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Vulcanized rubbers — Determination of tension set under constant elongation at normal and high temperatures

Caoutchoucs vulcanisés — Détermination de la déformation rémanente après allongement constant, aux températures normale et élevées

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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.

International Standard ISO 2285 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*, and circulated to the Member Bodies in August 1972.

It has been approved by the Member Bodies of the following countries :

Australia	India	Romania
Austria	Israel	South Africa, Rep. of
Belgium	Italy	Sweden
Canada	Mexico	Switzerland
Czechoslovakia	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Poland	U.S.A.

The Member Body of the following country expressed disapproval of the document on technical grounds :

Germany

Vulcanized rubbers — Determination of tension set under constant elongation at normal and high temperatures

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the tension set characteristics of vulcanized rubbers. The test is intended to measure the ability of rubbers of hardness within the range 30 to 94 International Rubber Hardness Degrees (I.R.H.D.) to retain their elastic properties after extension, at the 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 laboratory temperature.

NOTE — 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 vulcanizates in the same order of merit.

2 REFERENCES

ISO/R 471, *Standard atmospheres for the conditioning and testing of rubber test pieces*.

ISO 1826, *Rubbers — Time-lapse between vulcanization and testing*.

ISO 3383, *Rubber — General directions for achieving elevated or sub-normal temperatures for tests*.¹⁾

ISO . . . , *Rubber — Preparation of test pieces*.²⁾

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 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.

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 . . .

4.1.1 Strip test piece

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

1) At present at the stage of draft.

2) In preparation.

Strip test pieces may have wider ends for easier clamping outside the reference length, for example dumb-bells.

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 the figure shall be cut with a sharp die from a flat sheet $2,0 \pm 0,2$ 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 articles by cutting and buffing.

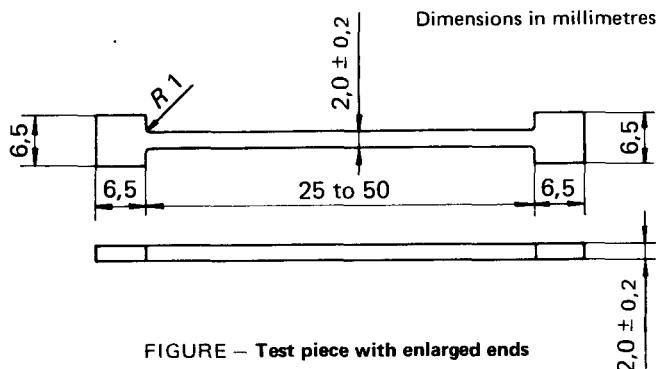


FIGURE — Test piece with enlarged ends

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 articles by cutting and buffing.

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

large ring test piece :

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

small ring test piece :

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

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. 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, three test pieces shall be used.

4.4 Time-lapse between vulcanization and testing

4.4.1 The time-lapse between vulcanization and testing shall be in accordance with ISO 1826.

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

4.5 Conditioning

Prepared test pieces shall be conditioned immediately before testing for a minimum period of 3 h at one of the defined standard laboratory temperatures in accordance with ISO/R 471, the same temperature being used throughout any one test or series of tests 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 into the clamps, or tab ends of test pieces with enlarged ends into the jaws, or fit ring test pieces over the pulley rims of the straining device. 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 a replacement test piece with a modified applied strain.

5.3 For tests at other than standard laboratory temperatures, place the strained test pieces in the oven operating at the test temperature, between 20 and 30 min after the specified strain has been reached; at the end of the test period, remove the test pieces from the oven and allow to cool in the strained condition for 30 to 35 min.

5.4 After the appropriate duration of straining (see 6.2), release 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 ± 3 min measure the reference length to the nearest 0,1 mm, using the rigid channel to straighten ring test pieces if required.

6 STRAIN, DURATION AND TEMPERATURE OF TEST

6.1 Strain

The percentage strain, calculated by the formula

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

where

l_0 is the original unstrained reference length and

l_s is the strained reference length,

shall be one of the following values :

- (25 ± 2,5) %
- (50 ± 5) %
- (100 ± 10) %
- (200 ± 10) %
- (300 ± 10) %

The strain value should be selected in accordance with the final application. For normal use it is recommended that elongations higher than one-third of the breaking elongation at the test temperature should not be used. The value (100 ± 10) % is preferred unless the above considerations dictate otherwise.

6.2 Duration of test

The strained test pieces shall be exposed for $24 \pm \frac{0}{2}$ h or, alternatively, if a longer test period is required, for $72 \pm \frac{0}{2}$ h or $168 \pm \frac{0}{2}$ h, the period commencing 30 min after the original straining has been effected.

6.3 Temperature of test

The test pieces shall be exposed in a strained condition at one of the following temperatures :

standard laboratory temperature

- 70 ± 1 °C
- 85 ± 1 °C
- 100 ± 1 °C
- 125 ± 2 °C
- 150 ± 2 °C

If a temperature other than standard laboratory temperature is used, 70 ± 1 °C shall be preferred.

For special cases, higher standard temperatures may be used.

7 EXPRESSION OF RESULTS

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

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

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 particulars :

- a) a reference to this International Standard;
- b) a full description of the sample and any relevant facts about its pre-test history;
- c) the preparation of the test pieces, for example whether moulded or cut;
- d) the curing conditions applied to the test pieces, if known;
- e) the duration and temperature of conditioning of the test pieces prior to testing;
- f) the type and dimensions of test pieces;
- g) the number of test pieces tested;
- h) the strain applied;
- i) the duration and temperature of the test;
- j) any non-standardized procedures adopted;
- k) the mean or median value of the tension set, in per cent, for the test pieces tested;
- l) the date of the test.

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