INTERNATIONAL STANDARD

ISO 3384

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Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression at ambient and at elevated temperatures

Caoutchouc vulcanisé ou thermoplastique — Détermination de la relaxation de contrainte en compression à température ambiante et aux températures élevées

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 3384 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 3384:1999), of which it constitutes a minor revision designed to update the normative references. It also incorporates the Amendment ISO 3384:1999/Amd. 1:2001, *Precision data*.

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”.

The processes responsible for stress relaxation may 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 whilst 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 air oven ageing test described in ISO 11346[1)].

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 may 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.

1) ISO 11346, *Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use*.

Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression at ambient and at elevated temperatures

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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.

1 Scope

This International Standard specifies two methods 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.

Two forms of test piece are permitted: cylindrical test pieces and rings. Different shapes and sizes of test piece give different results, and comparison of results should be limited to 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.

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 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 37:2005, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

ISO 188:1998, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

ISO 1817, *Rubber, vulcanized — Determination of the effect of liquids*

ISO 3601-1:2002, *Fluid power systems — O-rings — Part 1: Inside diameters, cross-sections, tolerances and size identification code*

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 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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 standard laboratory temperature. The test pieces are stored at the test temperature.

NOTE 1 The two methods, A and B, of carrying out the measurement 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 may be preferred, and in applications where temperature cycling from normal to an elevated temperature is a problem, method B may be preferred.

NOTE 2 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 from chromium-plated or stainless steel or another corrosion-resistant material, between the faces of which the test pieces are compressed. Flatness, surface roughness, parallelism and rigidity of the plates are all important.

When the apparatus is disassembled, the compression plates shall be flat to within 0,01 mm. The finish of the surface shall not be worse than Ra 0,4 µm (see ISO 4287). When the apparatus is assembled without a test piece, the gap between the plates shall not vary by more than ± 0,01 mm.

When the test assembly is subjected to the test load with a test piece between the plates, neither compression plate shall bend by more than 0,01 mm.

The plates shall be of sufficient size to ensure that the whole of the compressed test piece is within the area of the plates and can expand freely laterally.

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. The preferred device is one that monitors the test piece during the whole duration of the test, in which case continuous measurement of the change in counterforce with time is possible. The deformation of the test piece shall be kept within ± 0,01 mm for the duration of the test.

Alternatively, a compression-testing machine can 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.

5.3 Test chamber, complying with the requirements of ISO 188:1998, method A.

For tests in air, a well designed, uniformly heated air oven shall be used, provided with adequate temperature control to maintain the specified air temperatures within the tolerance specified in 7.2. For tests in liquids, the compression device shall be totally immersed in the 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, for example a PT100 element, class A or better. The temperature-sensing element shall be mounted so that it is located not more than 2 mm from a surface of the test piece, in one of the compression plates.

6 Test piece

6.1 Type and preparation of test piece

6.1.1 General

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

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

6.1.2 Cylindrical test pieces

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

6.1.3 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 37:2005.

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