INTERNATIONAL STANDARD

**ISO 2921**

Fifth edition
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# Rubber, vulcanized — Determination of low-temperature retraction (TR test)

*Caoutchouc vulcanisé — Détermination du retrait à basse température (essai TR)*



Reference number
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## Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This fifth edition cancels and replaces the fourth edition (ISO 2921:2005), which has been technically revised, mainly to include an annex specifying a calibration schedule for the apparatus used.

# Rubber, vulcanized — Determination of low-temperature retraction (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.**

**IMPORTANT — Certain procedures specified in this International Standard might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.**



## 1 Scope

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

This International Standard does not cover thermoplastic rubbers, as many thermoplastic elastomers have a yield point in the range of 5 % to 20 % elongation. This fact might affect the result when carrying out TR tests on thermoplastic rubbers, and the results obtained from such tests should be analysed with caution.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18899:2004, *Rubber — Guide to the calibration of test equipment*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

## 3 Principle

A test piece is stretched at standard laboratory 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 determined.

## 4 Apparatus

**4.1 Retraction apparatus**, comprising the components specified in 4.2 to 4.8 (see also Figure 1).

**4.2 Heat-transfer medium**, liquid or gaseous, which remains fluid at the test temperature and which does not appreciably affect the material being tested, as prescribed in ISO 23529.

Gases may be employed as the heat-transfer medium provided the design of the apparatus is such that results obtained using them will duplicate those obtained with liquids.

The following fluids have been used satisfactorily:

a) for temperatures down to −60 °C, silicone fluids of kinematic viscosity of about 5 $mm^2/s$ at ambient temperature, which are usually suitable owing to their chemical inertness towards rubbers, their non-flammability and their non-toxicity;

b) for temperatures down to −73 °C, ethanol;

c) for temperatures down to −120 °C, methylcyclohexane cooled by liquid nitrogen (found to be satisfactory with the use of suitable apparatus).

**4.3 Temperature-measuring device**, capable of measuring the temperature to within 0,5 °C over the whole range of temperatures over which the apparatus is to be used.

The temperature sensor shall be positioned near the test pieces.

**4.4 Temperature control**, capable of maintaining the temperature of the heat-transfer medium to within ±1 °C.

**4.5 Container for the heat-transfer medium**: A bath for a liquid medium or a test chamber for a gaseous medium, with means of heating the heat-transfer medium.

**4.6 Means of agitating the heat-transfer medium**: A stirrer for liquids or a fan or blower for gases, which ensures thorough circulation of the heat-transfer medium. It is important that the stirrer also moves the liquid vertically to ensure a uniform temperature in the liquid.

**4.7 Stopwatch or other timing device**, calibrated in seconds.

**4.8 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 1).

The rack shall be designed to maintain a slight tension (10 kPa to 20 kPa in air) on each test piece and to permit them 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 each test piece to be read, at any time during the test, with an accuracy of ±0,25 mm or better.

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 ±0,5 % may be used.

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

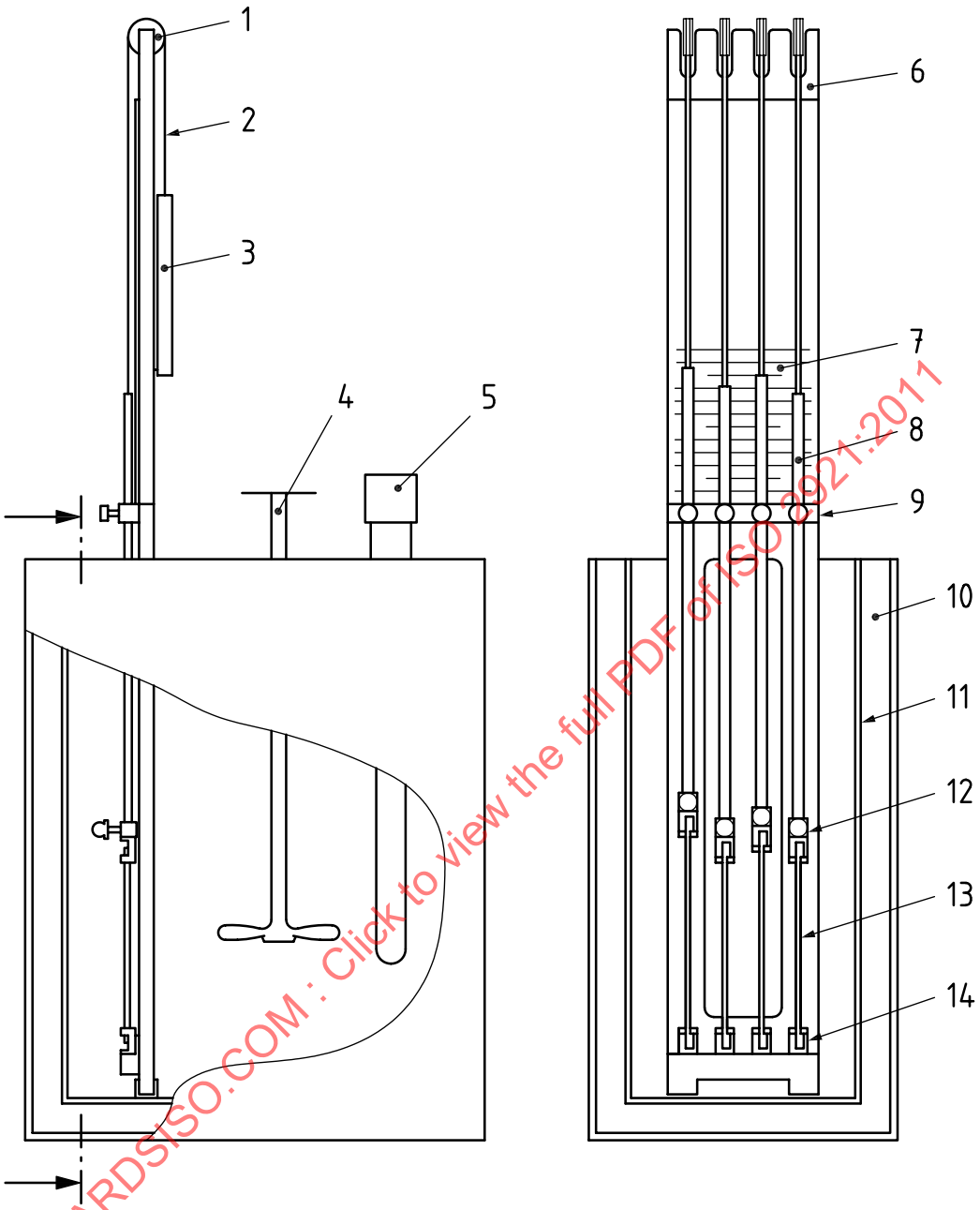
## 5 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in Annex A.

## 6 Test pieces

### 6.1 Preparation

Test pieces shall be prepared in general accordance with ISO 23529.






**Key**

1 pulley
2 cord
3 counterweight
4 stirrer
5 heating device (immersion heater) for heat-transfer medium
6 rack
7 removable graduated scales
8 upper part of upper test piece holder
9 locking device for upper test piece holder
10 thermal insulation
11 bath for heat-transfer medium
12 upper test piece holder
13 test piece
14 lower test piece holder

**Figure 1 — Retraction apparatus**

## 6.2 Types

### 6.2.1 Standard test piece

The standard test piece shall be a strip with enlarged ends for clamping, with dimensions in accordance with Figure 2. The reference length shall be either 100 mm ± 0,2 mm or 50 mm ± 0,2 mm. The test piece with a reference length of 100 mm ± 0,2 mm is preferred for tests with small elongations and the test piece with a reference length of 50 mm ± 0,2 mm for tests with larger elongations. Test pieces shall be cut with a sharp die from a flat sheet 2 mm ± 0,2 mm thick. The sheets may be prepared by moulding or from finished products by cutting and buffing.

The 50 mm test piece may be used also with 50 % elongation if the reading accuracy of the measurement system is ±0,125 mm or better.

### 6.2.2 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).

Note that such test pieces do not necessarily give the same values of retraction temperature as do the two sizes of standard test piece specified in 6.2.1, and comparison between the values obtained using different types of test piece should be avoided.

## 6.3 Number

At least three test pieces shall be used for each test.

Dimensions in millimetres

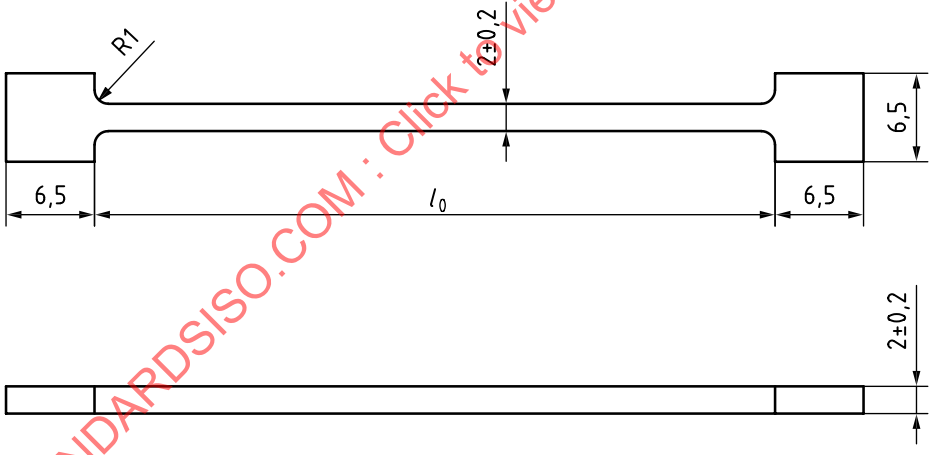



**Figure 2 — Test piece**



## 6.4 Conditioning

**6.4.1** Unless otherwise specified for technical reasons, the procedure in 6.4.2 to 6.4.5 shall be followed.

**6.4.2** The time-interval between vulcanization and testing shall be in accordance with ISO 23529.

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

**6.4.4** Test pieces shall be conditioned, immediately before testing, at one of the standard laboratory temperatures specified in ISO 23529.

**6.4.5** If samples that are apt to crystallize are exposed to low storage temperatures before testing, crystallization can occur that significantly 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 a standard laboratory temperature for at least 30 min, but not more than 60 min.

## 7 Procedure

The bath shall contain enough heat-transfer medium (4.2) to cover the test pieces with at least 25 mm of liquid during testing.

Cool the heat-transfer medium, whilst stirring, to below –70 °C as described in ISO 23529.

While the liquid is cooling, insert the test pieces in the rack (4.8) and, at the standard laboratory temperature being used, stretch each test piece from the reference length to the chosen elongation and lock it in position. Ensure that the test pieces are only kept stretched at the standard temperature for the minimum time.

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

a) provided 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 elongation at break if 250 % is unobtainable,

   3) 350 %, if the elongation at break is greater than 600 %.

When the heat-transfer medium 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 min ± 2 min in the bath between –70 °C and –73 °C. Release the locking device for the upper holder and allow the test pieces 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 °C ± 2 °C.

If an elongated test piece retracts to its original length at –70 °C, cool to a lower temperature, using, if necessary, another heat-transfer medium.

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

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 used, depending on the purpose of the test and the material under investigation.

NOTE Different elongations do not necessarily give the same results.

## 8 Expression of results

The percentage retraction $r$ may be read from the graduated scales or calculated from the following equation:

$$r = \frac{l_s - l_r}{l_s - l_0} \times 100$$

where

$l_s$ is the stretched length in the locked position;

$l_r$ is the retracted length at the temperature concerned;

$l_0$ is the reference length.

Plot $r$ against the corresponding temperature on a graph.

From the graph, read the temperatures which correspond to retractions of 10 %, 30 %, 50 % and 70 %. These temperatures are designated TR10, TR30, TR50 and TR70.

Calculate the median value of three determinations of the temperature for TR10, TR30, TR50 and TR70.

## 9 Test report

The test report shall include the following information:

a) sample details:
   1) a full description of the sample and its origin,
   2) compound details and cure details, where appropriate,
   3) the method of preparation of the test pieces from the sample, for example moulded or cut;

b) test method:
   1) a reference to the test method used, i.e. the number of this International Standard,
   2) the type of test piece used;

c) test details:
   1) the standard laboratory temperature used,
   2) the time and temperature of conditioning prior to test,
   3) the elongation at freezing,
   4) the heat-transfer medium used,
   5) details of any procedures not specified in this International Standard;

d) test results:
   1) the number of test pieces used,
   2) the median values of TR10, TR30, TR50 and TR70,
   3) the individual values of TR10, TR30, TR50 and TR70 for each test piece;

e) the date of the test.

# Annex A
(normative)

# Calibration schedule

## A.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration is carried out in the “as-received” condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

## A.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table A.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code-letter. The code-letters used in the calibration schedule are:

C requirement to be confirmed, but no measurement;

N initial verification only;

S standard interval as given in ISO 18899;

U in use.