International Standard

ISO 3384-1

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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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 document 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 third edition cancels and replaces the second edition (ISO 3384-1:2019), which has been technically revised.

The main changes are as follows:

— addition of new ITP results in Annex A.

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.

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 lifetime 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.

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.

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

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

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 terminology databases for use in standardization at the following addresses:

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

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

3.1
compression stress relaxation
R(*t*)
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, 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 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 a *Ra* can be obtained by a grinding or polishing operation.

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.

5.2.1 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.

5.2.2 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 188:2023, 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.

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 ISO 37:2017, Annex A.

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.

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.