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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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Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Apparatus	2
5 Test pieces	4
6 Procedure	6
7 Expression of results	8
8 Test report	9

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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 2285 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This sixth edition cancels and replaces the fifth edition (ISO 2285:2001), which has been revised principally to update the normative references (ISO 471, ISO 4648 and ISO 4661-1 have been replaced by ISO 23529). In addition, the text has been clarified in places.

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

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.

CAUTION — Certain procedures specified in this International Standard may 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 International Standard 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 recommended for product design or the evaluation of low-creep materials. For these, reference should be made to ISO 8013. No agreement between the results of this test or those of ISO 8013 should be inferred.

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 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 188, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

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

ISO 23529:2004, *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 (see also the definitions of creep functions given in ISO 8013).

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 can be provided, for example a threaded rod, provided that the tolerances on extension speed are met (see 6.1.2). Suitable stops or graduations can 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 can 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 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 in accordance with ISO 23529:2004, method B.

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

For ring test pieces, the reference length can 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).

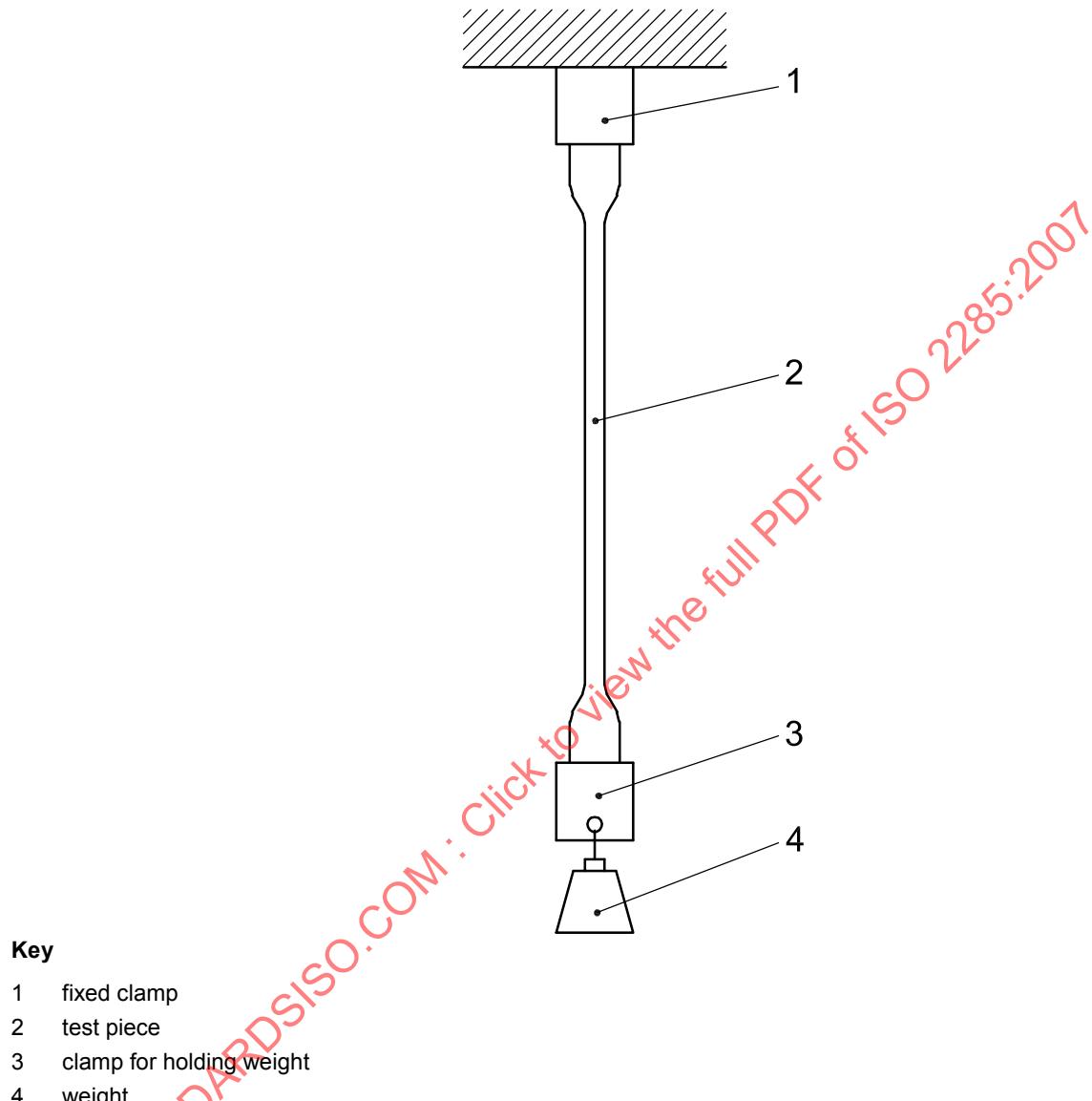


Figure 1 — Straining device

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 23529:2004, 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 test length in accordance with ISO 23529:2004, method B.

5 Test pieces

5.1 Preparation

Test pieces shall be prepared in accordance with ISO 23529. They shall be cut from flat sheet $2\text{ mm} \pm 0,2\text{ mm}$ thick (except large ring test pieces, which shall be cut from sheet $4\text{ mm} \pm 0,2\text{ mm}$ 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 $0,05\text{ mm}$.

5.2 Test pieces for testing under constant elongation

5.2.1 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.

5.2.2 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.

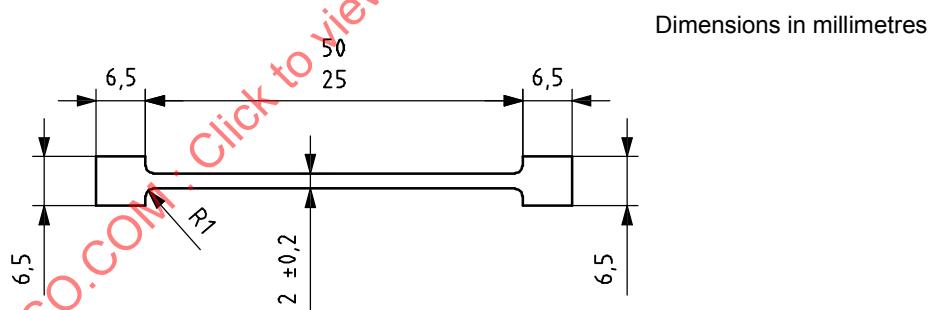


Figure 2 — Test piece with enlarged ends

5.2.3 Ring test pieces

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

Large ring test piece:

thickness: $4\text{ mm} \pm 0,2\text{ mm}$

outer diameter: $52,6\text{ mm} \pm 0,2\text{ mm}$

inner diameter: $44,6\text{ mm} \pm 0,2\text{ mm}$

Small ring test piece:

thickness: $2 \text{ mm} \pm 0,2 \text{ mm}$
 outer diameter: $33,5 \text{ mm} \pm 0,2 \text{ mm}$
 inner diameter: $29,5 \text{ mm} \pm 0,2 \text{ mm}$

5.3 Test pieces for testing under constant load

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

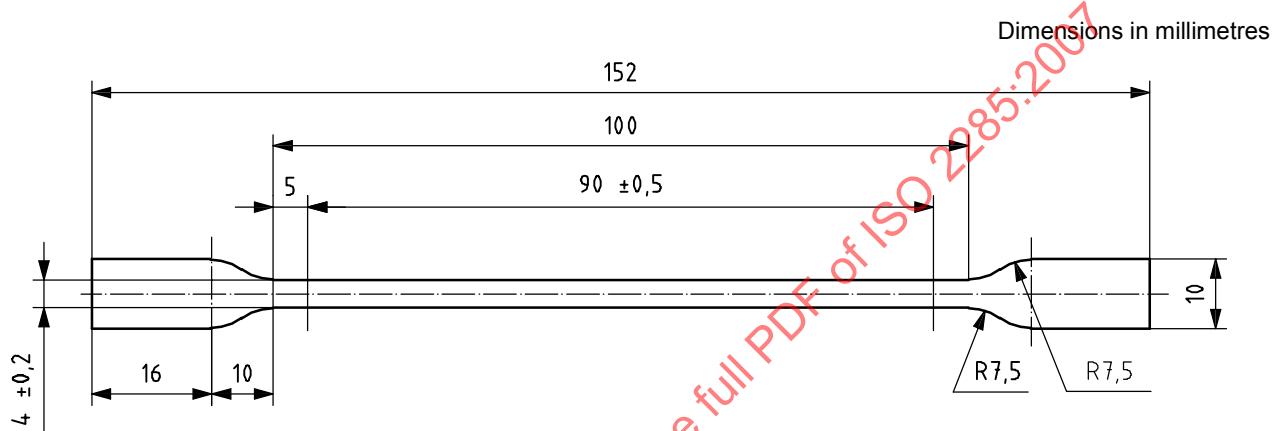


Figure 3 — Test piece for constant loading

5.4 Marking

5.4.1 General

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

5.4.2 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.

5.4.3 Tests at constant load

Two reference marks shall be marked on the central part of the test piece at a distance of $90 \text{ mm} \pm 0,5 \text{ mm}$ from each other.

5.5 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.)

5.6 Time interval between forming the material 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.

5.7 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.

6 Procedure

6.1 Testing under constant elongation

6.1.1 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.

6.1.2 Stretching the test pieces

Extend the test pieces to the required strain at a speed of between 2 mm/s and 10 mm/s. 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 6.1.4.1, 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.

6.1.3 Exposure at the test temperature

6.1.3.1 Tests at standard laboratory temperature

Maintain the strained test pieces at the standard laboratory temperature.

Immediately following the end of the test period, release the strain at a rate of 2 mm/s to 10 mm/s, 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).

6.1.3.2 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 at a speed of 2 mm/s to 10 mm/s, remove the test pieces from the clamps or pulleys and lay them on a flat non-sticky wooden surface. After $(30 \pm 0)^3$ min, measure the reference length to the nearest 0,1 mm.

Method B

At the end of the test period, release the strain at a speed of 2 mm/min to 10 mm/min with the test pieces still in the oven held at the test temperature. After $(30 \pm 0)^3$ 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 $(30 \pm 0)^3$ min, measure the reference length to the nearest 0,1 mm.

Method C

At the end of the test period, remove the straining device from the oven and leave the test pieces in the strained condition. After $(30 \pm 0)^3$ min, release the strain at a speed of 2 mm/min to 10 mm/min, remove the test pieces from the clamps or pulleys and lay them on a flat non-sticky wooden surface. After $(30 \pm 0)^3$ min, measure the reference length to the nearest 0,1 mm.

6.1.4 Test conditions

6.1.4.1 Strain

Calculate the strain λ , in %, using the equation:

$$\lambda = (L_2 - L_1) \times 100 / L_1$$

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 % \pm 1,5 %

20 % \pm 2 %

25 % \pm 2,5 %

50 % \pm 5 %

75 % \pm 7,5 %

100 % \pm 10 %

200 % \pm 10 %

300 % \pm 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 1/3 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 % \pm 2 % is preferred for thermoplastic rubbers.

6.1.4.2 Test period

The test pieces shall normally be strained for a period of $(24\text{--}2)^0$ h, $(72\text{--}2)^0$ h or $(168\text{--}2)^0$ 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.

6.1.4.3 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, $70\text{ }^\circ\text{C} \pm 1\text{ }^\circ\text{C}$ is recommended.

6.2 Testing under constant load

6.2.1 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 $2,5\text{ MPa} \pm 0,1\text{ MPa}$, based on the original cross-sectional area of the test piece. The load shall be applied without shock.

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

6.2.2 Elongation

$30\text{ s} \pm 2\text{ s}$ after loading the test piece, measure the distance between the reference marks (L_2).

6.2.3 Creep

$60\text{ min} \pm 1\text{ min}$ after loading the test piece, measure the length between the reference marks (L_3).

6.2.4 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 $10\text{ min} \pm 1\text{ min}$. Measure the distance between the reference marks again (L_4).

7 Expression of results

7.1 General

Calculate the individual test results as shown in 7.2 or 7.3. 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.