INTERNATIONAL STANDARD

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Rubber, vulcanized — Determination of crystallization effects under compression

Caoutchouc vulcanisé — Détermination des effets de la cristallisation sous compression

ISO 6471:1994(E)

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 6471 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Sub-Committee SC 2, *Physical and degradation tests*.

This second edition cancels and replaces the first edition (ISO 6471:1983). Clause 2 has been updated and a new clause "Definitions" has been added.

INTERNATIONAL STANDARD

ISO 6471:1994(E)

Rubber, vulcanized — Determination of crystallization effects under compression

1 Scope

This International Standard specifies a method for the determination of the tendency of vulcanized rubbers to crystallize, and the time dependence of crystallization, by measurement of the recovery of compressed test pieces.

Crystallization, which occurs more rapidly under high compression, reduces the recovery process.

An alternative method of assessing the tendency to crystallize, based on measurements of increase in hardness, is given in ISO 3387. The latter requires longer test durations for vulcanizates, but the test method is applicable to both raw rubber and compounds.

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:—[1], *Rubber — Times, temperatures and humidities for conditioning and testing.*

ISO 815:1991, *Rubber, vulcanized or thermoplastic — Determination of compression set at ambient, elevated or low temperatures.*

ISO 3387:—[2], *Rubber — Determination of crystallization effects by hardness measurements.*

1) To be published. (Revision of ISO 471:1983)

2) To be published. (Revision of ISO 3387:1978)

ISO 4648:1991, *Rubber, vulcanized or thermoplastic — Determination of dimensions of test pieces and products for test purposes.*

ISO 4661-1:1993, *Rubber, vulcanized or thermoplastic — Preparation of samples and test pieces — Part 1: Physical tests.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 crystallization: A physical process comprising a phase transition of the first order which in rubbers leads to an increase in hardness and stiffness and a decrease in recovery.

3.2 recovery: The ability of a test piece to recover its shape after deformation and conditioning under specified conditions of temperature and deformation over a given period, determined by measuring the height of the test piece a pre-determined time after release at the test temperature.

3.3 half-time to crystallization: The time required for the recovery to attain exactly one half of its final value.

4 Principle

A test piece is compressed under low strain and the recovery, in the absence of crystallization, is determined. The same or another test piece is then compressed under high strain and the recovery, after crystallization, is determined. From these values, an assessment is made of the tendency of the rubber to crystallize.

1

5 Apparatus[3]

5.1 Compression device, consisting of two parallel, flat, highly polished stainless-steel plates, between the faces of which the test pieces are compressed. The finish of the surface of the compression plates shall be not worse than 0,2 μm arithmetic-mean deviation from the mean line of the profile. The compression device shall be connected to suitable equipment for compressing the test pieces to the specified compression within 30 s. It shall be capable of setting and maintaining a compression of 20 % to 60 % throughout the duration of the test and shall be such that it is possible to keep it in a low-temperature bath or enclosure at the specified test temperature. Care shall be taken to ensure that there is no conduction of heat to the test piece, for example through metal parts which are connected to the outside of the bath or enclosure. It shall be capable of releasing the compression at low temperature.

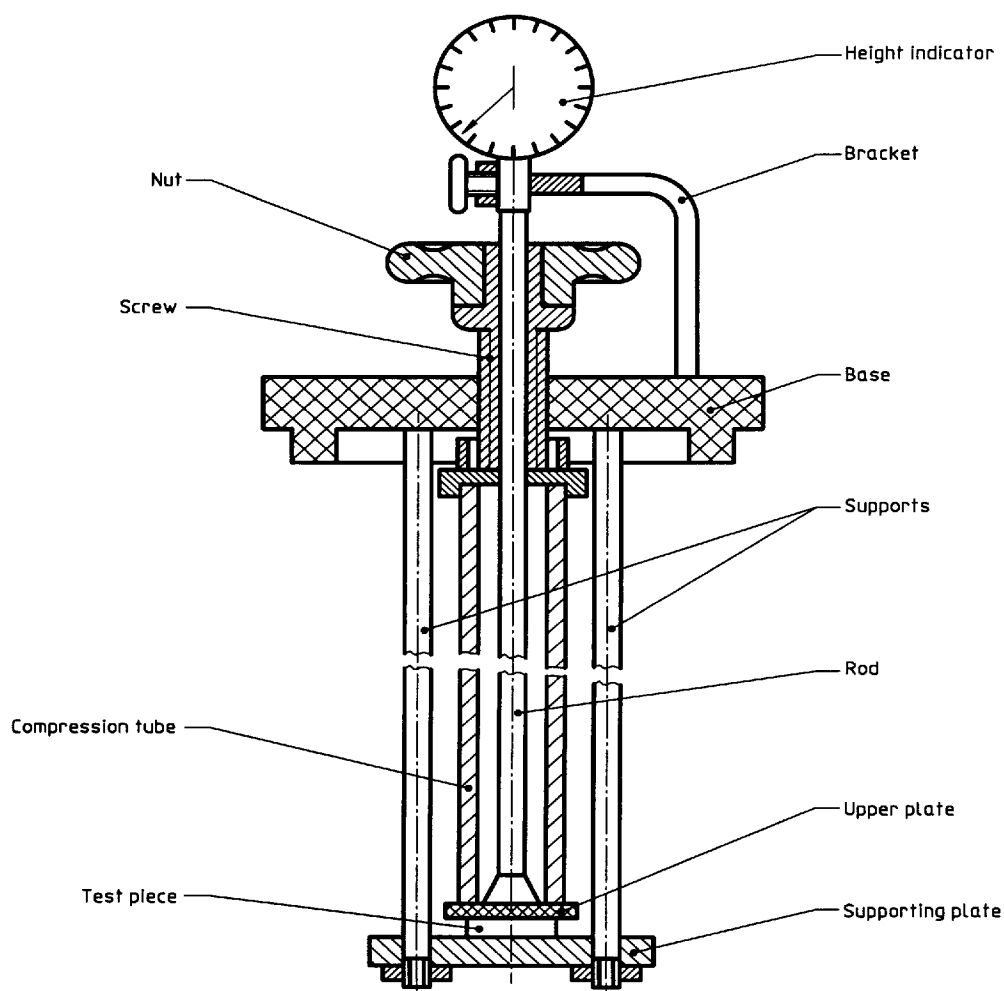Suitable apparatus is shown in figure 1.

Figure 1 — Apparatus for determination of crystallization under compression

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3) Apparatus similar to that described in ISO 815 is suitable provided that it is capable of compressing the test pieces to 60 % at standard temperature.

2

5.2 Height-measuring device, capable of measuring the height of the test piece with an accuracy of ± 0,01 mm at standard temperature and at the test temperature.

5.3 Low-temperature bath or enclosure.

The heat-transfer medium may be any liquid which remains fluid at the test temperature and which will not affect the materials being tested.

NOTE 1   Liquids which have been found suitable for use at low temperature include acetone, ethanol, butanol and silicone oil.

CAUTION — When working with flammable liquids, appropriate safety precautions should be taken.

When working for periods of 5 h or more, a gaseous medium may be used. Carbon dioxide, air and nitrogen are commonly used gaseous media.

The temperature of the heat-transfer medium shall be controlled to within 1 °C.

5.4 Temperature-measuring device, capable of measuring the temperature to within 1 °C over the range − 75 °C to + 30 °C.

6 Test pieces

6.1 Preparation

Test pieces may be prepared by moulding or, in accordance with ISO 4661-1, by cutting from moulded sheets or finished products.

6.2 Types

Three types of test piece may be used:

— Type 1 (preferred) is a cylinder of diameter 9 mm ± 1 mm and height $(10 \,^{+0,2}_{-0,3})$ mm.

— Type 2 is a cylinder of diameter 13 mm ± 0,5 mm and height 6,3 mm ± 0,3 mm.

— Type 3 is cuboid, 9 mm ± 1 mm square and of height $(10 \,^{+0,2}_{-0,3})$ mm.

NOTE 2   Measurements made with test pieces of different sizes may not give the same results, and comparison of the results obtained in such cases should therefore be avoided.

6.3 Number

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

6.4 Time interval between vulcanization and testing

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

6.5 Conditioning

6.5.1 Test pieces shall be conditioned for a period of 3 h immediately before testing at one of the standard temperatures specified in ISO 471.

6.5.2 When testing rubbers which crystallize at standard temperature (for example those based on polychloroprene or polyurethane rubbers), the test piece shall be heated before testing at 70 °C ± 1 °C for 30 min ± 3 min, then cooled to standard temperature for 30 min ± 3 min and tested immediately.

7 Test conditions

7.1 If it is desired to study the time dependence of crystallization, the tests may be carried out at any desired temperature, degree of compression or holding time (see 9.3).

7.2 For the rapid determination of the relative tendency of rubbers to crystallize, and for comparing rubbers of similar composition, the tests shall be carried out using the temperature, degree of compression and holding time given in table 1 for the particular type of rubber concerned.

8 Procedure

8.1 Determination of recovery in the absence of crystallization

8.1.1 Measure the initial height h_0 of the uncompressed test piece, in millimetres to the nearest 0,01 mm, at standard temperature, using method A specified in ISO 4648:1991 or with the height-measuring device (5.2) in the test apparatus (see figure 1), at a pressure of 20 kPa ± 3 kPa.

3

ISO 6471:1994(E)

Table 1

Type of rubber	Temperature θ_1 °C	Nominal compression %		Holding time min	
		e_0	e_1	t_0	t_1
Natural	− 25	20	60	10	60
Synthetic polyisoprene	− 25	20	60	10	60
Polychloroprene	− 10	20	60	10	60
Polybutadiene	− 55	20	60	10	60
Polydimethylsiloxane	− 55	20	50	5	30
Polyphenylsiloxane	− 70	50	50	5	30
Polyurethane	+ 5	20	60	10	30

8.1.2 Calculate to the nearest 0,1 mm the test piece height h_1 required to give the degree of compression e_0 specified in 7.2, using the equation

$$h_1 = h_0\left(1 - \frac{e_0}{100}\right)$$

8.1.3 Compress the test piece in the compression device (5.1) at standard temperature to the required height h_1 (see 8.1.2).

8.1.4 Maintain the device with the compressed test piece at standard temperature for 60 s ± 10 s. If necessary, it is permissible to increase this time to 24 h, but results can only be compared if the same time has been used.

8.1.5 Place the compressed test piece in the low-temperature bath (5.3) at the test temperature θ_1, and maintain it at this temperature for the holding time t_0 (see 7.2). Release the test piece from loading without removing it from the bath and hold it in the bath at the test temperature θ_1 for a recovery time t_2 of 10 min ± 1 min. Measure the height h_2 of the recovered test piece at the test temperature θ_1, to the nearest 0,01 mm.

8.2 Determination of recovery after crystallization

In general, new test pieces should be used, but, if necessary, the test pieces used for the test in the absence of crystallization (see 8.1) may be used. If the same test pieces are used, and if the rubber does not crystallize at room temperature, it shall first be conditioned for at least 30 min at standard temperature. If it does crystallize at standard temperature, it shall be conditioned as specified in 6.5.2.

8.2.1 Measure the initial height of the test piece h_0, in millimetres, at standard temperature, using method A specified in ISO 4648:1991.

8.2.2 Carry out the procedure specified in 8.1.2 to 8.1.5 with the same test temperature θ_1 and recovery time t_2, but with different values of compression e_1 and holding time t_1 (see 7.2). Measure the height h_3 of the recovered test piece.

9 Expression of results

9.1 Recovery

Calculate the values of the recovery in the absence of crystallization K_0 and after crystallization K_1 from the equations

$$K_0 = \frac{h_2 - h_1}{h_0 - h_1}$$

and

$$K_1 = \frac{h_3 - h_1}{h_0 - h_1}$$

where

h_0 is the initial height of the test piece;

h_1 is the height of the compressed test piece;

h_2 is the height of the test piece after release and recovery in the absence of crystallization;

h_3 is the height of the test piece after release and recovery after crystallization.

Record the values of K_0 and K_1 to the nearest ± 0,01.

Calculate the arithmetic-mean values of K_0 and K_1. The individual values of the test pieces should agree to within ± 0,05 of the arithmetic mean. If they do not, a further three test pieces shall be tested and the arithmetic-mean value of all the results reported.

4

9.2 Tendency to crystallize

The tendency to crystallize, y, is given by the equation

$$y = 1 - R$$

where

$$R = \frac{K_1}{K_0}$$

(This is valid only if $K_0 \geqslant 0,3$.)

9.3 Half-time to crystallization

If necessary, plot a graph of R against the holding time t_1 (or lg t_1) for each compression and test temperature, and draw a smooth curve through the points. From this curve, determine the half-time to crystallization, t_H [i.e. the time after which $R = 0,5$ (see figure 2)].

10 Test report

The test report shall include the following information:

a) Test piece details:

   1) a full description of the test piece and its origin;

2) details of the compound, cure time and temperature, if appropriate;

3) the type of test piece;

4) the method of preparation of the test piece from the sample.

b) The test method and test details:

   1) a reference to this International Standard;

   2) the type of compression device used and the heat-transfer medium;

   3) the standard temperature used;

   4) the conditioning procedure used;

   5) the test temperature (θ_1);

   6) the degrees of compression (e_0 and e_1);

   7) the holding times (t_0 and t_1);

   8) the time between compression and cooling of the test piece.
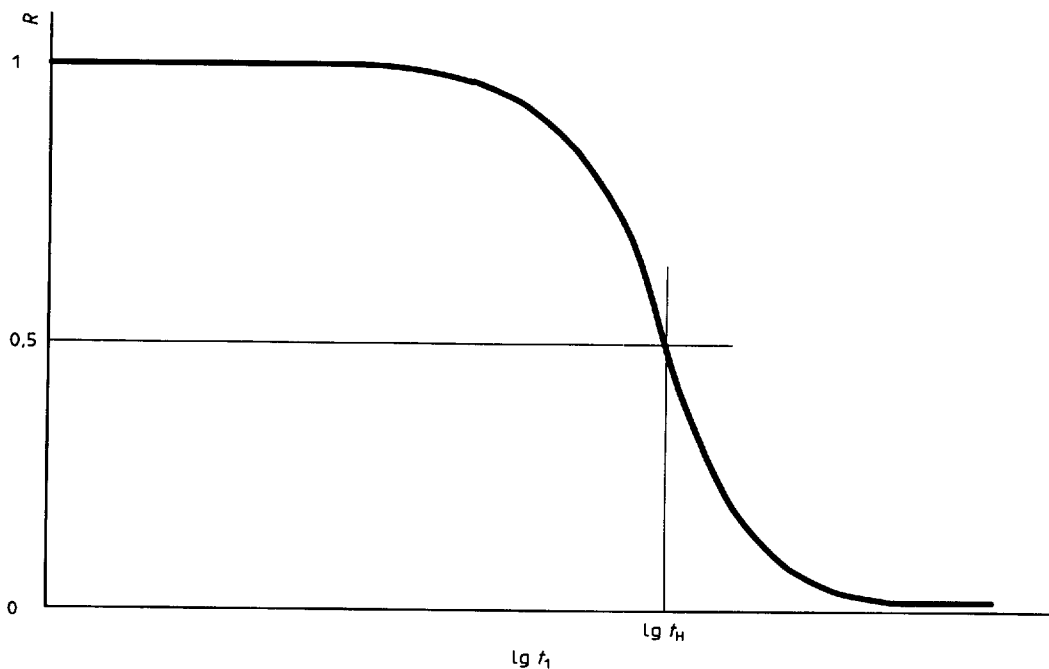

Figure 2 — Determination of half-time to crystallization

ISO 6471:1994(E)

c) The test results:

   1) the number of test pieces tested;

   2) the mean values of R and y;

3) the half-time to crystallization, if determined;

4) the individual results, as required.

d) The date of the test.

6

ISO 6471:1994(E)

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