
**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 mesurage de la contrainte de
relaxation en traction*





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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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 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 fifth edition cancels and replaces the fourth edition (ISO 6914:2013), which has been technically revised to further align 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.

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 (all parts). 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 can 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.

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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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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 can 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.