International Standard 6471

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Rubber, vulcanized — Determination of crystallization effects under compression

Caoutchouc vulcanisé — Détermination des effets de la cristallisation sous compression

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Foreword

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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 6471 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in March 1982.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Portugal
Austria	India	Romania
Belgium	Italy	South Africa, Rep. of
Canada	Korea, Rep. of	Spain
China	Mexico	Sweden
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	USSR
France	Poland	

No member body expressed disapproval of the document.

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Rubber, vulcanized — Determination of crystallization effects under compression

1 Scope and field of application

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 References

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

ISO 1653, *Vulcanized rubbers — Determination of compression set under constant deflection at low temperatures.*

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

ISO 3387, *Rubbers — Determination of crystallization effects by hardness measurements.*

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

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

3 Principle

Compression of a test piece under low strain and determination of the recovery (in the absence of crystallization). Compression of a test piece under high strain and determination of the recovery (after crystallization). From these values, evaluation of the tendency of the rubber to crystallize.

4 Apparatus[1)]

4.1 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 test duration 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 by conduction through metal parts

1) Apparatus similar to that described in ISO 1653 is suitable provided that it is capable of compressing the test pieces to 60 % at standard laboratory temperature.

which are connected with 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.

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

4.3 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 — Among the liquids which have been found suitable for use at low temperatures are acetone, ethanol, butanol and silicone fluid.

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 or nitrogen are commonly used gaseous media.

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

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

5 Test pieces

5.1 Preparation

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

5.2 Types

Three types of test pieces may be used :

— Type 1 (preferred) comprises a cylinder, of diameter 9 ± 1 mm and of height $10 \pm \begin{matrix}0,2\\0,3\end{matrix}$ mm.

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

— Type 3 is cuboid, 9 ± 1 mm square and of height $10 \pm \begin{matrix}0,2\\0,3\end{matrix}$ mm.

NOTE — 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.

5.3 Number

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

5.4 Time interval between vulcanization and testing

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

5.5 Conditioning

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

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

6 Test conditions

6.1 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 8.3).

6.2 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 temperatures, degrees of compression and holding times given in the table, according to the type of rubber.

Table

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

7 Procedure

7.1 Determination of recovery in the absence of crystallization

7.1.1 Measure the initial height, h_0, of the uncompressed test piece, in millimetres to the nearest 0,01 mm, at standard laboratory temperature, using the method A1 specified in ISO 4648 or with the height measuring device in the test device (see 4.2 and figure 1) with a pressure of 20 ± 3 kPa.

7.1.2 To the nearest 0,1 mm, calculate the test piece height, h_1, required to give the degree of compression e_0 specified in 6.2, using the equation

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

7.1.3 Compress the test piece in the compression device (4.1) at standard laboratory temperature to the required height h_1 (7.1.2).

7.1.4 Maintain the device with the compressed test piece at standard laboratory temperature for 60 ± 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.

7.1.5 Place the compressed test piece in a low temperature bath at the test temperature θ_1, and maintain it at this temperature for the holding time t_0 (see 6.2). 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 ± 1 min. Measure the height of the recovered test piece, h_2, at the test temperature θ_1, to the nearest 0,01 mm.

7.2 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 (7.1) 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 laboratory temperature. If it does crystallize at standard laboratory temperature, it shall be conditioned as specified in 5.5.2.

7.2.1 Measure the initial height of the test piece, h_0, in millimetres, at standard laboratory temperature using the method specified in ISO 4648.

7.2.2 Carry out the procedure specified in 7.1.2 to 7.1.5 with the same test temperature θ_1 and recovery time t_2, but with different values of compression (e_1) and of holding time (t_1) (see 6.2). Measure the height of the recovered test piece, h_3.

8 Expression of results

8.1 Recovery

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

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

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 shall be reported.

8.2 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 $K_0 \geqslant 0{,}3$.)

8.3 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)].

9 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 laboratory temperature,

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;

c) 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) individual results, as required;

d) the date of test.