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**Rubber, vulcanized or  
thermoplastic — Determination  
of stress in tension under non-  
isothermal conditions**

*Caoutchouc vulcanisé ou thermoplastique — Détermination de la  
contrainte en traction dans des conditions non isothermes*

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Published in Switzerland

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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 third edition cancels and replaces the second edition (ISO 12493:2017), which has been technically revised.

The main changes are as follows:

- another method (method B) for measuring tensile stress under non-isothermal conditions has been added. In this method, the change of stress is measured at a given strain and under variation of temperature at a given heating rate as a function of temperature.

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

Usually, stress relaxation tests (see ISO 6914) are performed at constant temperature conditions because temperature has a strong impact on the relaxation time constants of rubber materials. Depending on the respective relaxation time constant or, more realistic, relaxation time spectrum, those measurements are more or less time consuming and can require testing times of several days, weeks or even months. Accelerated stress relaxation tests can be performed under non-isothermal conditions, if the temperature is increased at a certain constant heating rate, because relaxation processes are thermally activated and therefore occur faster at higher temperatures. Thus, the entire stress relaxation behaviour of the material can be scanned within a short period of time, typically a few hours. This method is designated as the temperature scanning stress relaxation (TSSR) test method and offers the ability to characterize rubber – vulcanized or thermoplastic – by short-term measurements. TSSR tests cannot replace conventional isothermal stress relaxation measurements, but are considered as a comparative test method, e.g. for the purpose of material pre-selection or in order to determine the state of crosslink density – which is an important reason for ageing phenomena – of a sample.

During non-isothermal testing, the material undergoes not only stress relaxation, but additional phenomena occur which need to be considered by adequate corrections. Most important in this case is an increase of retractive forces due to the Gough-Joule effect and thermal expansion of the sample. Whereas the latter can be numerically compensated by considering a proper value of the coefficient of thermal expansion (CTE), the increase of retractive forces offer the opportunity to calculate the crosslink density of the material, based on fundamental theory of rubber elasticity. Furthermore, an enhanced sensitivity of the test, with respect to specific relaxation processes is achieved by determination of the first derivative of the measured stress-temperature curve. Similar to stress relaxation measurements in the time domain, the latter can be used to calculate a corresponding relaxation temperature spectrum, instead of a relaxation time spectrum.

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# Rubber, vulcanized or thermoplastic — Determination of stress in tension under non-isothermal conditions

**WARNING 1** — Users of 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 users to establish appropriate safety and health practices and 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 two methods for measuring stress in tension under non-isothermal conditions.

- Method A: The thermal stress is measured for various pre-strain and temperature conditions as a function of time.
- Method B: The change of stress is measured in a test piece at a given strain and under variation of temperature at a given heating rate as a function of temperature. In this way, the determination of the thermal-mechanical behaviour of a rubber can be accelerated, e.g. for the purpose of comparative testing of aging or estimating the upper limit of the operating temperature.

The measurement device, which is equipped with a suitable heating chamber, is used to record the stress as a function of time or temperature until the sample breaks or the stress has approached zero or for a certain time.

## 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, *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

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  
thermal stress**

$\sigma_T$   
force per initial unit area that is developed in the test piece upon heating

Note 1 to entry: It is expressed in N/m<sup>2</sup> or Pa.

**3.2  
maximum thermal stress**

$\sigma_{T,max}$   
peak value of the thermal stress recorded during the test

**3.3  
thermal stress after a specified time**

$\sigma_{T,t}$   
stress induced in the test piece upon heating for a specified time  $t$

**3.4  
pre-strain**

strain to which the test piece is subjected at the beginning of the test

Note 1 to entry: It is expressed as:

$$\frac{l_f - l_i}{l_i} * 100$$

where

$l_i$  is the initial length;

$l_f$  is the length after strain.

**3.5  
pre-stress**

force per initial unit area that results from the pre-strain

Note 1 to entry: It is expressed in N/m<sup>2</sup> or Pa.

**4 Principle**

A test piece is held at a constant pre-strain in a tensile mode at a constant temperature. When the pre-stress resulting from the given pre-strain has reached an apparent equilibrium value, the temperature of the test piece is increased. In method A, the test temperature is reached as quickly as possible, in method B, heating is carried out at a constant rate. The thermal stress developed at the elevated temperature is measured.

**5 Apparatus**

**5.1 Test machine**

**5.1.1 General**

The setup of the measuring devices can be done in different ways. Two possibilities are described below.

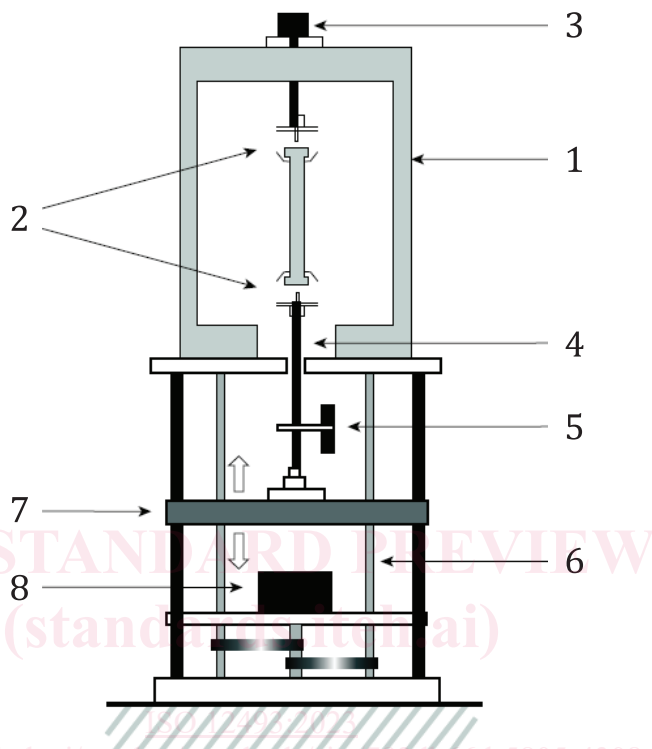
**5.1.2 Thermal-stress testing machine**

An example of a test machine for measuring the thermal stress developed in rubbery materials when heated is shown in [Figure 1](#). Two clamps hold the test piece in a temperature-controlled chamber, with



the upper clamp connected to a load cell and the bottom clamp connected to a crosshead. The crosshead is moved using a screw driven by a motor to impose a pre-strain on the test piece. The thermal stress developed when the temperature is raised is transmitted to the load cell and the output is recorded to give the variation in stress as a function of time.

The test machine shall be in accordance with ISO 5893 with force measurement to class 1 and the machine shall be capable of setting the pre-strain to within  $\pm 0,1$  at a speed of  $(20 \pm 2,5)$  mm/min.



#### Key

- 1 temperature-controlled chamber
- 2 clamps
- 3 load cell
- 4 rod
- 5 linear variable differential transformer
- 6 screw
- 7 crosshead
- 8 motor

**Figure 1 — Example of thermal-stress testing machine**

The temperature-controlled chamber shall be capable of raising the temperature at a rate of at least 30 K/min and maintaining the test piece at the required temperature as specified in ISO 23529. A suitable volume for the chamber is 3 l to 5 l. A temperature-sensing device shall be located within the chamber near the test piece.

Load cell and crosshead shall be in accordance with ISO 18899:2013.

#### 5.1.3 Stress relaxometer

Another example of a test machine consists of an electrically driven linear actuator equipped with 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 may 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.

The force measuring system and strain measuring system shall be in accordance with the requirements specified for ISO 18899:2013.

## 5.2 Oven

The oven shall be in accordance with ISO 188:2011, cell-type oven, for testing of a single test piece. For the heating chamber, other shapes than cylindrical are acceptable.

## 5.3 Thickness- and width-measuring devices

Instruments for measuring the thickness and width of the test piece shall be in accordance with ISO 23529.

## 6 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in [Annex A](#).

## 7 Test piece

### 7.1 Dimensions

#### 7.1.1 Method A:

The test piece shall be prepared by cutting from moulded flat sheet and shall have the shape and dimensions shown in [Figure 2](#). In addition, the thickness shall be  $(2 \pm 0,2)$  mm. The test piece shall have a smooth surface and be free from irregularities.

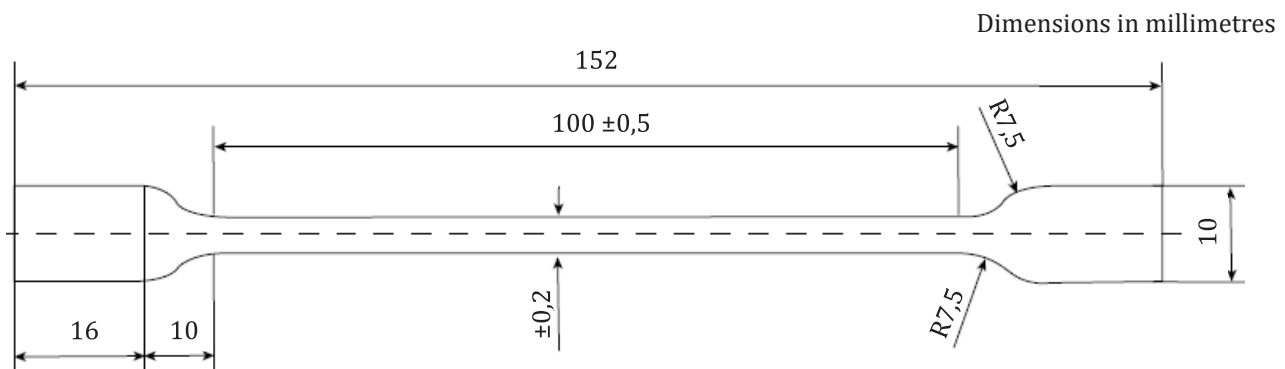


Figure 2 — Test piece for thermal-stress measurements

#### 7.1.2 Method B:

Wherever possible, the test specimens shall be dumb-bell-shaped, e.g. types 2 or 4 in accordance with ISO 37:2017. The test piece shall be prepared by cutting from moulded flat sheet. In addition, the thickness shall be  $(2 \pm 0,2)$  mm. The test piece shall have a smooth surface and be free from irregularities. For certain applications, both smaller and larger samples can be used.

## 7.2 Number of test pieces

The preferred number of test pieces is three for each set of test conditions, but for routine and screening tests, one or two test pieces are acceptable.

## 7.3 Time lapse between moulding and testing

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

## 7.4 Conditioning

Test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

Test pieces shall be conditioned at a standard laboratory temperature for at least 3 h immediately before being measured and tested.

If the test is to be carried out at a starting temperature other than a standard laboratory temperature, the test pieces shall be conditioned at the test temperature immediately prior to testing for a period sufficient to ensure that they have reached the test temperature (see ISO 23529).

If materials are tested, which need a relaxation process after sample manufacturing (e.g. injection molding for thermoplastic elastomers), a tempering process of 2 h at 80 °C is recommended to reduce the variation of the measurement results.

## 8 Test conditions

### 8.1 Heating rate and temperature interval under investigation

Method A: The elevated temperatures shall be selected from those specified in ISO 23529 unless otherwise necessary for technical reasons. Recommended test temperatures are 60 °C, 100 °C and 140 °C.

Method B: The duration of test depends on the heating rate and on the temperature interval under investigation. Preferably, a heating rate of 2 K/min shall be applied, if appropriate. The temperature interval under investigation shall be adapted to the decay of the stress.

The test shall be stopped at sample rupture or when the stress approaches zero. 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,  $F_0$ , reaches a predetermined value (e.g. 0,5).

During the entire test, the force shall be monitored as a function of temperature, where the data acquisition rate shall be sufficiently high. For tests performed with a heating rate of 2 K/min, the data acquisition rate shall have a value of at least 1 data point per second. In case of other heating rates, data acquisition rate shall be adapted appropriately.

### 8.2 Strain of the sample

Method A: A minimum of three pre-strains at each temperature shall be selected. Recommended pre-strains are 20 %, 40 % and 60 %.

Method B: Preferably, a strain of 50 % should be applied to the sample. Alternatively, in case of filled vulcanizates, a strain of 100 % might be more appropriate in order to reduce the influence of filler-filler interactions. The strain  $\varepsilon_t$  is defined as the percentage difference of the distance  $L$  between the clamps in the final position, and the distance of the clamps  $L_0$  in initial position, with reference to the distance  $L_0$ . Other strain values are acceptable, if appropriate. For instance, in case of samples of very high hardness or low value of ultimate strain, lower strain values are recommended.