

BS ISO 815-1:2014



BSI Standards Publication

Rubber, vulcanized or thermoplastic — Determination of compression set

Part 1: At ambient or elevated temperatures

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National foreword

This British Standard is the UK implementation of ISO 815-1:2014. It supersedes BS ISO 815-1:2008 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/22, Physical testing of rubber.

A list of organizations represented on this committee can be obtained on request to its secretary.

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INTERNATIONAL
STANDARD

ISO
815-1

Second edition
2014-09-01

**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



Reference number
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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.

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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 815-1:2008), which has been technically revised, mainly by addition of a calibration schedule ([Annex B](#)).

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 1 — 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.

WARNING 2 — Certain procedures specified in this part of ISO 815 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 part of ISO 815 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 18899:2004, *Rubber — Guide to the calibration of test equipment*

ISO 23529:2010, *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 clamping device.

A typical assembly is 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 plates shall be

- sufficiently rigid to ensure that, with a test piece under load, no compression plate bends by more than 0,01 mm and
- of sufficient size to ensure that the whole of the test piece, when compressed between the plates, remains within the area of the plates.

NOTE A surface finish no 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.

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, method A or method B, and capable of maintaining the compression assembly and test pieces at the test temperature within the tolerance specified in [7.2](#).

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.

4.3 Pair of tongs, for handling the test pieces.

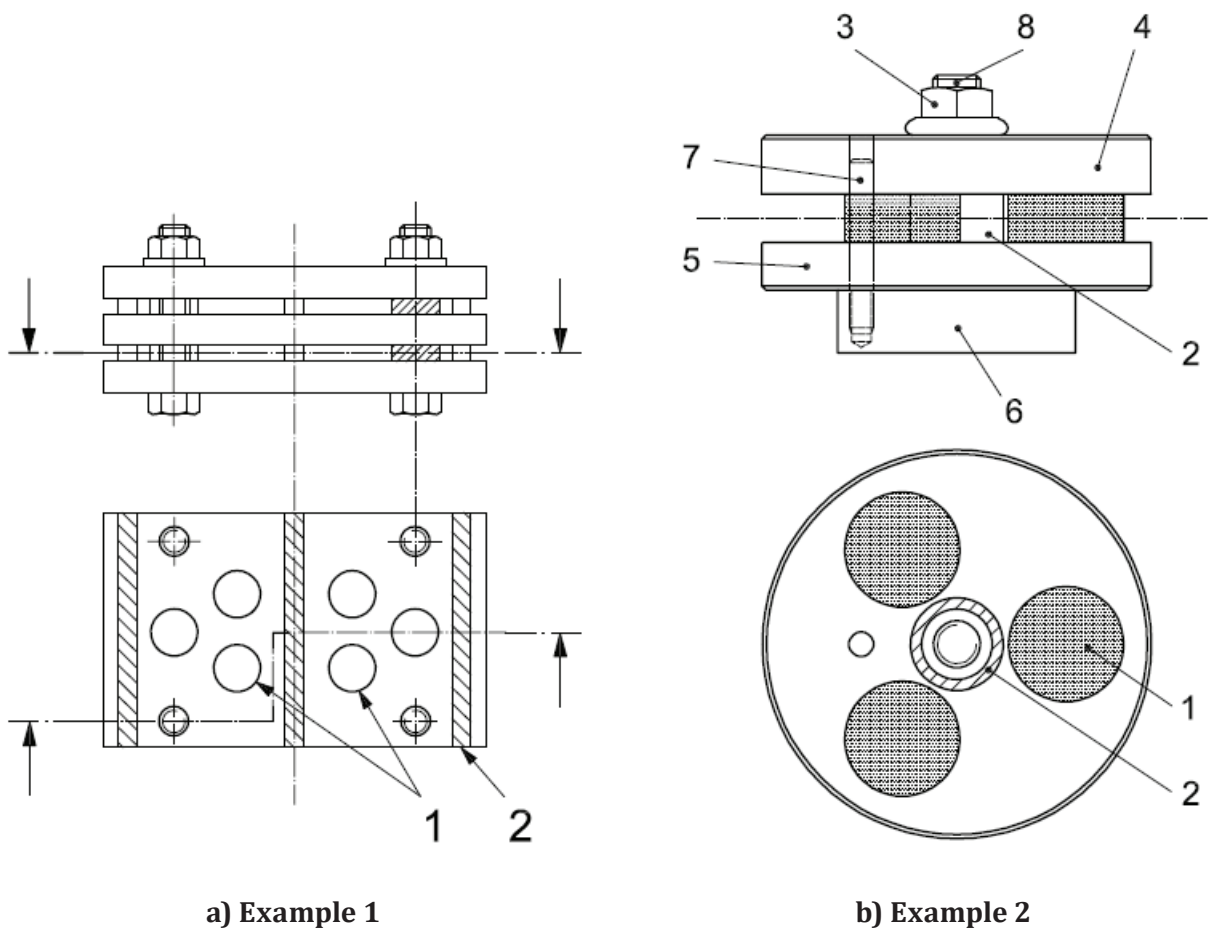
4.4 Thickness gauge, with an accuracy of $\pm 0,01$ mm (see ISO 23529:2010, 7.1), having a flat circular foot of $4,0$ mm \pm $0,5$ mm in diameter and a flat solid base-plate and exerting a pressure of 22 kPa \pm 5 kPa

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.

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.

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



Key

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

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

5 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in [Annex B](#).

6 Test pieces

6.1 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.

6.2 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 [6.1](#) 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.

6.3 Number of test pieces

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

6.4 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).

6.5 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.

7 Test conditions

7.1 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).

7.2 Temperature of test

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

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.

8 Procedure

8.1 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 $100\text{ mm}^2/\text{s}$ 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.

8.2 Thickness measurement

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

8.3 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 ± 2) % of the original thickness of the test piece except for higher hardnesses, for which the applied compression shall be (15 ± 2) % or (10 ± 1) % (see 4.1.2).

8.4 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 test temperature (see 7.2).

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).

8.5 Terminating the test

8.5.1 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.

8.5.2 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 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 $30 \text{ min} \pm 3 \text{ min}$ 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.

8.6 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.

9 Expression of results

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

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

where

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

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

h_s is the height of the spacer, in millimetres.

Report the result to the nearest 1 %.

10 Precision

See [Annex A](#).

11 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.

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 apparatus 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 for the testing 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).

Table A.1 — Type 1 precision for compression set at 100 °C

Material	Average	Within lab		Between lab	
		<i>r</i>	(<i>r</i>)	R	(<i>R</i>)
Type A test piece					
EPDM	10,3	2,7	26	4,0	38
NBR	19,8	3,3	17	4,3	21
SBR	41,1	4,7	11	13,6	33
Pooled values	23,7	3,6	15	8,6	36
Type B test piece					
EPDM	14,8	3,3	22	4,5	30
NBR	24,4	4,3	18	7,7	32
SBR	44,9	5,1	11	14,0	33
Pooled values	28,0	6,0	15	10,0	35

Annex B (normative)

Calibration schedule

B.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded on any calibration report or certificate. It shall be reported whether calibration is made in the 'as-received' condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is general fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

B.2 Schedule

Verification/calibration of the test apparatus is a normative part of this standard. The frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory using ISO 18899 for guidance.

The calibration schedule given in [Table B.1](#) has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a more specific or detailed calibration procedure than in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code letter.

The code letters used in the calibration schedule are:

- C: requirement to be confirmed but no measurement,
- N: initial verification only,
- S: standard interval as given in ISO 18899,
- U: in use.

Table B.1 — Calibration schedule

Parameter	Requirement	Procedure ISO 18899:2004	Verification frequency	Notes
Compression plates	Two parallel, flat, highly polished chromium-plated steel or stainless-steel plates	C	N	Roughness profile <i>Ra</i> not worse than 0,4 µm has been found suitable.
	Plates not to distort by more than 0,01 mm when load applied	C	N	
	Plates of sufficient size to ensure that the whole of the test piece remains within the area of the plates after compression	C	U	
Steel spacer(s)	of such size and shape that contact with the compressed test piece is avoided	C	U	
	height 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.	15.2	U	
Clamping device	Simple screw device or any other technology	C	N	
Oven	complying with the requirements specified in ISO 188 capable of maintaining the compression assembly and test pieces at the test temperature within the tolerance specified in 7.2. steady-state temperature within the specified tolerances reached in the interior of the test pieces in not more than 3 h.	See ISO 188	S	
Thickness gauge	with an accuracy of ±0,01 mm having a flat circular foot of 4 mm ± 0,5 mm in diameter and a flat solid base-plate	15.2	S	for digital gauges, a resolution of 0,001 mm is needed to obtain the required accuracy.
	exerting a pressure of 22 kPa ± 5 kPa for rubber of hardness equal to or greater than 35 IRHD or a pressure of 10 kPa ± 2 kPa if the hardness is less than 35 IRHD.	22.2	N	
Timing device	precision of ±1 s	23.1	S	

In addition to the items listed in the table, use of the following is implied, which needs calibrating in accordance with ISO 18899:

- instruments for determining dimensions of the test pieces.

Bibliography

- [1] ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*
- [2] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

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