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ISO TC 45/SC 2/WG 2

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Rubber, vulcanized or thermoplastic — Determination of compression set — Part 1: At ambient or elevated temperatures

Caoutchouc vulcanisé ou thermoplastique — Détermination de la déformation rémanente après compression — Partie 1: A températures ambiantes ou élevées

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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.

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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).

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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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 815-1:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

- normative references have been updated in Clause 2.
- a new precision statement has been added in Annex A.

A list of all parts in the ISO 815 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber, vulcanized or thermoplastic — Determination of compression set — Part 1: At ambient or elevated temperatures

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 determine the applicability of any other restrictions.

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 methods for the determination of the compression set characteristics of vulcanized and thermoplastic rubbers at ambient (one method) or elevated temperatures (three methods, A, B, and C, depending on the way the test piece is released at the end of the test).

The methods are 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 1 When rubber is held under compression, physical or chemical changes that prevent the rubber returning to its original dimensions after release of the deforming force can occur. The result is a set, the magnitude of which depends on the time and temperature of compression as well as on the time, temperature, and conditions of recovery. At elevated temperatures, chemical changes become increasingly more important and lead to a permanent set.

NOTE 2 Short-time compression set tests, typically for 24 h, at elevated temperatures are commonly used as a measure of the state of cure, a means of material classification, and a specification to ensure the quality of a compound. Longer tests, typically for 1 000 h, at elevated temperatures take account of the effect of ageing and are often used to predict service performance, including that of sealing materials. Short-time tests at ambient temperature show mainly the effect of physical changes (re-orientation of the molecular chains and the fillers).

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 188:2011, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

ISO 23529:2016, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

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3 Terms and definitions

No terms and definitions are listed in this document.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

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4 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 standard laboratory temperature or a fixed elevated temperature. The compression is released and, after the test piece has been allowed to recover at a standard laboratory temperature or the elevated temperature for a specified time, the thickness of the test piece is again measured.

5 Apparatus

5.1 Compression assembly, consisting of compression plates, steel spacers, and clamping device.

A typical assembly is shown in Figure 1.

5.1.1 Compression plates, consisting of two parallel, flat, highly polished plates made of chromium-plated steel or stainless-steel or any corrosion-resistant material between the faces of which the test piece is compressed.

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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 and can expand freely laterally.

NOTE A surface finish not worse than Ra 0,4 μm (see ISO 4287) has been found to be suitable. Such an Ra can be obtained by a grinding or polishing operation.

5.1.2 Steel spacer(s), to provide the required compression.

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

- $(25 \pm 2) \%$ for hardnesses below 80 IRHD,
- $(15 \pm 2) \%$ for hardnesses between 80 IRHD and 89 IRHD,
- $(10 \pm 1) \%$ for hardnesses of 90 IRHD and higher.

5.1.3 Clamping device, a simple screw device (see Figure 1) is adequate.

5.2 Oven, in accordance with the requirements specified in ISO 188:2011, method A or method B, and capable of maintaining the compression assembly and test pieces at the test temperature within the tolerance specified in 8.2.

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NOTE Test results obtained with ovens for method A can be different from those obtained with ovens for method B.

The time to reach a steady-state temperature depends on the type of oven and the overall heat capacity of the compression assembly. To obtain comparable results in the case of an elevated test temperature and a 24 h test duration, it is necessary to reach the steady-state temperature within the specified tolerances in the interior of the test pieces in not more than 3 h.

5.3 Pair of tongs, for handling the test pieces.

5.4 Thickness gauge, with an accuracy of $\pm 0,01$ mm (see ISO 23529:2016, 9.1), having a flat circular foot of $4,0 \text{ mm} \pm 0,5 \text{ mm}$ in diameter and a flat solid base-plate and exerting a pressure of $22 \text{ kPa} \pm 5 \text{ kPa}$ for solid rubber of hardness equal to or greater than 35 IRHD, or a pressure of $10 \text{ kPa} \pm 2 \text{ kPa}$ if the hardness is less than 35 IRHD.

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

After testing at elevated temperature, an unexpected deformation of the test piece is sometimes observed. More particularly, the two flat surfaces can be deformed, which complicates the thickness measurement. In this case, the diameter of the gauge used to measure the thickness should be chosen carefully to allow precise measurement.

5.5 Timing device, for measuring the recovery time, with an accuracy of ± 1 s.

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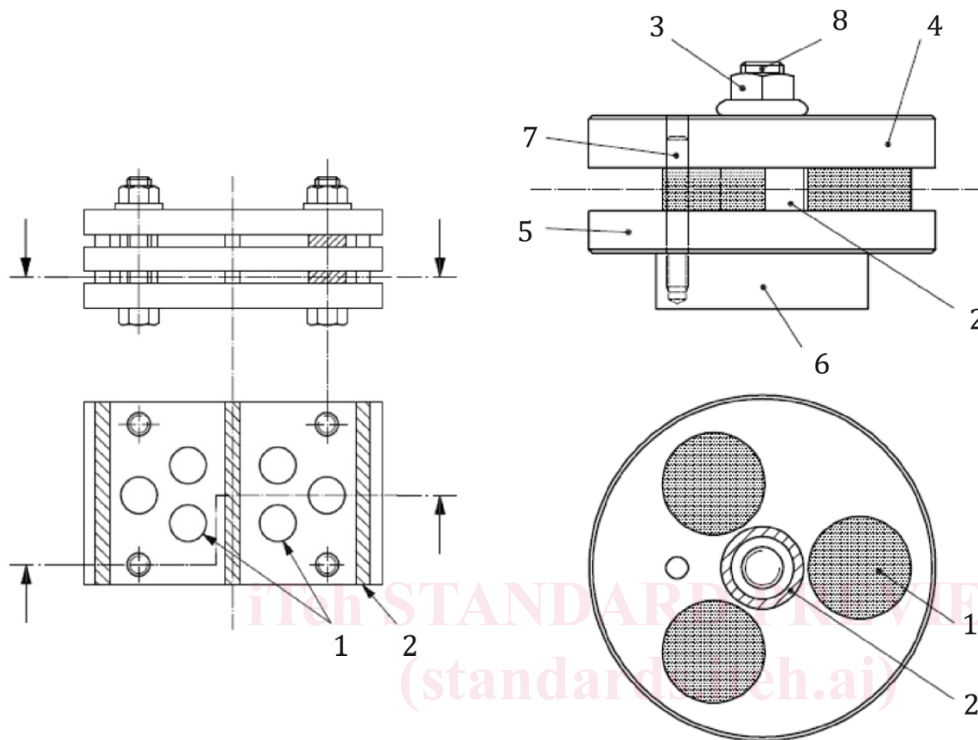
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a) Example 1

b) Example 2

Key

- 1 test piece
- 2 spacer
- 3 nut
- 4 upper plate

- 5 lower plate
- 6 part formed for clamping in a device
- 7 locating pin
- 8 screw

Figure 1 — Examples of assemblies for the determination of compression set

6 Calibration

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

7 Test pieces

7.1 Dimensions

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

- Type A: a cylindrical disc of diameter $29,0 \text{ mm} \pm 0,5 \text{ mm}$ and thickness $12,5 \text{ mm} \pm 0,5 \text{ mm}$.
- Type B: a cylindrical disc of diameter $13,0 \text{ mm} \pm 0,5 \text{ mm}$ and thickness $6,3 \text{ mm} \pm 0,3 \text{ mm}$.

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.

7.2 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 7.1 and shall be prepared by laminating discs or rubber cut from sheets without adhesives. Discs can 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 various methods described above can 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 might be necessary to adjust the cure of moulded test pieces to be representative of different thicknesses of sheets or mouldings.

7.3 Number of test pieces

A minimum of three test pieces shall be tested, separately or as a set.

7.4 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 three months. In other cases, tests shall be made within two months of the date of receipt of the product by the purchaser (see ISO 23529).

7.5 Conditioning

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

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.

8 Test conditions

8.1 Duration of test

The exposure time shall be 24_{-2}^{+0} h, 72_{-2}^{+0} h, (168 ± 2) h, or multiples of 168 h, measured from the moment of placing the compression assembly in the oven (5.2).

8.2 Temperature of test

The temperature of test shall be one of the standard laboratory temperatures $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ or $27^{\circ}\text{C} \pm 2^{\circ}\text{C}$ (see ISO 23529) for tests at ambient temperature, and one of the following temperatures for tests at elevated temperatures: $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$, $55^{\circ}\text{C} \pm 1^{\circ}\text{C}$, $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$, $85^{\circ}\text{C} \pm 1^{\circ}\text{C}$, $100^{\circ}\text{C} \pm 1^{\circ}\text{C}$, $125^{\circ}\text{C} \pm 2^{\circ}\text{C}$, $150^{\circ}\text{C} \pm 2^{\circ}\text{C}$, $175^{\circ}\text{C} \pm 2^{\circ}\text{C}$, $200^{\circ}\text{C} \pm 2^{\circ}\text{C}$, $225^{\circ}\text{C} \pm 2^{\circ}\text{C}$, or $250^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

NOTE As oven temperatures are increased, the results become increasingly dependent upon the thermal stability of the rubber. At still higher temperatures, surface oxidation of the test piece makes a significant contribution to the observed compression set. There is no simple correlation between the compression set observed at elevated temperatures and that observed at room temperature.

9 Procedure

9.1 Preparation of compression assembly

With the compression assembly (5.1) at standard laboratory temperature, carefully clean the operating surfaces. Apply a thin coating of lubricant to the faces of the compression plates (5.1.1) that will come into contact with the test pieces. The lubricant used shall have no substantial action on the rubber during the test and it shall be described in the test report (see Clause 12).

NOTE For most purposes, a silicone or fluorosilicone liquid having a nominal kinematic viscosity of $100 \text{ mm}^2/\text{s}$ at standard laboratory temperature is a suitable lubricant.

If for any reason a lubricant is not used, this shall be mentioned in the test report.

9.2 Thickness measurement

Measure the thickness at the centre of each test piece to the nearest 0,01 mm, at standard laboratory temperature.

9.3 Applying the compression

Place the test pieces between the pairs of compression plates together with the spacer(s) (5.1.2), avoiding contact between test pieces and bolts or spacer(s). Tighten the clamping device (5.1.3), so that the plates are drawn together uniformly until they are in contact with the spacer(s). The applied compression shall be $(25 \pm 2) \%$ of the original thickness of the test piece except for higher hardnesses, for which the applied compression shall be $(15 \pm 2) \%$ or $(10 \pm 1) \%$ (see 5.1.2).

9.4 Starting the test

If the tests are conducted at elevated temperature, introduce the compression assembly containing the test pieces without delay into the central part of the oven (5.2) operating at test temperature (see 8.2).

If the tests are conducted at ambient temperature, keep the compression assembly containing the test pieces in an air-conditioned room at standard laboratory temperature (see ISO 23529).

9.5 Terminating the test

9.5.1 At ambient temperature

If the test is conducted at ambient temperature, release the test pieces after the required test duration (see 8.1) and transfer them to a wooden bench. Leave them to recover for 30 min ± 3 min at standard laboratory temperature and then measure their thickness.

9.5.2 At elevated temperature

Method A: After the required test duration (see 8.1), remove the compression assembly from the oven, immediately release the test pieces and transfer the test pieces quickly to a wooden bench. Leave them to recover at a standard laboratory temperature for 30 min ± 3 min, and then measure their thickness.

Method A shall be used unless otherwise specified.

Method B: After the required test duration, remove the compression set assembly from the oven, allow it to cool to a standard laboratory temperature, but for a minimum of 30 min and a maximum of 120 min, then release the test pieces and, after a further 30 min ± 3 min at standard laboratory temperature measure their thickness.

Method C: After the required test duration, do not remove the compression assembly from the oven but immediately release the test pieces and keep them in the oven. Leave them to recover at the test temperature for 30 min ± 3 min and, after a further 30 min ± 3 min at standard laboratory temperature, measure their thickness.

NOTE The temperature of the test piece after release from compression can affect the rate and extent of recovery and hence the value of compression set. Recovery at elevated temperature is generally quicker than at standard laboratory temperature and results in a lower compression set.

9.6 Internal examination

After completing the test, cut the test pieces into two pieces along a diameter. If any internal defects are found, such as gas bubbles, repeat the test.

10 Expression of results

NOTE The rubber industry uses the term equation for the relationships herein termed formula. The term formula is used to describe the table of ingredients in a rubber compound.

The compression set, expressed as a percentage of the initial compression, is given by Formula (1):

$$\frac{h_0 - h_1}{h_0 - h_s} \times 100 \quad (1)$$

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