
**Rubber, vulcanized or thermoplastic —
Determination of tension set at normal and
high temperatures**

*Caoutchouc vulcanisé ou thermoplastique — Détermination
de la déformation rémanente après allongement aux températures
normales et élevées*

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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.

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.

International Standard ISO 2285 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

This fourth edition cancels and replaces the third edition (ISO 2285:1988). Two new methods of cooling (A and B) (see 5.4) have been added. Some minor editorial changes have been made.

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Rubber, vulcanized or thermoplastic — Determination of tension set at normal and high temperatures

1 Scope

This international standard specifies a method for determining the tension set characteristics at constant strain of vulcanized or thermoplastic rubbers. The test is intended to measure the ability of rubbers to retain their elastic properties after extension, at a standard 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 temperature.

Note 1 - Three types of test piece are described : strips, strips with enlarged ends and rings. Tests made with different types of test piece do not necessarily give the same value of tension set ; neither will tests made at different temperatures necessarily place the materials in the same order of merit.

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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 471:1995, *Rubber - Temperatures, humidities and times for conditioning and testing.*

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

ISO 4661-1:1993, *Rubber, vulcanized or thermoplastic - Preparation of samples and test pieces - Part 1 : Physical tests.*

3 Apparatus

3.1 Straining device, consisting of a metal rod or other suitable guide fitted with pairs of holders, one fixed and one movable, 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 ends in a firm position for strip test pieces with 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 screwed rod, provided that the tolerances on extension speed are met (see 5.1). Suitable stops or graduations may also be provided to avoid over-extension in the initial straining of the test piece.

Straining devices shall be so designed that, when used at high temperatures in an oven, they can be placed with the reference lengths of the test pieces perpendicular to the direction of air flow; they shall also be of minimal mass in order to avoid excessive lag in the attainment of temperature equilibrium after introduction into an oven.

Multiple unit straining devices may be used, provided that the foregoing requirements are met.

3.2 Oven (if the test is to be carried out at a temperature above the standard laboratory temperature), conforming to the requirements of ISO 3383.

Note 2 - Different types of ovens are described in ISO 188 - Rubber, vulcanized or thermoplastic - Accelerated ageing and heat resistance tests. Tests made with different types of ovens may not be comparable.

3.3 Measuring device, suitable for measuring length to the nearest 0,1 mm.

For strip test pieces, a bench marker shall be provided to mark the length used for measurement, hereinafter called the reference length.

For strip test pieces with enlarged ends, the length of the narrow portion shall be used as the reference length.

For ring test pieces, the reference length may alternatively be the inner diameter of the ring, in which case a graduated cone, allowing measurements to be made to the nearest 0,1 mm, may be used. If measurements are 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 Test piece

4.1 Preparation

Test pieces shall be prepared in general accordance with ISO 4661-1.

4.1.1 Strip test piece

Strips between 2 and 10 mm wide shall be cut with a sharp die from a flat sheet 2 mm \pm 0,2 mm thick of the material under test. A width of 6 mm is preferred. The sheets may be prepared by moulding or from finished products by cutting and buffing.

Strip test pieces (for instance dumb-bells) may have wider ends for easier clamping outside the reference length.

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

4.1.2 Strip test piece with enlarged ends

Test pieces of the shape shown in Figure 1 shall be cut with a sharp die from a flat sheet 2 mm \pm 0,2 mm thick of the material under test. The length of the narrow section (reference length) shall be between 25 and 50 mm. The sheets may be prepared by moulding or from finished products by cutting and buffing.

4.1.3 Ring test piece

Ring test pieces shall be cut from a flat sheet by means of a pair of concentric circular dies or rotating cutters. The separation of the two cutting edges of such dies or cutters shall not differ from the average value by more than 0.05 mm. The sheets may be prepared by moulding or from finished products by cutting and buffing.

The following two sizes of ring test piece may be used:

large ring test piece :

thickness : 4 mm \pm 0,2 mm
outer diameter: 52,6 mm \pm 0,2 mm
inner diameter: 44,6 mm \pm 0,2 mm

small ring test piece:

thickness : 2 mm \pm 0,2 mm
outer diameter: 33,5 mm \pm 0,2 mm
inner diameter: 29,5 mm \pm 0,2 mm

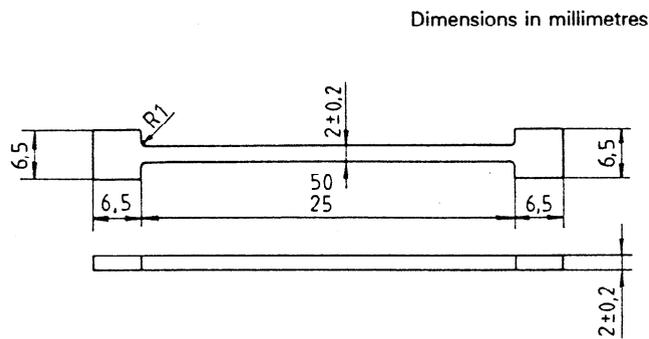


Figure 1 — Test piece with enlarged ends

Note 3 - When cutting strip test pieces, take into consideration the orientation of the material due to moulding.

Note 4 - For thermoplastic rubbers, annealing of sample can be useful before cutting test pieces to release stresses embedded during the manufacturing process.

4.2 Marking

A reference length shall be marked on strip test pieces using a suitable bench marker and ink which does not affect the material and which withstands the temperature of test. The reference length shall be between 25 and 50 mm, measured between the inner sides of the marks. The preferred length of strip test pieces and strip test pieces with enlarged ends is 50 mm. Ring test pieces shall be straightened by means of the rigid channel (see 3.3) 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.

4.3 Number of test pieces

For each test, at least three test pieces shall be used.

4.4 Time-interval between forming the material and testing

4.4.1 The time-interval between forming the material and testing shall be in accordance with ISO 471.

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

4.5 Conditioning before testing

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 471; the same temperature being used throughout any one test or series of test intended to be comparable.

5 Procedure

5.1 Measure the unstrained reference length to the nearest 0,1 mm at the standard laboratory temperature. Place the ends of strip test pieces (4.1.1) into the clamps, or tab ends of test pieces with enlarged ends (4.1.2) into the jaws, or fit ring test pieces (4.1.3) over the pulley rims of the straining device (3.1). Extend the test pieces to the required strain at a speed between 2 and 10 mm/s.

With ring test pieces, rotate the pulleys slightly by hand to equalize the strain in the two halves of the ring, ensuring that the reference length remains centralized between the pulleys

5.2 Between 10 and 20 min after the specified strain has been reached, measure the strained reference length to the nearest 0,1 mm. When the inner diameter of ring test pieces is used as the reference length, it may 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, does not conform to the appropriate standard value, taking account of tolerances, discard the test piece and prepare and test replacement test piece with a modified applied strain.

5.3 For tests at the standard laboratory temperature, maintain the strained test piece at the standard laboratory temperature.

At the end of the test period (6.2) release immediately the strain at a speed of 2 to 10 mm/s, remove the test pieces from the clamps or pulleys and lay free on a wooden surface. After 30 min +3/0 min, measure the reference length to the nearest 0,1 mm.

5.4 For tests at other than standard laboratory temperature, place the strained test pieces in the oven (3.2), operating at the test temperature, between 20 and 30 min after the specified strain has been reached.

There are three methods of cooling/strain releasing, of which method A is the preferred alternative.

Method A

At the end of the test period (6.2) remove the straining device from the oven, release immediately the strain at a speed of 2 to 10 mm/s, remove the test pieces from the clamps or pulleys and lay flat on a wooden surface. After 30 min +3/0 min, measure the reference length to the nearest 0,1 mm.

Method B

At the end of the test period (6.2) release the strain at a speed of 2 to 10 mm/s with the test pieces still in the oven held at the test temperature. After 30 min +3/0 min, remove the straining device from the oven, remove the test pieces from the clamps or pulleys and lay flat on a wooden surface. After 30 min +3/0 min, measure the reference length to the nearest 0,1 mm.

Method C

At the end of the test period (6.2) remove the straining device from the oven and let the test pieces stay in the strained condition. After 30 min +3/0 min, the strain is released at a speed of 2 to 10 mm/s, the test pieces are removed from the clamps or pulleys and laid flat on wooden surface. After 30 min +3/0 min, measure the reference length to the nearest 0,1 mm.

6 Test conditions (strain, duration, temperature)

6.1 Strain

The percentage strain is calculated by the formula :

$$\frac{l_s - l_0}{l_0} \times 100$$

where

l_0 is the original unstrained reference length; and
 l_s 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 value should be selected in accordance with the type of rubber (vulcanized or thermoplastic rubber) and with the final application. For vulcanized rubbers, it is recommended that elongations higher than 1/3 of the elongation at break at the test temperature should not be used ; the value 100 % ±10 % is preferred unless the above considerations dictate otherwise. For thermoplastic rubbers, if a yield point exists, results are only valid calculated at strain values below the yield point. When possible, the value 20 % ±2 % is preferred for thermoplastic rubbers.

6.2 Test period

The test pieces shall be exposed to strain for a period of 24 h 0/-2 h or, alternatively, for 72 h 0/-2 h or 168 h \pm 2 h, the period commencing 30 min after the original straining has been effected. If a longer test period is required, it shall be selected from ISO 471.

6.3 Temperature of test

The test pieces shall be exposed in a strained condition to a temperature selected from ISO 471.

If no temperature is specified in the relevant product specification, 70 °C \pm 1 °C shall be preferred.

7 Expression of results

Calculate the tension set, as a percentage of initial strain, using the formula :

$$\frac{l_1 - l_0}{l_s - l_0} \times 100$$

where

- l_0 is the original unstrained reference length;
- l_s is the strained reference length;
- l_1 is the reference length after recovery.

The mean value of the results for the three test pieces shall be calculated. The individual values of the three test pieces shall agree within 10 % of the mean value. If they do not, the test shall be repeated using three further test pieces and the median of the six results shall be calculated and quoted in the test report.

8 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) sample details:
 - 1) a full description of the sample and its origin;
 - 2) compound details and curing conditions, if known;
 - 3) the direction in which the test pieces were cut, relative to the grain;
 - 4) the type and dimensions of test pieces;
 - 5) the preparation of the test pieces, for example whether moulded or cut;
 - 6) the details of any sample preparation, e.g. buffing;

c) test details:

- 1) the duration and temperature of conditioning;
- 2) the strain applied ;
- 3) the duration and temperature of the test;
- 4) method of cooling/strain releasing used (method A, B or C)
- 5) any deviation from the standard procedure;

d) test results:

- 1) number of test pieces used;
- 2) the mean or median value of the tension set, in per cent;
- 3) the range of individual results, if required;

e) the date of the test.

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