INTERNATIONAL STANDARD

ISO 815-2

First edition
2008-02-01

Rubber, vulcanized or thermoplastic — Determination of compression set —

Part 2: At low temperatures

Caoutchouc vulcanisé ou thermoplastique — Détermination de la déformation rémanente après compression —

Partie 2: À basses températures

Reference number
ISO 815-2:2008(E)

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Published in Switzerland

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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.

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 815-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Together with ISO 815-1, ISO 815-2 cancels and replaces ISO 815:1991, as well as Technical Corrigendum ISO 815:1991/Cor.1:1993, which have been technically revised. The main modifications concern changes in tolerances to improve the precision of the test method. In addition, a new compression assembly has been specified which makes it possible to perform all measurements inside the low-temperature cabinet without moving the test pieces or opening the cabinet door.

ISO 815 consists of the following parts, under the general title *Rubber, vulcanized or thermoplastic — Determination of compression set*:

— *Part 1: At ambient or elevated temperatures*

— *Part 2: At low temperatures*

Rubber, vulcanized or thermoplastic — Determination of compression set —

Part 2:
At low temperatures

WARNING — Persons using this part of ISO 815 should be familiar with normal laboratory practice. This part of ISO 815 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.

CAUTION — Certain procedures specified in this part of ISO 815 may 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 part of ISO 815 specifies a method for the determination of the compression set characteristics of vulcanized and thermoplastic rubbers at low temperatures.

The method is intended to measure the ability of rubbers of hardness within the range 10 IRHD to 95 IRHD to retain their elastic properties at specified temperatures after prolonged compression at constant strain (normally 25 %) under one of the alternative sets of conditions described. For rubber of nominal hardness 80 IRHD and above, a lower compression strain is used: 15 % for a nominal hardness from 80 IRHD to 89 IRHD and 10 % for a nominal hardness from 90 IRHD to 95 IRHD.

NOTE When rubber is held under compression, physical or chemical changes can occur that prevent the rubber returning to its original dimensions after release of the deforming force. The result is a set the magnitude of which depends on the time and temperature of compression as well as on the time and temperature of recovery. At low temperatures, changes resulting from the effects of glass hardening or crystallization become predominant and, since these effects are reversed by raising the temperature, it is necessary for all measurements to be undertaken at the test temperature.

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 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 23529:2004, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Principle

A test piece of known thickness is compressed at standard laboratory temperature to a defined strain, which is then maintained constant for a specified time at a fixed low temperature. The compression is released and the test piece is allowed to recover at this temperature. The thickness is measured either at intervals after the

release of the strain (so that an assessment of compression set characteristics can be obtained by plotting recovery against time at the low temperature) or a specified time after the release of the strain.

4 Apparatus

4.1 Compression apparatus, consisting of compression plates, spacer(s) (optional), a thickness gauge, a temperature sensor and a device for applying a specified pressure for thickness measurements. The measurement pressure for thickness measurements is the same as that specified in ISO 23529.

The compression apparatus shall be capable of applying the compression and maintaining it during the whole duration of the test, and it shall be possible to keep the apparatus in a low-temperature cabinet at the specified test temperature. Care shall be taken to ensure that there is as little influence on the test as possible by the conduction of heat away from the test piece, for example through metal parts which are connected with the outside of the low-temperature cabinet.

The part of the apparatus with the compression plates and the test piece shall be within the low-temperature cabinet during the whole of the test, but the thickness gauge may be located outside the cabinet.

An example of a compression apparatus is shown in Figure 1.

It shall be possible to release the compression of the test piece without opening the low-temperature cabinet.

An example of a quick-release mechanism for this purpose is shown in Figure 2.

4.1.1 Compression plates, comprising a pair of parallel, flat, highly polished chromium-plated steel or highly polished stainless-steel plates, between the faces of which the test piece is compressed. The finish Ra of the surface of the compression plates shall be not worse than 0,4 µm from the mean line of the profile (see ISO 4287). The plates shall be

— sufficiently rigid to ensure that, with a test piece under load, no compression plate bends by more than 0,01 mm;

— of sufficient size to ensure that the whole of the test piece, when compressed between the plates, remains within the area of the plates.

4.1.2 Steel spacer(s), to provide the required compression, if necessary. Whether spacer(s) will need to be used or not will depend on the design of the compression apparatus.

If used, the spacer(s) shall be of such size and shape that contact with the compressed test piece is avoided.

The height of the spacer(s) shall be chosen so that the compression applied to the test piece is

(25 ± 2) % for hardnesses below 80 IRHD;

(15 ± 2) % for hardnesses between 80 IRHD and 89 IRHD;

(10 ± 1) % for hardnesses of 90 IRHD and higher.

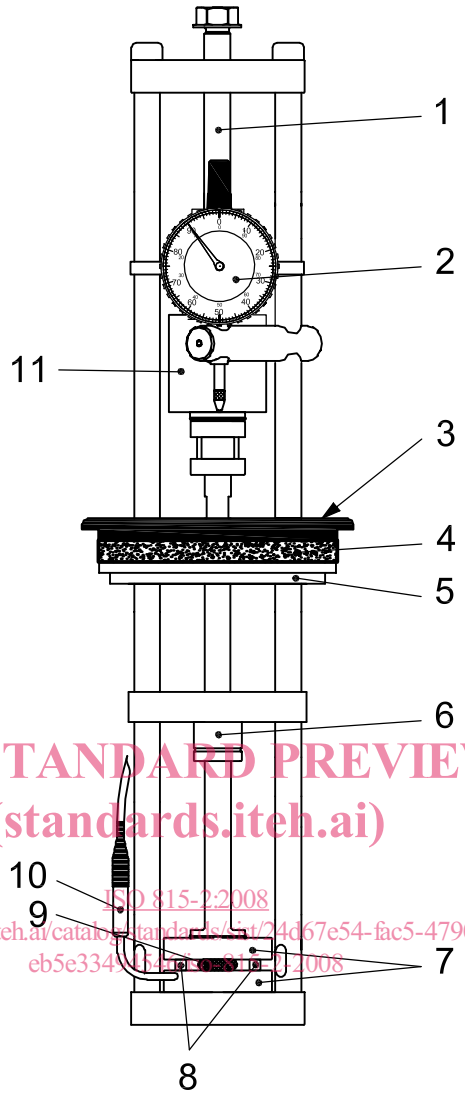
4.1.3 Thickness gauge, with an accuracy of $\pm$ 0,01 mm, exerting a pressure of 22 kPa $\pm$ 5 kPa for solid rubber of hardness equal to or greater than 35 IRHD or a pressure of 10 kPa $\pm$ 2 kPa if the hardness is less than 35 IRHD. The thickness gauge shall have a device for applying a pre-load by means of a deadweight or load-cell system so that it exerts the specified pressure.

The thickness measurement may also be done using the compression plates.

For comparative purposes, the same dimensions of the circular foot shall be used.

NOTE When using a digital gauge, a resolution of 0,001 mm is needed to obtain the required accuracy.

4.1.4 Temperature measurement device, inserted in one of the plates, measuring the temperature directly with an accuracy of $\pm$ 0,5 °C.



Key

1 screw
2 thickness measurement gauge
3 rubber lid
4 insulating lid
5 aluminium lid
6 linear bearing
7 compression plates
8 spacers (optional)
9 test piece
10 temperature sensor
11 weight for pre-loading test piece

Figure 1 — Example of a compression apparatus

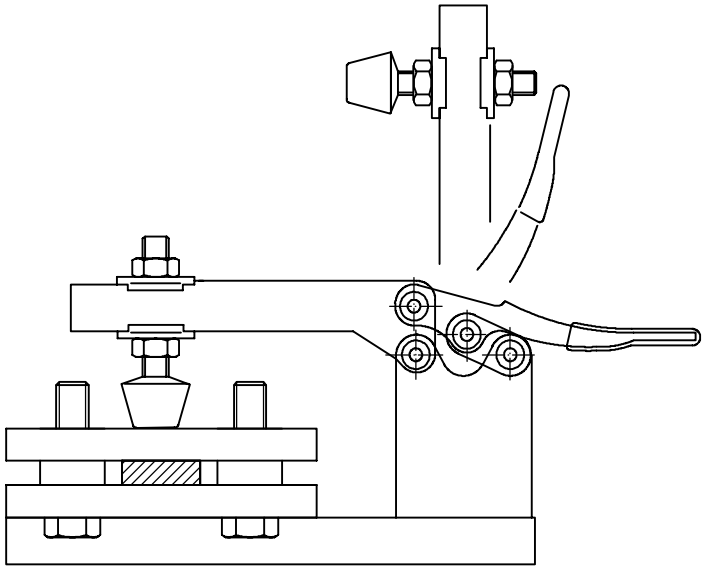


Figure 2 — Example of a quick-release mechanism

4.2 Low-temperature cabinet, capable of maintaining the compression apparatus and test pieces at the test temperature within the tolerance limits specified in 6.2. The low-temperature cabinet may be mechanically refrigerated or it may be cooled directly by dry ice or liquid nitrogen.

The cabinet shall be designed so that it is possible to release the test pieces and carry out the subsequent thickness measurements without direct contact, e.g. by means of hand-holes fitted with gloves or a remote-handling device. The cabinet shall be capable of maintaining the temperature within the specified limits while these operations are being carried out.

The time to reach a steady-state temperature depends on the type of cabinet and the overall heat capacity of the compression apparatus. To obtain test results which can be compared with each other, it is necessary, in the case of a 24 h test, to reach the steady-state temperature, within the specified limits, in the interior of the test pieces in not more than 3 h.

4.3 Timing device, for measuring the recovery time, with a precision of ± 1 s.

5 Test pieces

5.1 Dimensions

The test pieces shall be one of two sizes, designated type A and type B:

— type A: a cylindrical disc of diameter 29 mm ± 0,5 mm and thickness 12,5 mm ± 0,5 mm;

— type B: a cylindrical disc of diameter 13 mm ± 0,5 mm and thickness 6,3 mm ± 0,3 mm.

These two types do not necessarily give the same values for compression set, and comparison of results obtained using test pieces of different sizes shall be avoided when comparing one compound with another.

Type A test pieces are preferred for testing rubbers having low compression set, because of the greater accuracy attainable using these larger test pieces.

Type B test pieces are preferred when it is required to cut test pieces from products. In this case, the test pieces shall be taken as near to the centre of the product as possible, unless otherwise specified. When possible, the test piece shall be cut in such a way that its axis is parallel to the direction of compression of the product in service.

5.2 Preparation

The test pieces shall be prepared by moulding each disc, whenever possible. Preparation by cutting out each disc or by laminating not more than three discs is permitted. The use of test pieces prepared by laminating several discs for control of finished products shall be agreed between interested parties.

Cutting shall be performed in accordance with ISO 23529. When cupping (the formation of a concave surface) is a problem, the test piece shape can be improved by cutting it in two stages: first cut an oversize test piece and then trim it to the exact dimensions with a second cutter.

Laminated test pieces shall conform to the dimensions specified in 5.1 and shall be prepared by laminating discs or rubber cut from sheets without adhesives. Discs may be compressed by a few percent for 1 min so that they stick together. The number of discs laminated to produce a test piece shall not exceed three. The total thickness shall then be measured,

Test pieces prepared by the different methods described above may give different results and comparison of values shall be avoided.

NOTE Attention is drawn to the marked effects of the state of cure on compression set values. It may be necessary to adjust the cure of moulded test pieces to be representative of different thicknesses of sheets or mouldings.

5.3 Number

Three test pieces shall be tested separately or at the same time, depending on the purpose of the test.

5.4 Time-interval between production and testing

For all test purposes, the minimum time between production and testing shall be 16 h.

For non-product tests, the maximum time between production and testing shall be 4 weeks and, for evaluations intended to be comparable, the tests, as far as possible, shall be carried out after the same time-interval.

For product tests, whenever possible, the time between production and testing shall not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt of the product by the purchaser (see ISO 23529).

5.5 Conditioning

Samples and test pieces shall be protected from light and heat as much as possible during the interval between production and testing.

In the case of crystallization studies, test pieces shall be conditioned (to remove any existing crystallization) immediately before testing by heating them in an oven at 70 °C for 45 min. They shall then be conditioned at a standard laboratory temperature.

Prepared test pieces shall be conditioned immediately before testing for a minimum period of 3 h at one of the standard laboratory temperatures specified in ISO 23529. The same temperature shall be used throughout any one test or series of tests intended to be comparable.

Test pieces of thermoplastic rubbers shall be annealed before testing by heating in an oven at a temperature and for a length of time that are appropriate to the material in order to release internal stresses caused by the moulding process. They shall then be conditioned at a standard laboratory temperature.

NOTE 70 °C for 30 min is suitable for many materials.