
**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: À températures ambiantes ou élevées



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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 815-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Together with ISO 815-2, ISO 815-1 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 and the inclusion of the ageing ovens specified in ISO 188, with a view to improving the precision of the method.

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 1: At ambient or elevated 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 methods for the determination of the compression set characteristics of vulcanized and thermoplastic rubbers at ambient or elevated temperatures.

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 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 elevated temperatures, chemical changes become increasingly more important and lead to a permanent set.

NOTE 2 Short-time compression set tests, typically for 22 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 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 188:2007, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

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

ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

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 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.

4 Apparatus

4.1 Compression assembly, consisting of compression plates, steel spacers and a clamping device. Typical assemblies are shown in Figure 1.

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 $0,4\text{ }\mu\text{m}$ 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 $0,01\text{ mm}$;
- 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

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 Clamping device: A simple screw device (see Figure 1) is adequate.

4.2 Oven, complying with the requirements specified in ISO 188:2007, method A or method B, and capable of maintaining the compression assembly with the test pieces at the test temperature within the tolerance limits specified in 6.2.

NOTE Test results obtained with ovens for method A may be different may 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.

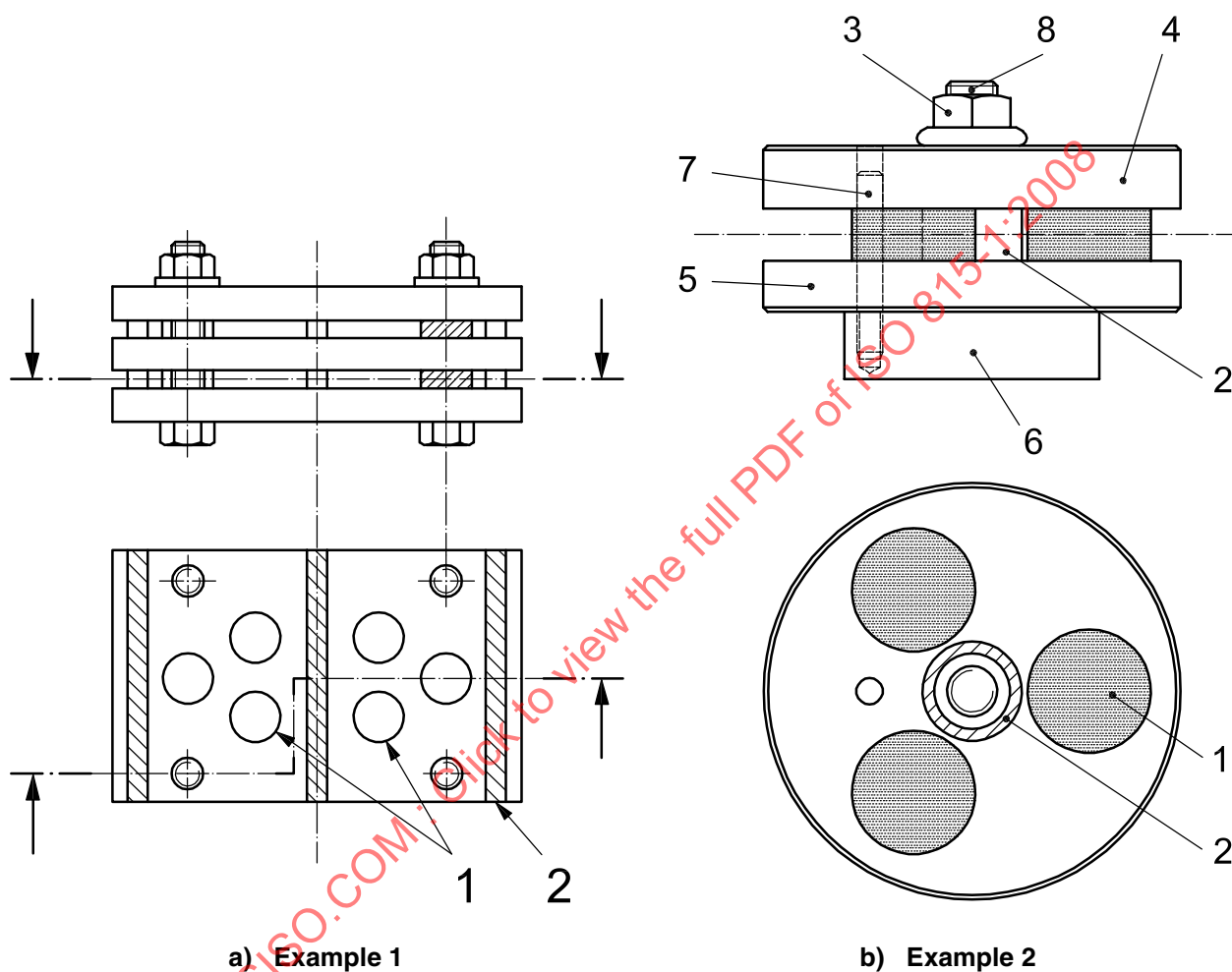
4.3 Pair of tongs, for handling the test pieces.

4.4 Thickness gauge, with an accuracy of $\pm 0,01\text{ mm}$ (see ISO 23529:2004, 7.1), having a flat circular foot $4\text{ mm} \pm 0,5\text{ mm}$ 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. For comparative tests, the same dimensions of the circular foot shall be used.

NOTE When using a digital gauge, a resolution of $0,001\text{ mm}$ 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.

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



Key

- 1 test pieces
- 2 spacer
- 3 nut
- 4 upper plate
- 5 lower plate
- 6 part formed for clamping in a vice
- 7 locating pin
- 8 screw

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

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

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 various 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 of test pieces

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

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.

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.

6 Test conditions

6.1 Duration of test

The exposure time shall be 24_{-2}^0 h, 72_{-2}^0 h, 168_{-2}^0 h or multiples of 168 h, measured from the moment of placing the compression assembly in the oven (4.2).

6.2 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 elevated-temperature tests: $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$; $55^{\circ}\text{C} \pm 1^{\circ}\text{C}$; $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$; $85^{\circ}\text{C} \pm 1^{\circ}\text{C}$; $100^{\circ}\text{C} \pm 1^{\circ}\text{C}$; $125^{\circ}\text{C} \pm 2^{\circ}\text{C}$; $150^{\circ}\text{C} \pm 2^{\circ}\text{C}$; $175^{\circ}\text{C} \pm 2^{\circ}\text{C}$; $200^{\circ}\text{C} \pm 2^{\circ}\text{C}$; $225^{\circ}\text{C} \pm 2^{\circ}\text{C}$ 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.

7 Procedure

7.1 Preparation of compression assembly

With the compression assembly (4.1) at standard laboratory temperature, carefully clean the operating surfaces. Apply a thin coating of lubricant to the faces of the compression plates (4.1.1) 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 10).

NOTE For most purposes, a silicone or fluorosilicone liquid having a nominal kinematic viscosity of $0,01 \text{ m}^2/\text{s}$ 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.

7.2 Thickness measurement

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

7.3 Applying the compression

Place the test pieces between the pairs of compression plates together with the spacer(s) (4.1.2), avoiding contact between test pieces and bolts or spacer(s). Tighten the clamping device (4.1.3) 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 4.1.2).

7.4 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 (4.2) operating at the test temperature (see 6.2).

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).

7.5 Terminating the test

7.5.1 At ambient temperature

If the test is conducted at ambient temperature, release the test pieces after the required test duration (see 6.1) and transfer them to a wooden bench. Leave them to recover for $30 \text{ min} \pm 3 \text{ min}$ at standard laboratory temperature and then measure their thickness.

7.5.2 At elevated temperature

Method A: After the required test duration (see 6.1), 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 $30 \text{ min} \pm 3 \text{ min}$, and then measure their thickness.

Method A shall be used unless otherwise specified.

Method B: After the required test duration, remove the compression 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 $30 \text{ min} \pm 3 \text{ min}$ 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 $30 \text{ min} \pm 3 \text{ min}$ and, after a further $30 \text{ min} \pm 3 \text{ min}$ 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.

7.6 Internal examination

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

8 Expression of results

8.1 The compression set, expressed as a percentage of the initial compression, is given by the formula:

$$\frac{h_0 - h_1}{h_0 - h_s} \times 100$$

where

h_0 is the initial thickness, in millimetres, of the test piece;

h_1 is the thickness, in millimetres, of the test piece after recovery;

h_s is the height, in millimetres, of the spacer.

Report the result to the nearest 1 %.

9 Precision

See Annex A.

10 Test report

The test report shall include the following information:

- a) sample details:
 - 1) a full description of the sample and its origin,
 - 2) compound and cure details, where appropriate,
 - 3) the method of preparation of test pieces from samples, for example whether moulded or cut;
- b) test method:
 - 1) a full reference to the test method used, i.e. the number of this part of ISO 815,
 - 2) the type of test piece used, i.e. A or B, and whether or not it was laminated,
 - 3) the method of cooling used after the test, i.e. A, B or C, and the exact cooling time for method B,
 - 4) the nature of the lubricant used,
 - 5) whether the test pieces were tested separately or as a set;
- c) test details:
 - 1) the standard laboratory temperature used,
 - 2) the temperature and times of conditioning and of recovery,
 - 3) the duration and temperature of test,
 - 4) the compression used,
 - 5) details of any procedures not specified in this part of ISO 815;
- d) test results:
 - 1) the number of test pieces used,
 - 2) the initial thickness of the test pieces, if required,
 - 3) the thickness of the test pieces after recovery, if required,
 - 4) the median value of the compression set and the individual test results;
- e) the date of the test.

Annex A (informative)

Precision

A.1 General

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. Consult this for precision concepts and nomenclature. Annex B gives guidance on the use of repeatability and reproducibility.

A.2 Precision details

A.2.1 An interlaboratory test programme (ITP) was organized in 1986 by the Laboratoire de Recherches et de Contrôle du Caoutchouc et des Plastiques (LRCCP). Three materials (vulcanized rubber compounds) were used: SBR, NBR and EPDM.

Test pieces were distributed to all laboratories and tested at 100 °C in accordance with this part of ISO 815.

A.2.2 Both type A and type B test pieces were used.

A.2.3 Tests were conducted for 24 h at 25 % compression on three test pieces. The median compression set value was used as the “test result”. The compression set was measured after 30 min ± 3 min recovery time at standard laboratory temperature after removal from the assembly in accordance with method A.

A.2.4 A type 1 precision was measured in the ITP. The time period for repeatability and reproducibility is on a scale of days. A total of 19 laboratories participated in the test.

A.3 Precision results

A.3.1 The precision results are given in Table A.1 for compression set at 100 °C.

A.3.2 The symbols r , (r) , R , (R) , as used in Table A.1, are defined as follows:

r = repeatability in measurement units;

(r) = repeatability in percent (relative);

R = reproducibility in measurement units;

(R) = reproducibility in percent (relative).