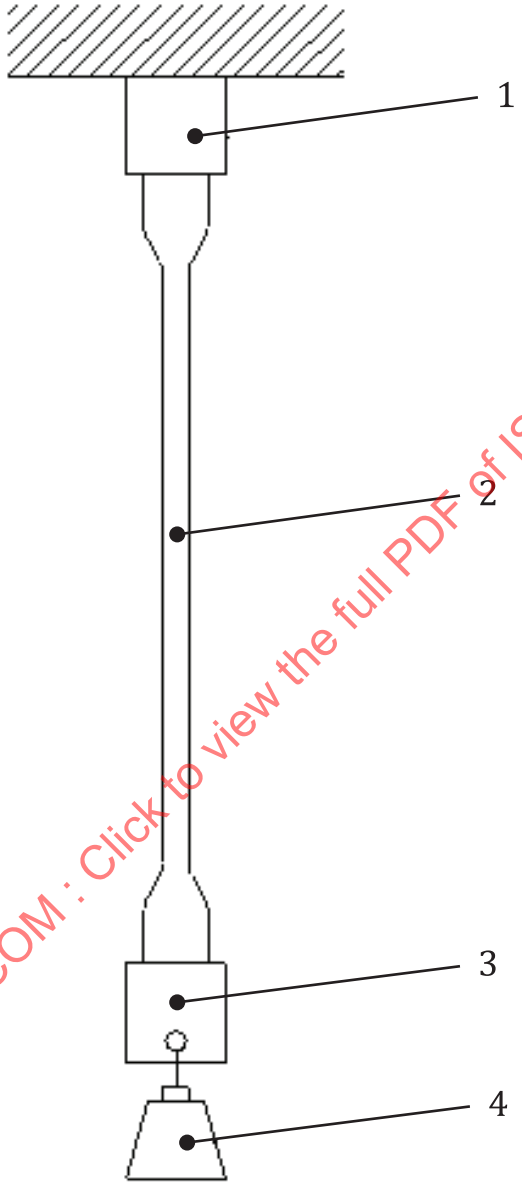
4.1.3 Length-measuring device, capable of measuring the reference length of the test piece to the nearest 0,1 mm.

For strip test pieces, a marker shall be provided to mark the length used as the reference length.

For ring test pieces, the reference length may be the inner diameter of the ring, in which case a graduated cone allowing measurements to be made to the nearest 0,1 mm shall be used. Alternatively, if measurements are to be made on a straight reference length, a rigid channel, 3,5 mm deep and 20 mm wide for large ring test pieces and 1,75 mm deep and 10 mm wide for small ring test pieces, shall be provided for straightening portions of such test pieces during marking and measuring of the reference length.

4.2 Constant-load measurements

4.2.1 Straining device, comprising clamps and weights, or equivalent, for loading the test pieces (see Figure 1).



Key

1 fixed clamp
2 test piece
3 clamp for holding weight
4 weight

Figure 1 — Straining device

4.2.2 Thickness- and width-measuring device, comprising a gauge for measuring the thickness and, where appropriate, the width of the test piece in accordance with ISO 23529:2016, method A.

The width of die-cut test pieces shall be taken as the distance between the cutting edges of the die in the narrow part, in which case a device capable of measuring the width to the nearest 0,05 mm in accordance with ISO 23529 shall be provided.

4.2.3 Length-measuring device, capable of measuring the reference length to the nearest 0,1 mm.

5 Calibration

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

6 Test pieces

6.1 Preparation

Test pieces shall be prepared in accordance with ISO 23529. They shall be cut from flat sheet 2 mm ± 0,2 mm thick (except large ring test pieces, which shall be cut from sheet 4 mm ± 0,2 mm thick) prepared by moulding or slitting and buffing in accordance with ISO 23529.

Strip test pieces and strip test pieces with enlarged ends shall be cut using a sharp die with a cutter as specified in ISO 23529.

Ring test pieces shall be cut from sheet by means of a pair of concentric circular dies or rotating cutters. The separation of the two cutting edges shall not differ from the average value by more than 0,05 mm.

6.2 Test pieces for testing under constant elongation

6.2.1 Strip test pieces

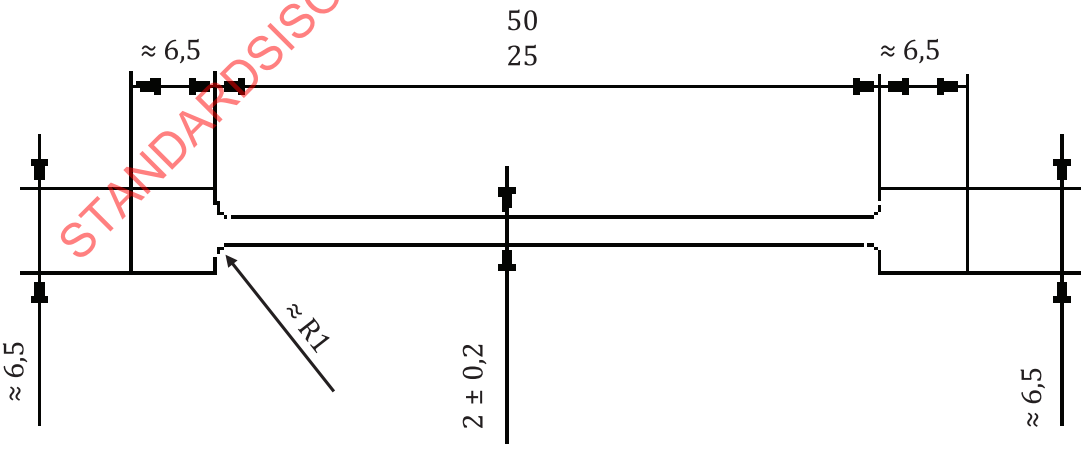
Strip test pieces shall be between 2 mm and 10 mm wide. A width of 6 mm is preferred.

The length of any given strip depends on the selected reference length and the type of straining device.

6.2.2 Strip test pieces with enlarged ends

Test pieces with enlarged ends shall be as shown in Figure 2, with a narrow section of length between 25 mm and 50 mm which shall be used as the reference length.

Dimensions in millimetres



Thickness 2 mm ± 0,2 mm

Figure 2 — Test piece with enlarged ends

6.2.3 Ring test pieces

Ring test pieces shall be of one of the following sizes:

Large ring test piece:

— thickness: 4 mm ± 0,2 mm;

— outer diameter: 52,6 mm ± 0,2 mm;

— inner diameter: 44,6 mm ± 0,2 mm.

Small ring test piece:

— thickness: 2 mm ± 0,2 mm;

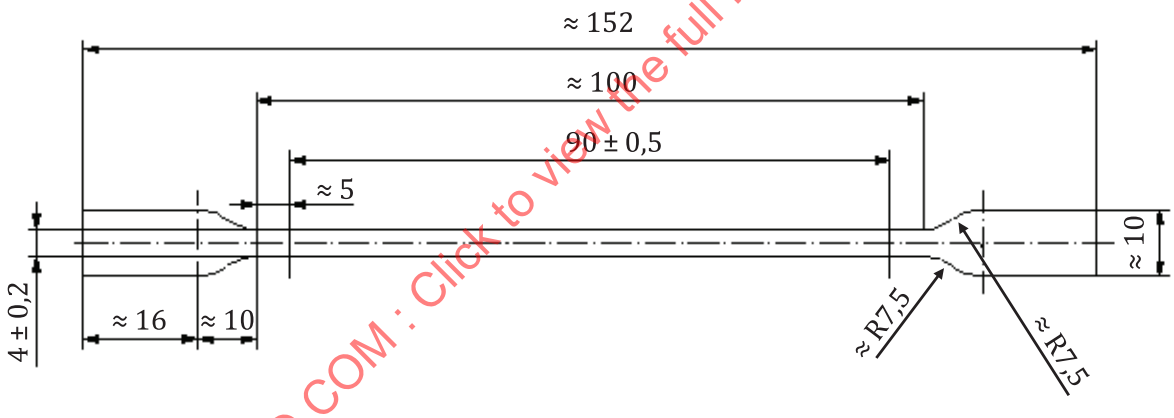
— outer diameter: 33,5 mm ± 0,2 mm;

— inner diameter: 29,5 mm ± 0,2 mm.

6.3 Test pieces for testing under constant load

Test pieces shall have the shape and dimensions shown in Figure 3.

Dimensions in millimetres



Thickness 2 mm ± 0,2 mm

Figure 3 — Test piece for constant loading

6.4 Marking

6.4.1 General

Reference marks shall be marked on the test pieces, using a suitable marker and an ink which does not affect the material and withstands the temperature of test.

6.4.2 Tests at constant elongation

The reference length shall be between 25 mm and 50 mm, measured between the inner sides of the marks. For strip test pieces, the preferred length is 50 mm. Ring test pieces shall be straightened by means of a rigid channel (see 4.1.3) and the reference length, preferably 40 mm for large ring test pieces and 25 mm for small ring test pieces, shall be marked on one of the straightened sides.

6.4.3 Tests at constant load

Two reference marks shall be marked on the central part of the test piece at a distance of 90 mm ± 0,5 mm from each other.

6.5 Number of test pieces

For each test, a minimum of three test pieces shall be used. (For material with a calendering grain, three test pieces shall be taken in each of two directions at right angles to each other.)

6.6 Time interval between forming and testing

The time interval between forming and testing shall be in accordance with ISO 23529.

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

6.7 Conditioning

Prepared test pieces shall be conditioned immediately before testing for a minimum period of 3 h at one of the standard laboratory temperatures defined in ISO 23529, the same temperature being used throughout any one test or series of tests intended to be comparable.

7 Procedure

7.1 Testing under constant elongation

7.1.1 Measurement of the test pieces

Measure the unstrained reference length (L_1) of each test piece to the nearest 0,1 mm at standard laboratory temperature. Insert the test pieces in the straining device in the appropriate way.

7.1.2 Stretching the test pieces

Extend the test pieces to the required strain. When testing ring test pieces, rotate the pulleys slightly by hand to equalize the strain in the two halves of the ring. Ensure that the reference length remains centralized between the two pulleys.

Between 10 min and 20 min after the specified strain has been reached, measure the strained reference length (L_2) to the nearest 0,1 mm. When the inner diameter of ring test pieces is used as the reference length, it can be calculated from the diameter of the pulleys and the distance between them, measured to the nearest 0,1 mm. If the strain, calculated as specified in 7.1.4.1, does not conform to the appropriate standard value, taking into account tolerances, discard the test piece and prepare and test a replacement test piece with a modified applied strain.

7.1.3 Exposure at the test temperature

7.1.3.1 Tests at standard laboratory temperature

Immediately following the end of the test period, release the strain, remove the test pieces from the clamps or pulleys and lay them free on a flat non-sticky wooden surface. After (30^{+3}_{0}) min, measure the reference length to the nearest 0,1 mm (L_5).

7.1.3.2 Tests at temperatures other than standard laboratory temperature

Between 20 min and 30 min after the specified strain has been reached, place the strained test pieces in an oven operating at the test temperature and leave them there for the specified period.

There are three methods of cooling the test pieces and releasing the strain, of which method A is preferred.

Method A

At the end of the test period, remove the straining device from the oven, immediately release the strain, remove the test pieces from the clamps or pulleys and lay them on a flat non-sticky wooden surface. After (30^{+3}_{0}) min, measure the reference length to the nearest 0,1 mm.

Method B

At the end of the test period, remove the straining device from the oven but leave the test pieces in the strained condition. After (30^{+3}_{0}) min, release the strain, remove the test pieces from the clamps or pulleys and lay them on a flat non-sticky wooden surface. After a further (30^{+3}_{0}) min, measure the reference length to the nearest 0,1 mm.

Method C

At the end of the test period, release the strain with the test pieces still in the oven held at the test temperature. After (30^{+3}_{0}) min, remove the straining device from the oven, remove the test pieces from the clamps or pulleys and lay them on a flat non-sticky wooden surface. After a further (30^{+3}_{0}) min, measure the reference length to the nearest 0,1 mm.

7.1.4 Test conditions

7.1.4.1 Strain

NOTE The rubber industry uses the term equation for the relationships herein termed formula. The term formula is used to describe the table of ingredients in a rubber compound.

Calculate the strain λ, in %, using Formula (1):

$$\lambda = (L_2 - L_1) \times \frac{100}{L_1} \quad (1)$$

where

L_1 is the original, unstrained, reference length;

L_2 is the strained reference length.

The strain shall be one of the following values:

15 % ± 1,5 %

20 % ± 2 %

25 % ± 2,5 %

50 % ± 5 %

75 % ± 7,5 %

100 % ± 10 %

200 % ± 10 %

300 % ± 10 %

The strain values should be selected in accordance with the type of rubber (vulcanized or thermoplastic) and with the final application. For vulcanized rubbers, it is recommended that elongations greater than one third of the elongation at break at the test temperature should not be used. The strain value of 100 % is the preferred one, unless the above considerations dictate otherwise. For thermoplastic rubbers which give a yield point, only results calculated at strain values below the yield point are valid. When possible, the value 20 % ± 2 % is preferred for thermoplastic rubbers.

7.1.4.2 Test period

The test pieces shall normally be strained for a period of $(24^{\ 0}_{-2})$ h, $(72^{\ 0}_{-2})$ h or $(168^{\ 0}_{-2})$ h, the period commencing 30 min after the strain has been applied. If a longer test period is required, it shall be selected from those given in ISO 23529.

7.1.4.3 Temperature of test

The temperature of test shall be selected from those given in ISO 23529.

If no temperature is specified in the relevant product specification, 70 °C ± 1 °C is recommended.

7.2 Testing under constant load

7.2.1 General

Measure the length of the test piece between the reference marks (L_1), the thickness and the width at standard laboratory temperature.

Clamp the test pieces into the straining device and load them to a stress of 2,5 MPa ± 0,1 MPa, based on the original cross-sectional area of the test piece. The load shall be applied without shock.

If, during testing, a stress of 2,5 MPa proves to be too large, a stress of 1,0 MPa may be used.

7.2.2 Elongation

30 s ± 2 s after loading the test piece, measure the distance between the reference marks (L_2).

7.2.3 Creep

60 min ± 1 min after loading the test piece, measure the distance between the reference marks (L_3).

7.2.4 Tension set

After the creep measurement, unload the test piece, remove it from the straining device and allow it to rest on a flat non-sticky wooden surface for 10 min ± 1 min. Measure the distance between the reference marks again (L_4).

8 Expression of results

8.1 General

Calculate the individual test results as shown in 8.2 or 8.3. In each case, calculate the mean value of the results for the three test pieces. The individual values for the three test pieces shall be within 10 % of

the mean value. If they are not, repeat the test using three further test pieces and report the median of all six results in the test report.

NOTE The rubber industry uses the term equation for the relationships hereafter termed formula. The term formula is used to describe the table of ingredients in a rubber compound.

8.2 Constant elongation

The tension set E_4 is given, in %, by Formula (2):

$$E_4 = 100(L_5 - L_1)/(L_2 - L_1) \tag{2}$$

where

L_1 is the original, unstrained, reference length;

L_2 is the strained reference length;

L_5 is the reference length after recovery.

8.3 Constant load

The elongation E_1 is given, in %, by Formula (3):

$$E_1 = 100(L_2 - L_1)/L_1 \tag{3}$$

The creep E_2 is given, in %, by Formula (4):

$$E_2 = 100(L_3 - L_2)/(L_2 - L_1) \tag{4}$$

The tension set E_3 is given, in %, by Formula (5):

$$E_3 = 100(L_4 - L_1)/L_1 \tag{5}$$

where

L_1 is the reference length, in mm, before loading;

L_2 is the reference length, in mm, 30 s after loading;

L_3 is the reference length, in mm, 60 min after loading;

L_4 is the reference length, in mm, after 10 min rest following the measurement of length L_3.

9 Test report

The test report shall include the following information:

a) details of sample and test pieces, i.e.:

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

2) details of the compound and the curing conditions, if known,

3) the direction, relative to the grain, in which the test pieces were cut,

4) the type of test piece used and its dimensions,

5) the method of preparation of the test pieces, for example whether moulded, cut or extruded,

6) details of any test piece preparation, for example buffing, and

7) the number of test pieces tested;

b) a full reference to the test method used;

c) test details, i.e.

1) the duration and temperature of conditioning,

2) the stress or elongation applied,

3) the duration and temperature of the test,

4) the method of cooling/strain release used (method A, B or C) (applies to constant-elongation method only), and

5) details of any deviation from the standard procedure;

d) test results, i.e.

1) the mean or median values of the individual test results, and

2) the individual results, if required;

e) the date of the test.