
International Standard



2921

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Rubber, vulcanized — Determination of low temperature characteristics — Temperature-retraction procedure (TR test)

Caoutchouc vulcanisé — Détermination des caractéristiques à basse température — Méthode température-retrait (essai TR)

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Descriptors : rubber, vulcanized rubber, tests, low temperature tests, measuring, contraction temperature.

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 developed by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 2921-1975), which had 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	USA
Hungary	Romania	USSR

No member body had expressed disapproval of the document.

Rubber, vulcanized — Determination of low temperature characteristics — Temperature-retraction procedure (TR test)

1 Scope and field of application

This International Standard specifies a method for the determination of the temperature-retraction characteristics of extended vulcanized rubber.

2 References

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1826, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

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

ISO 4661, *Rubber — Preparation of test pieces.*

3 Principle

Noting the temperature at which a specified retraction occurs as the temperature is increased at a uniform rate after the rubber test piece 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 standard test piece, other types of test pieces cut from products are described. These do not necessarily give the same values of retraction temperature, and comparison between the values obtained using different types of test pieces should be avoided.

4 Apparatus

4.1 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.

4.2 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.

4.3 Rack with test piece holders, equipped with a loading device, holders for one or more test pieces and a 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 the lowest possible friction occurs.

5 Test piece

5.1 Preparation

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

5.2 Types

5.2.1 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 and the test piece with reference length 50 mm for tests with larger elongations. Test pieces shall be 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.

5.2.2 Test pieces cut from products

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

5.3 Number

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

5.4 Conditioning

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

5.4.1 The time-interval between vulcanization and testing shall be in accordance with ISO 1826.

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

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

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 the 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 but not more than 60 min.

6 Procedure

The bath shall contain enough coolant (4.2) to cover the test piece during testing with at least 25 mm of liquid. Cool the coolant, whilst stirring, to below -70 °C as described in ISO 3383.

While the liquid is cooling, insert the test piece in the rack (4.3) 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, an 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 $10 + \frac{2}{0}$ 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 °C/min, the tolerances being such that the temperature rise during any 10 min interval is within 10 ± 2 °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 term 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.

7 Expression of results

The percentage retraction, r , may be 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 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.

8 Test report

The test report shall include the following information :

a) a full description of the sample and any relevant facts about its pre-test history;

b) the method of preparation of test pieces, for example whether moulded or cut;

- c) the curing conditions applied to the test pieces, if known;
- d) the time and temperature of conditioning of test pieces prior to testing;
- e) the full reference to the test method used, for example a reference to this International Standard;
- f) the type and dimensions of the test pieces;
- g) the number of test pieces tested;
- h) the elongation at freezing;
- j) the coolant used;
- k) the time and temperature of low temperature conditioning;
- m) any non-standardized procedures adopted;
- n) the calculated mean values of TR 10, TR 30, TR 50 and TR 70, together with the unit in which the results are reported;
- p) the date of test.

Dimensions in millimetres

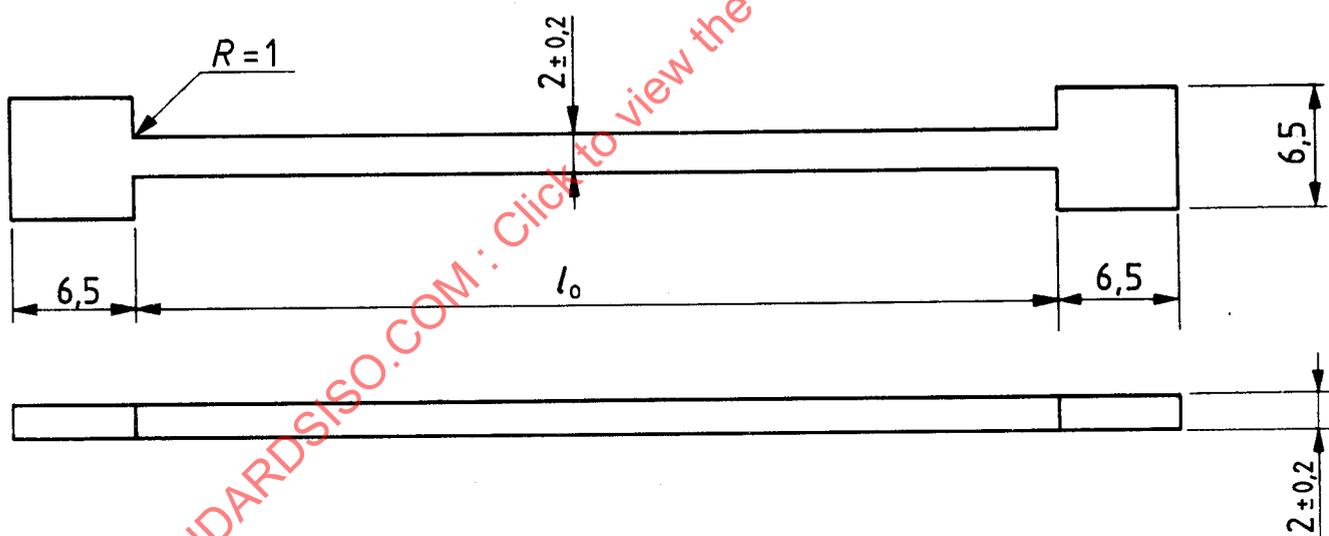


Figure 1 — Test piece

[Reference length l_0 is preferably $100 \pm 0,2$ mm for small elongations and $50 \pm 0,2$ mm for large elongations (see 5.2.1)]

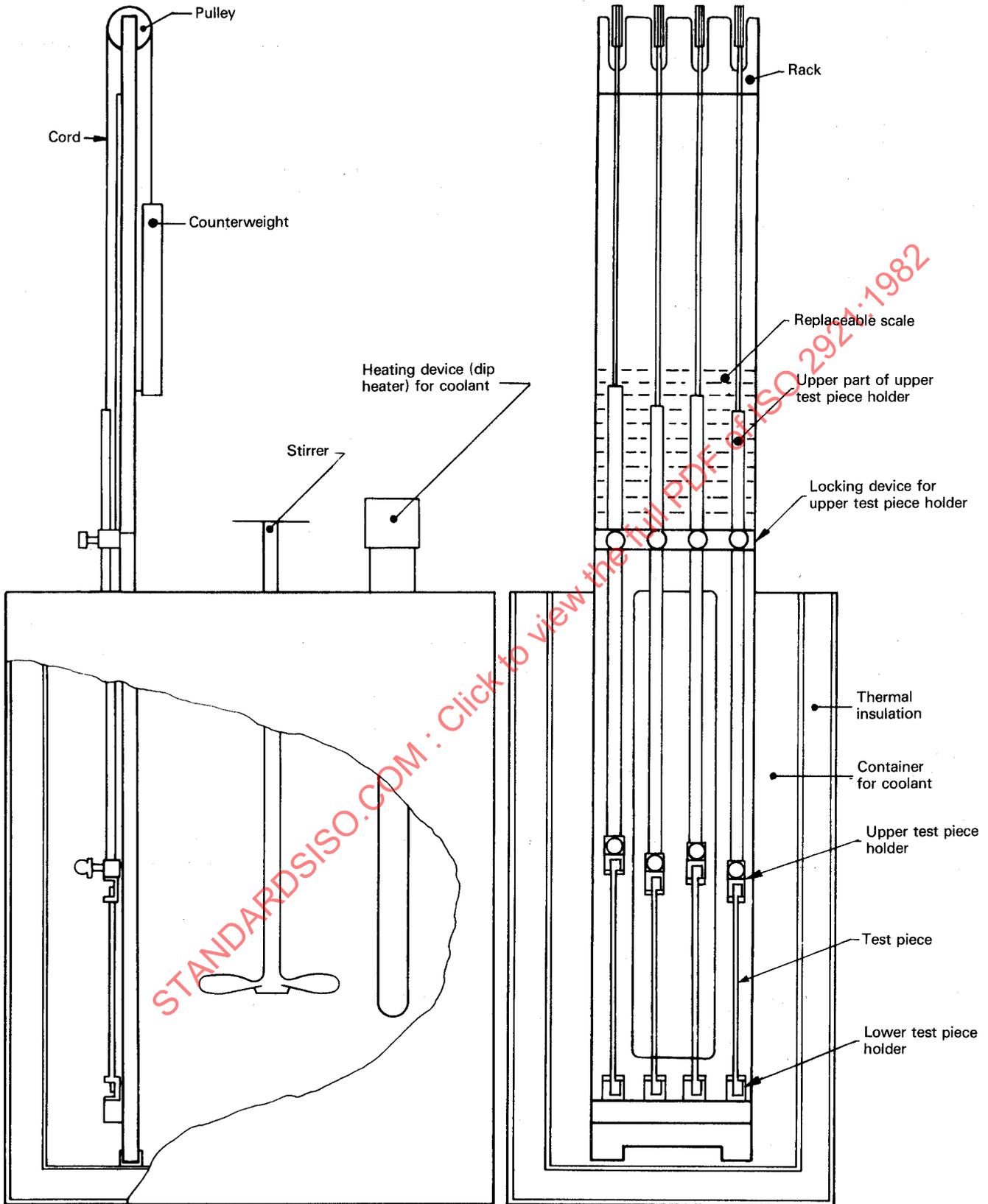


Figure 2 — Retraction apparatus