INTERNATIONAL STANDARD

ISO 3384-1

Second edition
2019-08

Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression —

Part 1: Testing at constant temperature

Caoutchouc vulcanisé ou thermoplastique — Détermination de la relaxation de contrainte en compression —

Partie 1: Essais à température constante

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Reference number
ISO 3384-1:2019(E)

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Foreword

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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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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.

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

This second edition cancels and replaces the first edition (ISO 3384-1:2011), which has been technically revised. It also incorporates the Amendment ISO 3384-1:2011/Amd.1:2013.

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

— test procedures have been improved in detail;

— the requirement for compression device (5.1) has been harmonized with other International Standards;

— content of ISO 3384-1:2011/Amd.1:2013 has been incorporated (Annex B).

A list of all parts in the ISO 3384 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.

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Introduction

When a constant strain is applied to rubber, the force necessary to maintain that strain is not constant but decreases with time; this behaviour is called “stress relaxation”. Conversely, when rubber is subjected to a constant stress, an increase in the deformation takes place with time; this behaviour is called “creep”.

Tests in compression are normally made under continuous stress conditions (i.e. the test piece remains strained throughout the test), and are hence a measure of sealing force. Note that the terms continuous and discontinuous used in this standard refer to whether the measure of force is made continuously of at intervals.

Tests to use stress relaxation in tension as a measure of ageing are given in ISO 6914.

The processes responsible for stress relaxation can be physical or chemical in nature, and under all normal conditions both types of process will occur simultaneously. However, at normal or low temperatures and/or short times, stress relaxation is dominated by physical processes, while at high temperatures and/or long times chemical processes are dominant.

If the life-time of a material is to be investigated, it can be determined using the method described in ISO 11346.

In addition to the need to specify the temperatures and time intervals in a stress relaxation test, it is necessary to specify the initial stress and the previous mechanical history of the test piece since these can also influence the measured stress relaxation, particularly in rubbers containing fillers.

The most important factor in achieving good repeatability and reproducibility when making stress relaxation tests is to keep the temperature and compression constant during all measurements.

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Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression —

Part 1:
Testing at constant temperature

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.

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1 Scope

This document specifies two procedures for determining the decrease in counterforce exerted by a test piece of vulcanized or thermoplastic rubber which has been compressed to a constant deformation and maintained thus at a predetermined test temperature.

The counterforce can be determined either by means of a continuous-measurement system or by a discontinuous-measurement one.

Two test methods are specified, method A and method B. In method A the compression and all measurements of counterforce are made at test temperature and in method B the compression and all measurements of counterforce are made at standard laboratory temperature.

Method A and method B do not give the same results, as in method B the shrinkage of the material from the test temperature to standard laboratory temperature is included in the result.

Two forms of test piece are specified in this document: cylindrical test pieces and rings. Comparison of results is valid only when made on test pieces of similar size and shape.

The use of ring test pieces is particularly suitable for the determination of stress relaxation in liquid environments.

This document deals only with testing at constant ambient or elevated temperature. Testing at temperatures below standard laboratory temperature is not specified. The methods have been used for low-temperature testing, but their reliability under these conditions is not proven.

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 37:2017, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

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

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

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ISO 23529:2016, *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:

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

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

3.1
compression stress relaxation
reduction in compressive force, expressed as a percentage of the initial force, which occurs with time after the application of a constant compressive strain

4 Principle

A test piece of vulcanized or thermoplastic rubber is compressed to a constant deformation and maintained at a predetermined test temperature. The decrease in counterforce is then measured.

In method A, the compression is applied and all counterforce measurements are made at the test temperature.

In method B, the compression is applied and all counterforce measurements are made at a standard laboratory temperature. The test pieces are stored at the test temperature.

The test can be conducted in a gaseous or a liquid environment.

The two measurement methods, A and B, do not give the same values of stress relaxation, and comparison of values obtained from the two methods should be avoided. The method selected for use depends on the purpose of the test. Thus, for fundamental studies and in applications where sealing at elevated temperatures is a problem, method A might be preferred, and in applications where temperature cycling from normal to an elevated temperature is a problem, method B might be preferred.

NOTE Other methods can be used for specific purposes, such as applying the compression at standard laboratory temperature and making all counterforce measurements at a different temperature.

5 Apparatus

5.1 Compression device, 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.

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.

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When the apparatus is assembled without a test piece, the gap between the plates shall not vary by more than ±0,01 mm for discontinuous jigs and not more than ±0,1 mm for continuous rigs.

NOTE The parallelism is not as critical for continuous rigs as they are only compressed once.

For ring test pieces, the plates shall have holes of at least 2 mm diameter drilled through their centre portions to allow equalization of pressure and circulation of fluid inside the ring-shaped test piece.

It shall be possible to connect the compression device to suitable equipment for compressing the test piece to the specified compression at the specified speed and for measuring the counterforce exerted by the compressed test piece with an accuracy of 1 % of the measured value.

The device shall be capable of setting the compression and maintaining it during the whole duration of the test, and it shall be possible to keep the device in an oven at the specified test temperature. Care shall be taken to ensure that there is no loss of heat from the test piece, for example by conduction through metal parts which are connected with the outside of the oven.

5.2 Counterforce-measuring device, capable of measuring compression forces in the desired range with an accuracy of 1 % of the measured value.

Continuous procedure

The device may be a continuous-measurement system which monitors the test piece during the whole duration of the test, making it possible to measure the change in the counterforce with time on a continuous basis. The deformation of the test piece shall be kept within ±0,01 mm for the duration of the test. If it is not possible to keep the deformation constant within this tolerance due to the spring effect in load cells, a correction may be done mechanically or mathematically.

This procedure is mostly used for testing according to method A, but can be used for method B using a temperature cycling oven.

Discontinuous procedure

Alternatively, a compression-testing machine may be used to measure the counterforce at prescribed time intervals. In this case, the force necessary to cause a slight increase in the compression of the test piece is measured. This additional compression shall be as small as possible and in no case greater than a force of 1 N for balance-type machines, or greater than 0,05 mm for stress/strain-type machines, applied in either case without overshoot. The whole of the force exerted by the test piece as a result of the extra compression shall act on the force-measuring device. It shall also be possible to repeat the compression to within ±0,01 mm from one measurement to another.

This procedure is mostly used for testing according to method B, but can be used for method A using an oven during the measurements.

5.3 Test environment

5.3.1 For tests in gaseous media, an air oven in accordance with the requirements of ISO 188 shall be used. An oven meeting the requirements specified for one of the ovens used in ISO 188:2011, method A, is recommended.

If the testing is done in nitrogen, oxidative ageing will be eliminated and the result will be due to thermal ageing only. This can be used to simulate conditions where the product is not exposed to air, such as seals used in oil or steam.

5.3.2 For tests in liquids, the compression device shall be totally immersed in a liquid in a bath, or a closed vessel for volatile or toxic fluids, such that free circulation of the liquid can take place through the holes in the compression plates. The liquid shall be maintained at the specified temperature by proper control of a heater and circulation of the liquid in the bath or, alternatively, by placing the liquid bath and compression device within an air oven as specified above.

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5.4 Temperature-measuring equipment, with a sensing element of appropriate precision. The temperature-sensing element shall be fitted in such a way that it accurately measures the temperature of the test piece.

NOTE A Pt100 sensor has been found to be suitable for temperature measurement.

6 Calibration

The requirements for calibration of the test apparatus are given in Annex B.

7 Test piece

7.1 Type and preparation of test pieces

7.1.1 General

Test pieces shall be prepared either by moulding or by cutting from moulded sheets or products, in accordance with ISO 23529.

NOTE The results obtained from test pieces of different sizes are not comparable.

7.1.2 Cylindrical test pieces

The test piece shall be a cylindrical disc of diameter 13,0 mm ± 0,5 mm and thickness 6,3 mm ± 0,3 mm.

7.1.3 Ring test pieces

The preferred ring test piece is a ring of square cross-section cut from a flat sheet of the test material by means of rotary cutters. For a suitable machine for the preparation of small ring test pieces, see Annex A of ISO 37:2017.

The dimensions of test pieces shall be:

— thickness: 2,0 mm ± 0,2 mm;

— inner diameter: 15,0 mm ± 0,2 mm;

— radial width: 2,0 mm ± 0,2 mm.

The sheets may be prepared by moulding or from finished articles by cutting and buffing.

Alternatively, an O-ring, size code ISO 3601-1-14 × 2,65-G-N, as specified in ISO 3601-1:2012 (internal diameter 14 mm and diameter of the cross-section 2,65 mm), may be used as the standard test piece.

O-rings of other dimensions, together with seals or gaskets of other configurations, may be used as non-standard test pieces where appropriate.

NOTE Some test machines have jigs in which the test piece is compressed by screwing a compression plate down on to stops. This gives a fixed strained thickness. Test pieces within the tolerances given above will not necessarily have the required compression strain when tested in such jigs. It is important that a compression strain within the limits given in 9.3.4 and 9.4.3 be achieved by careful matching of jig and test piece.

7.2 Measurement of dimensions of test pieces

The dimensions of test pieces shall be measured as specified in ISO 23529.

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7.3 Number of test pieces

The preferred number of test pieces is three, but for routine and screening tests two test pieces are acceptable.

7.4 Time interval between forming and testing

The interval between forming and testing shall be in accordance with ISO 23529.

7.5 Conditioning of test pieces

7.5.1 Prior to testing, the test pieces shall undergo first a thermal and then a mechanical conditioning as detailed in 7.5.2 and 7.5.3.

7.5.2 Thermal conditioning shall be carried out by heating the test pieces at 70 °C for 3 h. Following thermal conditioning, the test pieces shall be allowed to stand for a period of not less than 16 h and not more than 48 h at standard laboratory temperature prior to mechanical conditioning or testing.

NOTE Some test samples, especially of thermoplastic elastomers, might contain moulding stresses, and thermal conditioning to relieve these stresses might improve the reproducibility of the results.

7.5.3 Mechanical conditioning shall be carried out at one of the standard laboratory temperatures specified in ISO 23529, as follows.

Compress the test pieces to the same compression that will be used during the rest of the test and then immediately return them to zero stress; repeat this procedure to give a total of five cycles of deformation and immediate return.

Following mechanical conditioning, the test pieces shall be allowed to stand for a period of not less than 16 h and not more than 48 h at standard laboratory temperature prior to testing.

Mechanical conditioning has been found to improve test reproducibility, particularly for compounds containing substantial proportions of filler, but is not always appropriate for finished products and can, therefore, lead to results that are not typical of service. Such conditioning may be omitted provided thermal conditioning is still undertaken. This omission shall be mentioned in the test report.

8 Duration, temperature and test liquid

8.1 Duration of test

Unless otherwise specified, the duration of test shall be at least one week (168 h ±2 h).

If intermediate times are used, 3 h $^{0}_{-10}$ min, 6 h $^{0}_{-20}$ min, $\left(24\,^{0}_{-0,5}\right)$ h and $\left(72\,^{0}_{-1}\right)$ h are preferred. The test period begins after the initial compression. If longer test times are used, a logarithmic time-scale can be employed.

In method B, when compression is carried out at standard laboratory temperature, a conditioning period of 2 h (not included in the time of test) shall be allowed each time the test piece is conditioned for measurement at that temperature.

A much longer cooling period might be needed when testing in liquids and this time shall not be included in the test time.

NOTE To assist with the cooling, a fan can be used.

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8.2 Temperature of exposure

The temperature of exposure shall be chosen from the list of standard temperatures in ISO 23529. Temperatures of exposure which cause rapid degradation or evaporation of the test liquid shall be avoided. The temperature shall be kept as constant as possible during the test in accordance with ISO 23529.

8.3 Immersion liquids

The test liquid shall be chosen according to the particular application, but should preferably be one of those listed in ISO 1817.

Some liquids which are ageing faster than the rubber might need to be replaced during testing. The time for changing the liquid should not be included in the test time and the change should be mentioned in the test report.

9 Procedure

9.1 Preparation

Carefully clean the operating surfaces of the compression device. When testing in a gaseous medium, apply a thin coating of a lubricant having substantially no action on the rubber.

NOTE A silicone or fluorosilicone fluid (having a kinematic viscosity of about 0,01 m^2/s) and molybdenum disulfide have been found to be suitable lubricants.

9.2 Thickness measurement

9.2.1 Cylindrical test pieces

Measure the thickness of each test piece at the central portion with an accuracy of 0,01 mm, after thermal conditioning and before mechanical conditioning, at the chosen standard laboratory temperature, as specified in ISO 23529:2016, method A.

Use this measurement to calculate the necessary compression.

9.2.2 Ring test pieces

Measure the axial thickness of each test piece with an accuracy of 0,01 mm at four points approximately 90° apart around the ring after thermal conditioning and before mechanical conditioning, at the chosen standard laboratory temperature, as specified in ISO 23529. Use the average of the measurements to calculate the necessary compression. Individual measurements, on a single test piece, shall not differ by more than 0,05 mm. If they do, discard the test piece.

9.3 Method A

9.3.1 Bring the compression device and the test environment to the test temperature.

9.3.2 When testing in a liquid, the test piece and the operating surfaces of the compression device shall be gently lubricated with the test liquid. When testing in a gaseous medium, a thin coating of a lubricant having substantially no action on the rubber shall be applied (see 9.1).

9.3.3 Place the test piece in the preheated compression device (5.1) and wait until the test temperature is reached.

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9.3.4 Compress the test piece by (25 ± 2) % in the compression device at the test temperature or, if a compression of 25 % cannot be obtained, use a compression of (15 ± 2) % or lower, in steps of 5 %. Compress the test piece in a time between 30 s and 120 s. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which is used for measurement of the counterforce as mentioned in the discontinuous procedure in 5.2).

9.3.5 Measure the initial counterforce F_0 with an accuracy of 1 % of the measured value, at the test temperature, 30 min ± 1 min after completing the compression.

9.3.6 Repeat the measurement of the counterforce F_t after the times specified in 8.1, or continuously. Take all measurements at the test temperature.

After the last measurement at the test temperature, the test piece may be allowed to cool down to standard laboratory temperature and a further measurement of the counterforce made.

NOTE Valuable additional information can be obtained after the relaxation test has been finished. Research has shown that the amount of recovery (at the test temperature) is a measure of the permanent chemical reactions occurring alongside physical relaxation (see Reference [6]).

9.4 Method B

9.4.1 Bring the test environment to the test temperature.

9.4.2 When testing in a liquid, the test piece and the operating surfaces of the compression device shall be gently lubricated with the test liquid. When testing in a gaseous medium, a thin coating of a lubricant having substantially no action on the rubber shall be applied (see 9.1).

9.4.3 Compress the test piece by (25 ± 2) % in the compression device at a standard laboratory temperature or, if a compression of 25 % cannot be obtained, use a compression of (15 ± 2) % or lower, in steps of 5 %. Compress the test piece in a time between 30 s and 120 s. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which is used for measurement of the counterforce as mentioned in the discontinuous procedure in 5.2).

9.4.4 Measure the initial counterforce F_0 with an accuracy of 1 % of the measured value, at standard laboratory temperature, 30 min ± 1 min after completing the compression.

9.4.5 Immediately after measuring the counterforce, store the compressed test piece in the test environment (see 5.3) at the specified test temperature.

9.4.6 When making measurements of the counterforce F_t after each of the times specified (see 8.1), remove the apparatus from the test environment, maintain it at the standard laboratory temperature for 2 h, determine the counterforce and then return it to the test environment until next specified time. It is important that the apparatus and test piece reach thermal equilibrium within 2 h, and forced cooling can be necessary. The temperature shall be checked with the temperature sensor specified in 5.4.

As an alternative to taking out and returning the test jig, the temperature change can be done by using a programmable oven with heating and cooling.

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.

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The compression stress relaxation $R(t)$, after a specified duration of test t, expressed as a percentage of the initial counterforce, is given by Formula (1):

$$R(t)=\frac{F_0-F_t}{F_0}\times 100 \quad (1)$$

where

F_0 is the initial counterforce, measured after 30 min;

F_t is the counterforce measured after the specified duration of test t.

The median value of the results for the test pieces shall be taken. The individual values for the test pieces shall agree to within 10 % of the median value. If they do not, the test shall be repeated.

Stress relaxation values measured after different times of exposure shall be plotted as a function of time on a logarithmic or linear scale to facilitate the interpretation of the test data. For some applications, it is more useful to calculate compression stress ratio values, i.e. F_t/ F_0, after different times of exposure, rather than stress relaxation values. In this case, compression stress ratio values shall be presented graphically as a function of logarithmic or linear time.

11 Precision

For precision data, see Annex A.

12 Test report

The test report shall include the following information:

a) sample details:

   1) a full description of the sample and its origin;

   2) the method of preparation of the test pieces from the sample, e.g. whether moulded or cut;

   3) the compound details and cure conditions, where appropriate;

b) test method:

   1) a full reference to the test method used, i.e. the number of this document and the method utilized;

   2) the test procedure used, i.e. continuous or discontinuous;

   3) the type of test piece used;

c) test details:

   1) the number of test pieces tested;

   2) any special information concerning the apparatus, e.g. the method used for measuring the counterforce;

   3) the standard laboratory temperature used;

   4) the duration and temperature of conditioning of the test pieces prior to testing;

   5) the test duration and the temperature used;

   6) the compression used: 25 % or other (give details);

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7) the type of oven used;

8) the lubricant used;

9) any deviation, by agreement or otherwise, from the specified test procedure;

d) the individual test results and the median value as $R(t)$;

e) the dates of the test, when it was started and finished.

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Annex A
(informative)

Precision

A.1 General

An interlaboratory test programme (ITP) and the precision calculations to express the repeatability and reproducibility were performed in accordance with ISO/TR 9272[1)].

A.2 Precision details

A.2.1 The ITP was conducted in 1998. One material, an IR/SBR blend rubber compound, was used. Testing using method A was conducted at 23 °C and 100 °C and using method B at 100 °C. The test result was taken as the average value, for two test pieces, of the percent decrease in the initial counterforce after 168 h of relaxation. Twelve laboratories participated in the 23 °C testing using method A, eleven laboratories in the 100 °C testing using method A and seven laboratories in the 100 °C testing using method B.

A.2.2 The precision determined is a type 1 precision: fully prepared test pieces were submitted to the laboratories. The precision is also an intermediate-term precision with a span of 2 or 3 weeks between the two replications. This is required due to the relaxation-ageing period of 168 h for each replication of the test. This is in distinction to the more usual day 1/day 2 replication with a few days between replications.

A.2.3 Analysis of the data from all the laboratories (all three tests) resulted in:

— the results from three laboratories being declared outliers for method A at 23 °C;

— the results from two laboratories being declared outliers for method A at 100 °C;

— the result from one laboratory being declared an outlier for method B at 100 °C.

These results were rejected and the final analysis was conducted on the remaining data, i.e.:

— for method A at 23 °C: the results from nine laboratories;

— for method A at 100 °C: the results from nine laboratories;

— for method B at 100 °C: the results from six laboratories.

The revised database represents those laboratories that had good within-laboratory control of the testing (the results are in relatively good agreement).

A.3 Precision results

The precision data obtained from the final database are given in Table A.1. The precision (both repeatability and reproducibility) of method B at 100 °C is substantially worse than that for method A. No relative precision, (r) and (R), is given for this document.

1) Withdrawn.

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Table A.1 — Precision results

Method A, 168 h at 23 °C					
Material	Mean % relaxation	s_r	r	s_R	R
A	10,9	0,795	2,22	1,21	3,40
Method A, 168 h at 100 °C					
Material	Mean % relaxation	s_r	r	s_R	R
A	50,5	0,845	2,37	2,15	6,03
Method B, 168 h at 100 °C					
Material	Mean % relaxation	s_r	r	s_R	R
A	67,5	2,07	5,8	8,66	24,3

s_r is the repeatability standard deviation, in measurement units;

r is the repeatability, in measurement units (i.e. % relaxation);

s_R is the reproducibility standard deviation, in measured units;

R is the reproducibility, in measurement units (i.e. % relaxation).

A.4 Guidance for using precision results

For the general procedure for using precision results, see ISO 19983.

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