INTERNATIONAL STANDARD

ISO 812

Fourth edition
2017-07

Rubber, vulcanized or thermoplastic — Determination of low-temperature brittleness

Caoutchouc vulcanisé ou thermoplastique — Détermination de la fragilité à basse température

Reference number
ISO 812:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This fourth edition cancels and replaces the third edition (ISO 812:2011), of which it constitutes a minor revision.

The changes compared to the previous edition are as follows:

— the normative references in Clause 2 have been updated;

— the definition of "failure" has been added in 3.4;

— a note has been added in 4.2.

Rubber, vulcanized or thermoplastic — Determination of low-temperature brittleness

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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.

WARNING 2 — Certain procedures specified in this document 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 document specifies a method for determining the lowest temperature at which rubber materials do not exhibit brittle failure or the temperature at which half of the test pieces used in a test fail when impacted under specified conditions.

The temperatures thus determined do not necessarily relate to the lowest temperature at which the material can be used since the brittleness will be affected by the conditions of test and especially by the rate of impact. Data obtained by this method are, therefore, intended to be used to predict the behaviour of rubbers at low temperatures only in applications in which the conditions of deformation are similar to those specified in the test.

Three procedures are described:

— procedure A, in which the brittleness temperature is determined;

— procedure B, in which the brittleness temperature for 50 % failure is determined;

— procedure C, in which the test piece is impacted at a specified temperature.

Procedure C is used in the classification of rubber materials and for specification purposes.

NOTE A similar test for rubber-coated fabrics is described in ISO 4646.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 18899:2013, *Rubber — Guide to the calibration of test equipment*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
brittleness temperature
lowest temperature at which none of a set of test pieces fractures due to low-temperature embrittlement when tested under the specified conditions

3.2
50 % brittleness temperature
temperature at which 50 % of a set of test pieces fractures due to low-temperature embrittlement when tested under the specified conditions

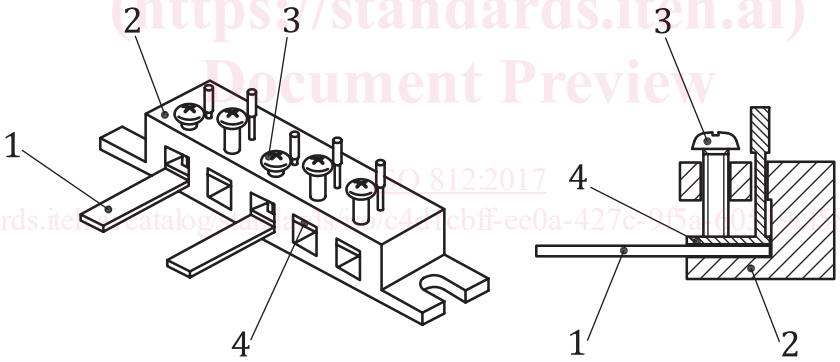
3.3
testing speed
relative linear velocity at impact between the striking edge of the test apparatus and a clamped test piece

3.4
failure
any crack, fissure or hole visible to the naked eye, or complete separation into two or more pieces

4 Apparatus and materials

4.1 Test piece clamp and striker, meeting the requirements of 4.1.1 to 4.1.3.

4.1.1 The test piece clamp shall be rigid and designed to hold the test piece(s) as cantilever beam(s). Each individual test piece shall be held firmly and securely in the clamp without distortion. A suitable example of a clamp is shown in Figure 1.



Key
1 test piece
2 body of clamp
3 holding screw
4 test piece holder

Figure 1 — Example of test piece clamp

4.1.2 The striking edge shall move relative to the test piece(s) along a path normal to the upper surface of the test piece(s) at a linear testing speed of 2,0 m/s ± 0,2 m/s at impact. The speed shall be maintained within this range for at least 6 mm of travel following the impact.

In order to obtain a speed within the specified limits during and after impact, care shall be taken to ensure that the striking energy is sufficient. It has been found that a striking energy of at least 3,0 J per test piece is necessary. It might, therefore, be necessary to limit the number of test pieces impacted at one time.

4.1.3 The principal dimensions of the apparatus [see Figures 2 a) and b)] shall be as follows:

a) the striking edge shall have a radius of 1,6 mm ± 0,1 mm;

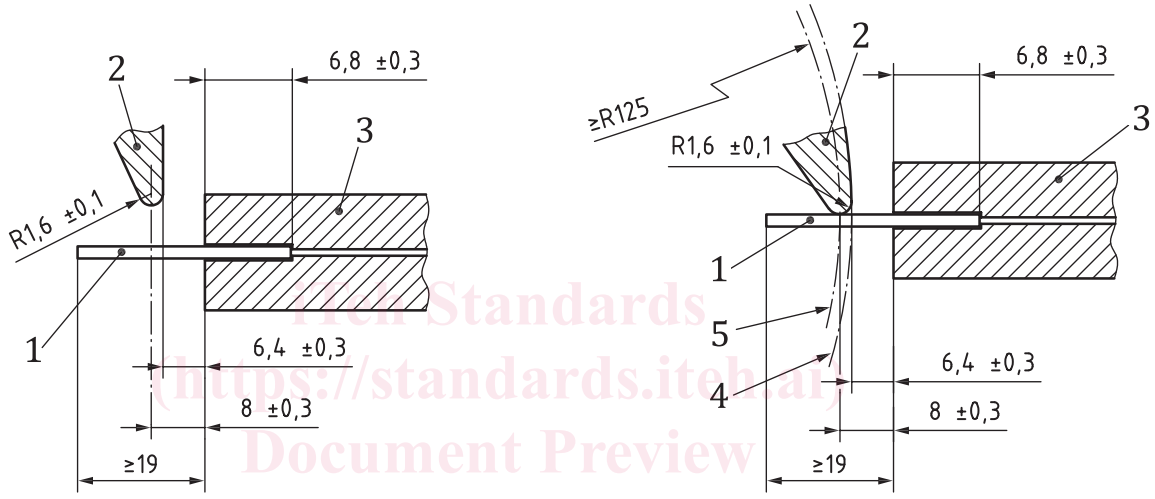
b) the clearance between the striker and the test piece clamp at impact shall be 6,4 mm ± 0,3 mm;

c) the separation between the point of impact of the striking edge and the test piece clamp shall be 8 mm ± 0,3 mm;

d) the clamping length of the test piece clamp shall be 6,8 mm ± 0,3 mm.

NOTE Commercial apparatus is available meeting the requirements of this document in which the striking edge is rotated by a motor or travels in a straight line under the action of a solenoid, gravity or a spring. A method for the speed calibration of a solenoid-actuated low-temperature impact tester is given in Annex A.

Dimensions in millimetres



a) Straight-line motion **b) Rotational motion**

Key

1 test piece
2 striker
3 test piece clamp
4 locus described by point on striker closest to test piece clamp
5 locus described by impact point on striker

Figure 2 — Test piece clamp and striker

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 can 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 are usually suitable owing to their chemical inertness towards rubbers, their non-flammability and their non-toxicity;

NOTE A kinematic viscosity of about 5 mm^2/s at ambient temperature has been found suitable.

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.

5 Calibration

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

6 Test pieces

Test pieces shall be either

— type A: a strip 26 mm to 40 mm long, 6 mm ± 1 mm wide and 2,0 mm ± 0,2 mm thick, or

— type B: a test piece 2,0 mm ± 0,2 mm thick and of the shape and dimensions given in Figure 3.

Test pieces shall be prepared in accordance with ISO 23529. They shall normally be punched from sheet using a suitable sharp die. Alternatively, type A test pieces can be prepared using sharp, parallel double-bladed cutters, in a single stroke. The strip so formed is then cut to the correct length.

Dimensions in millimetres

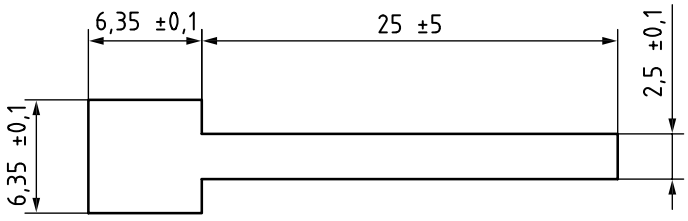


Figure 3 — Type B test piece

7 Time interval between manufacture and testing

Unless otherwise specified, the time interval between the date the material was formed and testing shall be in accordance with ISO 23529.

8 Procedure

8.1 Procedure A (determination of brittleness temperature)

8.1.1 Bring the bath or test chamber to a temperature below the expected lowest temperature of non-failure. In the case of a liquid heat-transfer medium, place sufficient liquid in the bath to ensure approximately 25 mm depth or more of the liquid over the test piece(s). Pre-cool the test piece clamp by immersing it in the cooled bath or test chamber.

8.1.2 Quickly mount the test piece(s) in the test piece clamp and immerse them for 5 min at the test temperature when using a liquid medium or for 10 min when using a gaseous medium (see also ISO 23529).

NOTE 1 For very soft materials, it might be necessary to use a device to support the test piece horizontally until just before the striker is released.

The free length of the test piece(s) shall be greater than 19 mm.

Test five type A or type B test pieces. If the available striking energy has the minimum value specified in 4.1.2, they may all be tested at the same time.

Proper tightening of the clamp is of the utmost importance. The clamp shall be tightened so that each test piece is held with approximately the same clamping torque.

NOTE 2 It has been reported that the temperature at which a test piece fails can be affected by the clamping torque. Clamping to a torque of 0,15 N·m to 0,25 N·m is suggested.

8.1.3 After immersion for the specified time at the test temperature, record the temperature and deliver a single impact blow to the test piece(s).

8.1.4 Remove the test pieces from the test piece clamp and allow them to reach standard laboratory temperature. Examine each test piece to determine whether or not it has failed. Failure is defined as any crack, fissure or hole visible to the naked eye or complete separation into two or more pieces. Where a test piece has not completely separated, bend it to an angle of 90° in the same direction as the bend caused by the impact. Then, examine it for cracks at the bend.

8.1.5 Repeat the test at each of a series of successively higher temperatures 10 °C apart, using a new set of test pieces at each temperature until no failure is obtained. Then, decrease the temperature to the highest value at which a failure was observed and carry out tests at temperatures increasing at 2 °C intervals to determine the temperature at which no failure is observed. Record this as the brittleness temperature.

If crystallization or time-dependent effects of plasticizers are to be studied, longer conditioning periods in a gaseous medium may be used.

8.2 Procedure B (determination of 50 % brittleness temperature)

8.2.1 Carry out the procedure described in 8.1.1 to 8.1.4, except that the starting temperature is that at which 50 % failure is expected.

8.2.2 If all of the test pieces fail at the starting temperature, increase the temperature by 10 °C and repeat the test. If none of the test pieces fail at the starting temperature, decrease the temperature by 10 °C and repeat the test. Increase or decrease the temperature in increments of 2 °C and repeat the test until the lowest temperature at which none of the test pieces fails and the highest temperature at which all of the test pieces fail is determined. Record the number of failures at each temperature. Use a new set of test pieces at each temperature. Determine the 50 % brittleness temperature by calculation using Formula (1) or by the graphical method described in 8.2.4.

8.2.3 Calculation: From the number of failures at each temperature, calculate the percentage of failures at each temperature to determine the 50 % brittleness temperature using Formula (1).

$$T_b = T_h + \Delta T\left(\frac{S}{100} - \frac{1}{2}\right) \quad (1)$$

where

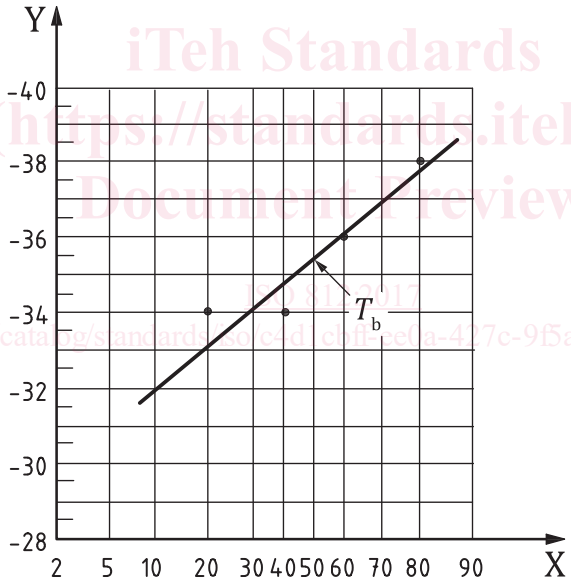
T_b is the 50 % brittleness temperature (°C);

T_h is the highest temperature at which all test pieces fail (°C);

ΔT is the interval between the test temperatures (°C);

S is the total of the percentages of failures at each temperature, from the temperature at which no test piece fails to the temperature at which they all fail, T_h (%).

8.2.4 Graphical method: From the number of failures at each temperature, calculate the percentage of failures at each temperature. Next, using normal probability paper as shown in Figure 4, plot these percentages against the temperature, taking the temperature on the linear scale and the percentage of failures on the probability scale and draw the best-fit straight line through the points. The temperature at the point of intersection of this line and the 50 % probability line is T_b, the 50 % brittleness temperature.



Key

X percentage of failures (%)

Y temperature (°C)

Figure 4 — Determination of 50 % brittleness temperature, T_b, by the graphical method

8.3 Procedure C (testing at a specified temperature)

8.3.1 Carry out the procedure described in 8.1.1 to 8.1.4, except that the temperature used is that specified in the material specification or material classification.

8.3.2 Report the material as satisfactory if no failure is observed in any one of the test pieces or unsatisfactory if any test pieces have failed.

