
**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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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 2921 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

This third edition cancels and replaces the second edition (ISO 2921:1982), which has been technically revised, in particular as regards the description of what constitutes a suitable coolant (see 4.2).

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

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.

1 Scope

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

NOTE — This International Standard has not been extended to include thermoplastic rubber as many thermoplastic elastomers have a yield point in the range of 5 % to 20 % elongation. This fact may affect the result when making TR tests and the test results should be analysed with caution.

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 Principle

A test piece is stretched at room temperature and then cooled to a sufficiently low temperature such that retraction does not occur upon removal of the stretching force. The stretching force is removed and the temperature increased at a uniform rate. The temperatures at which specified percentage retractions occur are calculated.

NOTE — In addition to the two sizes of 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 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 thermometer and a device for heating the coolant in accordance with ISO 3383.

4.2 Coolant, which does not affect the rubber material being tested, as prescribed in ISO 3383.

Gases may be employed as the coolant provided the design of the apparatus is such that tests using them will duplicate those obtained with liquids.

NOTE — The following fluids may be used:

- a) for temperatures down to $-60\text{ }^{\circ}\text{C}$, silicone oils of kinematic viscosity $5\text{ mm}^2/\text{s}$ at ambient temperature, owing to their chemical inertness towards rubbers, their non-flammability and their non-toxicity;
- b) for temperatures down to $-70\text{ }^{\circ}\text{C}$, methanol or ethanol;
- c) for temperatures down to $-120\text{ }^{\circ}\text{C}$, methylcyclohexane cooled by liquid nitrogen (found to be satisfactory with the use of suitable apparatus).

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 kPa 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 $\pm 1\text{ mm}$.

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 $\pm 1\%$, may be used.

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

5 Test pieces

5.1 Preparation

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

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 mm 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 $2,0\text{ mm} \pm 0,2\text{ mm}$ 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 piece cut from finished rubber products may be used (for example an O-ring with a cross-sectional diameter between 1,5 mm 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 471.

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 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 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 pieces in the rack (4.3) and, at the standard 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 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 between –70 °C and –73 °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 °C/min, the tolerance being such that the temperature rise during any 10 min interval is within $10 \text{ °C} \pm 2 \text{ °C}$.

Should the elongated test piece retract to the original length at –70 °C, use, if necessary, another coolant 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 percentage retraction every 2 min until retraction has reached 75 %.

NOTE 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 depending on the purpose of the test and the material under investigation.

NOTE 2 Different 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 from the equation:

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

where

l_s is the stretched length in the locked position;

l_r is the retracted length at the temperature concerned;

l_o is the reference length.

Plot r against the actual temperature on a graph.

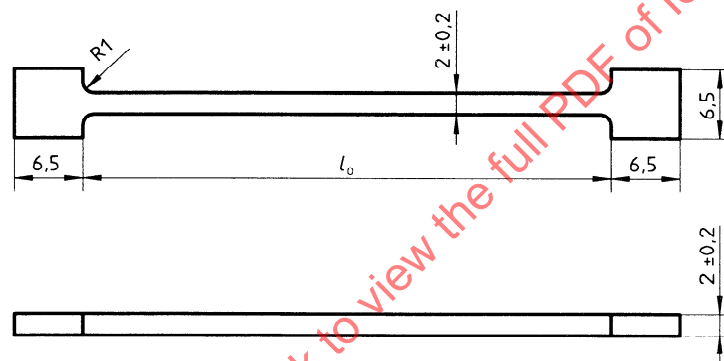
From the graph, read the temperatures which correspond to retractions of 10 %, 30 %, 50 % and 70 %. These temperatures are designated TR 10, TR 30, TR 50 and TR 70.

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 the test pieces, for example whether moulded or cut;
- c) the curing conditions used for the test pieces, if known;
- d) the time and temperature of conditioning of the 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 of test piece and its dimensions;
- g) the number of test pieces tested;
- h) the elongation at freezing;
- i) the coolant used;
- j) the time and temperature of low-temperature conditioning;
- k) any non-standardized procedures adopted;
- l) the calculated mean values of TR 10, TR 30, TR 50 and TR 70, together with the units in which the results are reported;
- m) the date of the test.



Dimensions in millimetres

Figure 1 – Test piece

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

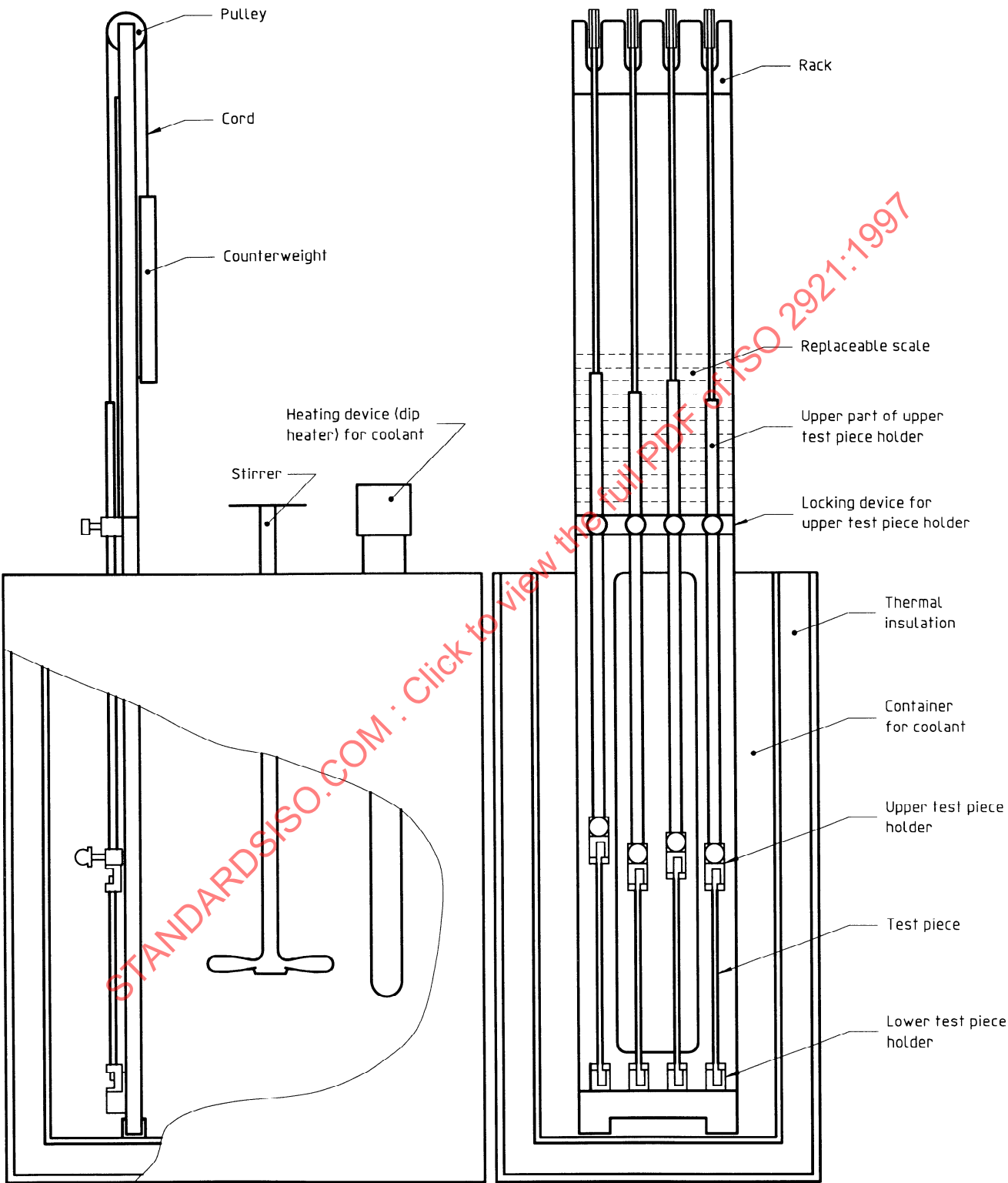


Figure 2 – Retraction apparatus

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