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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 mesurage de la contrainte de relaxation en traction

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Contents

Page

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).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 6914:2008), which has been aligned with ISO 23529 and completed with details regarding dimensions, test equipment and methods.

Introduction

The stress in a rubber test piece at a given elongation changes with time as a result of a combination of simultaneous physical and chemical processes. Chemical processes predominate in the case of thin test pieces exposed to an atmosphere containing oxygen at an elevated temperature for relatively long periods of time. Thus, the ageing characteristics of the rubber can be determined by measurement of the change of stress in a thin test piece deformed in tension after periods of exposure under such conditions.

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. Hence, this latter method is a measure of the change in modulus as a function of time.

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 International Standard 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 International Standard provide information about the structural changes that occur in the rubber during ageing.

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.

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.

Under intermittent strain conditions, the decay of stress provides a measure of the net effect of both degradative scission and crosslinking reactions.

The validity of the methods described in this International Standard 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 International Standard should therefore be regarded as complementary to ISO 188.

In addition, a distinction should be made between this test and the stress relaxation in compression tests as specified in ISO 3384-1, which is primarily intended for the testing of rubbers in applications, for example as seals, where resistance to stress relaxation is a functional property.

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

CAUTION — 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 International Standard 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.

In the case of both methods A and B, a stress relaxometer is used to record the stress at the temperature of ageing.

— 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.

The necessary calibration schedule for this type of measurement is given in Annex A.

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.

No agreement between the three methods should be inferred. The method used will depend on the purpose of the test.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 5893:2002, *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*

