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2921

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Vulcanized rubber — Determination of low temperature characteristics — Temperature-retraction procedure (TR test)

Caoutchoucs vulcanisés — Détermination des caractéristiques à basse température — Méthode température-retrait (essai TR)

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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 2921 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*, and circulated to the Member Bodies in December 1972.

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

Australia	India	South Africa, Rep. of
Belgium	Italy	Sweden
Brazil	Netherlands	Switzerland
Canada	New Zealand	Thailand
Czechoslovakia	Poland	United Kingdom
France	Portugal	U.S.A.
Hungary	Romania	U.S.S.R.

No Member Body expressed disapproval of the document.

Vulcanized rubber — Determination of low temperature characteristics — Temperature-retraction procedure (TR test)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determination of the temperature-retraction characteristics of extended vulcanized rubber. The temperature at which a specified retraction occurs is noted as the temperature is increased at a uniform rate after the rubber has been extended at room temperature and cooled to a sufficiently low temperature such that retraction does not occur upon release of the extending force.

NOTE — In addition to the two sizes of the standard test piece, other types of test piece cut from products are described. These do not necessarily give the same values of retraction temperature, and comparison between values obtained from different types of test piece is to be avoided.

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 Container for coolant, insulated and equipped with an agitator, a temperature meter and with a device for heating the coolant in accordance with ISO 3383.

3.2 Coolant, which does not affect the rubber material to be tested, as prescribed in ISO 3383. Among the liquids that have been found suitable for use at low temperatures are acetone, methanol, ethanol, butanol/silicone fluid and *n*-hexane with crushed dry ice (solid carbon dioxide) added. Care shall be taken to avoid polar liquids in combination with polar rubbers or hydrocarbons in combination with hydrocarbon rubbers.

Gaseous media may be employed as the coolant when the design of the apparatus is such that tests using it will duplicate those obtained with liquid media.

3.3 Rack with test piece holders, equipped with loading device, holders for one or more test pieces and locking device for the upper (movable) test piece holders (see figure 2).

The rack shall be designed to maintain a slight tension (10 to 20 kPa) on the test piece and to permit it to be stretched up to a maximum of 350 %; the design shall permit the upper test piece holder to be locked into position at the chosen elongation and subsequently released. Means shall be provided to enable the length of the test piece to be read, at any time during the test, with an accuracy of ± 1 mm.

Alternatively, a series of removable scales graduated to allow the retraction to be read directly as a percentage of the elongation of the frozen rubber with an accuracy of ± 1 % may be used.

The movable parts of the apparatus shall be constructed so that lowest possible friction occurs.

4 TEST PIECE

4.1 Preparation

Test pieces shall be prepared in general accordance with ISO . . .

4.2 Types

4.2.1 Standard test piece

The standard test piece shall be a strip with enlarged ends for clamping with dimensions in accordance with figure 1. The reference length shall be either 100 or 50 mm. The test piece with reference length 100 mm is preferred for tests with small elongations, the test piece with reference length 50 mm for tests with larger elongations. Test pieces shall be

1) At present at the stage of draft.

2) In preparation.

cut with a sharp die from a flat sheet $2,0 \pm 0,2$ mm thick. The sheets may be prepared by moulding or from finished articles by cutting and buffing.

4.2.2 Test piece cut from products

Alternatively, other types of test piece cut from finished rubber products may be used (for example an O-ring with a cross-sectional diameter between 1,5 and 4 mm).

4.3 Number

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

4.4 Conditioning

Unless otherwise specified for technical reasons, the following procedures shall be used :

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

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

4.4.3 Test pieces shall be conditioned, immediately before testing, at one of the standard laboratory temperatures specified in ISO/R 471.

NOTE — If samples that are apt to crystallize are exposed to low storage temperatures before testing, crystallization may occur that largely affects the TR values measured. If values for the material in uncrystallized condition are desired, the test pieces shall be decrystallized before testing by heating them in an oven at 70°C for 30 min. They shall then be conditioned at standard laboratory temperature for at least 30 min and no more than 60 min.

5 PROCEDURE

The bath shall contain enough coolant (3.2) to cover the test piece during testing by at least 25 mm of liquid. Cool, whilst stirring, the coolant to below -70°C as described in ISO 3383.

While the liquid is cooling, insert the test piece in the rack (3.3) and, at the standard laboratory temperature, stretch the reference length to the chosen elongation and lock it into position. Ensure that the test piece is only kept stretched at standard laboratory temperature for the minimum time.

The elongation shall be chosen in the light of the following criteria :

- a) if technical reasons do not dictate otherwise and to reduce the effect of crystallization, the elongation of 50 % shall be used;

b) one of the following elongations shall be used to study the combined effect of crystallization and low temperature :

- 1) 250 %;
- 2) half the ultimate elongation if 250 % is unobtainable;
- 3) 350 % if the ultimate elongation is greater than 600 %.

When the coolant has reached an equilibrium temperature below -70°C , place the rack with the test pieces in the bath. Allow to stand for 10 ± 2 min in the bath between -70°C and -73°C . Release the locking device of the upper holder and allow the specimens to retract freely. At the same time, raise the temperature of the liquid at the rate of $1^{\circ}\text{C}/\text{min}$, the tolerances being such that the temperature rise during any 10 min interval is within $10 \pm 2^{\circ}\text{C}$.

Should the elongated test piece retract to the original length at -70°C , use, if necessary, another cooling medium and cool to a lower temperature.

Take the first reading at -70°C and continue to read the actual temperature and the retracted length or the retraction every 2 min until retraction has reached 75 %.

NOTES

- 1 For the study of crystallization effects or the effect of long time exposure, longer times of exposure under strain at one or more selected low temperatures may be chosen according to the purpose of the test and the material under investigation.
- 2 Various elongations do not necessarily give the same results.

6 EXPRESSION OF RESULTS

The percentage retraction r is read from the graduated scales or calculated according to the formula :

$$r = \frac{l_s - l_r}{l_s - l_o} \times 100$$

where

- l_s is the stretched length in the locked position;
- l_r is the retracted length at the observed temperature;
- l_o is the reference length.

Plot r against the actual temperature on a diagram.

From the diagram, read the temperatures which show retractions of 10 %, 30 %, 50 % and 70 %. These temperatures are designated as TR 10, TR 30, TR 50 and TR 70.

Calculate the mean value of three determinations of the temperature for TR 10, TR 30, TR 50 and TR 70.

7 TEST REPORT

The test report shall include the following particulars :

- a) full description of the sample and any relevant facts about its pre-test history;
- b) preparation of test pieces, for example whether moulded or cut;
- c) curing conditions applied to test pieces, if known;
- d) time and temperature of conditioning of test pieces prior to testing;
- e) full reference to the test method, for example this International Standard or corresponding national standard;
- f) type and dimensions of test pieces;
- g) number of test pieces tested;
- h) elongation at freezing;
- i) coolant used;
- j) time and temperature of low temperature conditioning;
- k) any non-standardized procedures adopted;
- l) calculated mean values of TR 10, TR 30, TR 50 and TR 70, together with the unit in which the results are reported;
- m) date of test.

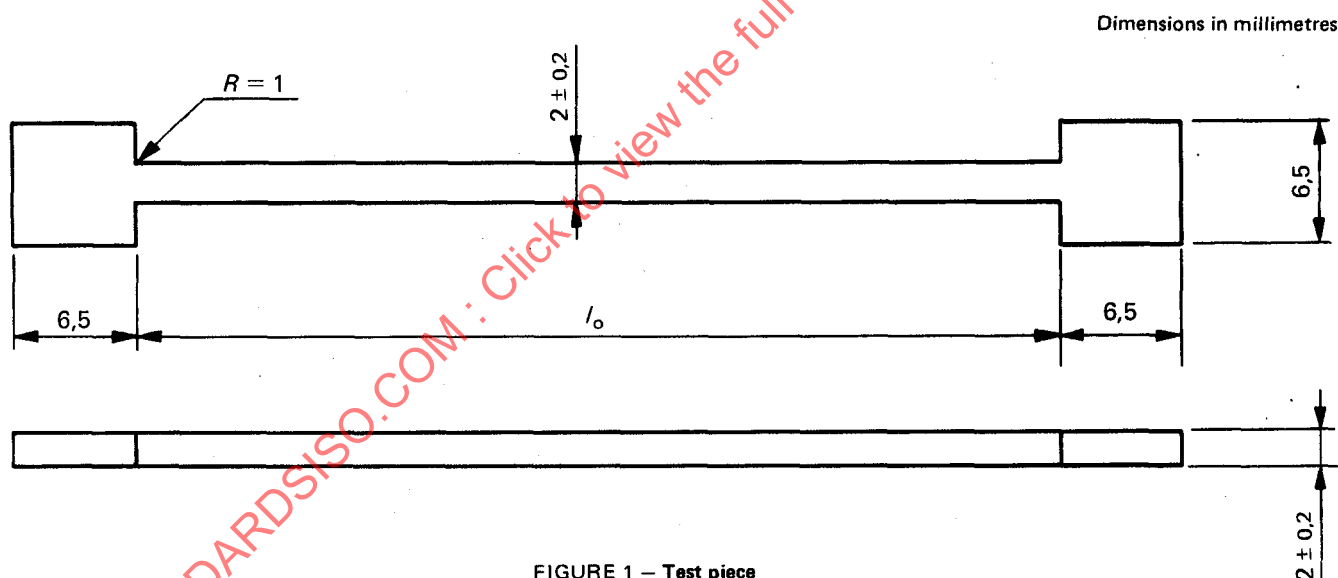


FIGURE 1 — Test piece
(Reference length l_0 shall be $100 \pm 0,2$ mm for small elongations and $50 \pm 0,2$ mm for large elongations)