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## Rubber, vulcanized or thermoplastic — Determination of ageing characteristics by measurement of stress relaxation in tension

*Caoutchouc vulcanisé ou thermoplastique — Détermination des caractéristiques de vieillissement par mesure de la contrainte de relaxation en traction*

ICS: 83.060

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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](http://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](http://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 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](http://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 5th edition cancels and replaces the 4th edition (ISO 6914:2013), which has been technically revised to increase alignment with ISO 3384-1.

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](http://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 with time takes place; this behaviour is called “creep”.

The process 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.

Stress relaxation tests in compression are suitable for obtaining a measure of the functional property of sealing force and procedures are given in ISO 3384. Stress relaxation tests in tension can be used as a measure of ageing as chemical processes will dominate in the case of a thin test piece exposed to an atmosphere containing oxygen at an elevated temperature for relatively long periods of time.

There are two variants of the technique. Measurements of stress can be made under either

- a) continuous strain conditions, or
- b) intermittent strain conditions.

In the case of a), continuous strain conditions, the test piece is held in extension throughout the ageing period in the oven. In the case of b), intermittent strain conditions, the test piece is aged in the oven in the unstressed state but, at periodic intervals, it is stretched to a fixed extended length for a short time in order to determine the stress.

NOTE 1 The terms “continuous stress relaxation” and “intermittent stress relaxation” are commonly used to describe the two principal variants of the technique. The latter term, “intermittent stress relaxation”, is a misnomer since no true relaxation of stress occurs and indeed the measured stress can increase with time. For this reason, the use of this term has been avoided in this document although it is fairly well established in the literature.

In a second version of the intermittent test, the test piece is periodically removed from the accelerated ageing atmosphere and the stress is measured under normal laboratory conditions. The advantage of this method is that it does not require the use of special apparatus since a conventional tensile-testing machine can be used for the measurement of stress.

Measurements made in accordance with the methods described in this document provide information about the structural changes that occur in the rubber during ageing, and are complementary: under continuous strain conditions, provided physical relaxation processes are not dominant, the decay of stress provides a measure of the degradative scission reactions in the network. Any new networks formed as a result of crosslinking reactions are considered to be in equilibrium at the test strain with the main network and, therefore, do not impose any new stresses. Under intermittent strain conditions, the decay of stress provides a measure of the net effect of both degradative scission and crosslinking reactions.

NOTE 2 Even under conditions conducive to chemical processes, some physical relaxation can occur. The extent to which it does so will depend on the viscoelastic characteristics of the rubber and on the test conditions and care should be exercised in the interpretation of the results. Physical relaxation is increased by fillers and will be more evident at short times and at lower temperatures. It is often found to be proportional to logarithmic time and is less temperature sensitive than chemical relaxation.

The validity of the methods described in this document depends on the uniformity of degradation in the rubber. For this reason, the thickness of the test pieces used is 1,0 mm to minimize the effect of oxygen diffusion on ageing.

The change in stress might be of direct interest, but the relative resistance of rubbers to ageing will depend on the properties being measured or required by the application. This document should, therefore, be regarded as complementary to ISO 188.

The lifetime of the material, if this is to be investigated, can be determined using the procedures described in ISO 11346.

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

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# Rubber, vulcanized or thermoplastic — Determination of ageing characteristics by measurement of stress relaxation in tension

**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 International Standard 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 describes three methods for the measurement of the change of stress in a test piece at a given elongation for the purpose of determining the ageing characteristics of a rubber.

- Method A is intended for measurement under continuous strain conditions.
- Method B is the preferred method for measurement under intermittent strain conditions.
- Method C is an alternative to method B for measurement under intermittent strain conditions in which the test piece is removed from the ageing environment for measurement of the stress at standard laboratory temperature.

Measurements at a single elevated ageing temperature can be used for quality control purposes as a measure of heat-ageing resistance. Measurements at a number of temperatures can be used for research and development purposes to estimate long-term ageing characteristics in accordance with the procedures described in ISO 11346.

Tests under continuous and intermittent strain conditions measure the results of different processes, and hence do not give the same results. The results of the intermittent methods B and C also cannot be expected to be the same as they use different test conditions.

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

ISO 5893:2019, *Rubber and plastics test equipment — Tensile, flexural and compression types (constant rate of traverse) — Specification*

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

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

## 3 Terms and definitions

No terms and definitions are listed in this document.

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/>

### 4 Principle

A test piece of vulcanized or thermoplastic rubber is elongated to a predetermined tensile strain and the force measured as a function of time in combination with exposure of the test piece to an elevated temperature.

In method A the extension is applied and the force measured over time at the test temperature.

In method B the extension is applied, the force measured and the strain relaxed at the test temperature. This process is repeated at intervals.

In method C the extension is applied, the force measured and the strain relaxed at standard laboratory temperature at intervals between which the test piece is exposed to the test temperature in the relaxed condition.

### 5 Apparatus

**5.1 Stress relaxometer**, (for method A or B) consisting of two grips which hold the test piece without slipping at a fixed extended length (to within  $\pm 1$  %) together with a means of measuring and recording the force on the test piece.

The grips shall be arranged such that the test piece can be positioned in an oven. The force-measuring system can be, for example, a calibrated spring or electronic load cell, but it shall be accurate and stable to within  $\pm 1$  % of the force reading throughout the duration of the test.

For method B, the stress relaxometer shall, in addition, be equipped with a device such that the test piece can be extended and relaxed at intervals. Repeated extension of the test piece shall be constant to within  $\pm 1$  % of the elongation.

A tensile machine equipped with a temperature-controlled cabinet can be used as a relaxometer although this is inconvenient for long times and necessitates manual intermittent application of the extension.

**5.2 Tensile-testing machine**, (for method C) using a constant rate of traverse, operating at 50 mm/min and complying with the requirements specified in ISO 5893:2019, force class 1 (measuring force to within  $\pm 1$  % of the measured value).

The machine shall be capable of cycling between fixed strain limits which are accurate to within  $\pm 1$  % of the maximum strain. The grips of the tensile-testing machine shall hold the test piece without slippage.

**5.3 Oven**, complying with the requirements specified for ISO 188:2011, method A (low air speed) or method B (high air speed), for ageing the test piece.

### 6 Calibration

The requirements for calibration of the test apparatus are given in [Annex A](#).



## 7 Test pieces

### 7.1 Type and preparation of test pieces

Test pieces shall be parallel-sided strips cut from a sheet. For the tests described in this International Standard, it is vital to ensure uniform degradation in the rubber. For this reason, the thickness of standard test pieces shall be  $(1,0 \pm 0,05)$  mm in order to minimize the effect of oxygen diffusion on ageing.

Samples of uniform thickness of less than 1,0 mm or more than 1,0 mm can be used, but these can give different results.

NOTE For temperatures above 125 °C, reducing the test piece thickness to 0,5 mm will be advantageous because of the increased effect of oxygen diffusion at higher temperatures.

Alternatively, product parts or complete products can be used as test pieces taking into account the requirement for thickness.

The other dimensions of the test pieces, i.e. width and length, shall be chosen to suit the sensitivity of the load-measuring device and the precision of the mechanism used for adjusting the strain, in order that the requirements of 5.1 and 5.2 relating to the accuracy of the force and the strain are satisfied.

### 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 for each test temperature, but for routine and screening tests, one or two test pieces are acceptable.

## 8 Storage and conditioning

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

Material and test pieces shall be protected from light as much as possible during the interval between vulcanization and testing. They shall not be allowed to come into contact with test sheets and test pieces of a different composition. This is necessary in order to prevent additives which can affect ageing, such as antioxidants, from migrating from one vulcanizate into other vulcanizates.

Test pieces shall be conditioned for a minimum of 3 h at one of the standard laboratory temperatures specified in ISO 23529 immediately before testing.

## 9 Test conditions

### 9.1 Duration of test

The duration of test should preferably be chosen from the following series:

1 h, 2 h, 4 h, 8 h, 24 h, 72 h, 168 h, and multiples of 7 d.

For methods A and B, the test period shall be considered to commence when the initial force measurement is made. For method C, the test period shall be considered to be the time in the oven, excluding the time for cooling and the measurement of force.

Alternatively, the test can be stopped when the stress indicator, expressed as the ratio of the force,  $F_t$ , at time  $t$  to the initial force,  $F_0$  (see [Clause 11](#)), reaches a predetermined value (e.g. 0,5).