INTERNATIONAL STANDARD

ISO 3384-2

First edition
2012-11-15

Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression —

Part 2: Testing with temperature cycling

Caoutchouc vulcanisé ou thermoplastique — Détermination de la relaxation de contrainte en compression —

Partie 2: Essais avec cycles de température

Reference number
ISO 3384-2:2012(E)

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.

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

ISO 3384 consists of the following parts, under the general title *Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression*:

— *Part 1: Testing at constant temperature*

— *Part 2: Testing with temperature cycling*

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 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.

If the life-time of a material is to be investigated, it can be determined using the method described in ISO 11346 (see the Bibliography).

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 can also influence the measured stress relaxation, particularly in rubbers containing fillers.

The two cycling test methods specified are designed to carry out the following:

— age the test piece by stress relaxation and determine the sealing force at low temperatures (method A);

— introduce thermal stress by stress relaxation and determine the sealing force at low temperatures (method B).

For products used in outdoor applications where the temperature can cycle between a low temperature (e.g. −40 °C) and a high temperature (e.g. 150 °C), it is important to also consider the shrinking of the rubber at low temperatures when assessing performance in the anticipated application and life-time.

For polymers that crystallize at low temperature, the crystallization will add to the shrinking of the rubber. For example, for hoses and seals in automotive applications, the product might work satisfactorily at the normal working temperature, but might leak at a low temperature.

Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression —

Part 2:
Testing with temperature cycling

IMPORTANT 1 — Persons using this part of ISO 3384 should be familiar with normal laboratory practice. This part of ISO 3384 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.

IMPORTANT 2 — Certain procedures specified in this part of ISO 3384 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 part of ISO 3384 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 then undergoes temperature cycling.

Method A: The temperature is cycled at intervals between a high temperature for ageing and a low temperature for checking the sealing force at this low temperature.

Method B: The temperature is cycled continuously between a high temperature and a low temperature to introduce thermal stress in the test piece.

The counterforce is determined by means of a continuous-measurement system.

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.

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 37:2011, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

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

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

ISO 18899:2004, *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.

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

3.2
thermal stress
mechanical stress induced in a body when some or all of its parts are not free to expand or contract in response to changes in temperature

4 Principle

A test piece of vulcanized or thermoplastic rubber is compressed to a constant deformation at which it is maintained. The decrease in counterforce is then measured.

The temperature is cycled between a high temperature and a low temperature to check the sealing force at this low temperature. The shrinkage of the rubber in going from the high to the low temperature decreases the counterforce.

5 Apparatus

5.1 Compression device, consisting of two parallel, flat, highly polished plates made from chromium-plated or stainless steel or other 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.

The surfaces of the compression plates shall be ground and polished. The compression plates shall be flat and parallel and shall not undergo any distortion when the test load is applied.

NOTE A finish to the surface giving a roughness profile *Ra* (see ISO 4287) of not worse than 0,4 µm has been found to be suitable. Such a roughness profile *Ra* can be obtained by grinding or polishing.

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 a temperature chamber at the specified test temperatures. 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 chamber.

5.2 Counterforce-measuring device, capable of measuring compression forces in the desired range with an accuracy of 1 % of the measured value.

The continuous-measurement system monitors the test piece during the whole duration of the test, thus making continuous measurement of the change in counterforce with time possible. The deformation of the test piece shall be kept within ±0,01 mm for the duration of the test.

5.3 Temperature chamber: For tests in air, a well designed, uniformly heated air temperature chamber shall be used, complying, for temperatures above standard laboratory temperature, with the requirements specified for one of the ovens used in ISO 188:2011, method A.

For cycling the temperature, the temperature chamber shall have a cooling and heating capability and be able to change the temperature at a rate of 1,0 °C/min ± 0,5 °C/min.

In the case of high-temperature tests and to avoid the surface oxidation of test pieces, other atmospheres may be used, such as nitrogen. 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 a temperature chamber as specified above.

NOTE It is recommended that the air used for air exchange be passed through an air dryer to give it a dew point not higher than −40 °C in order to avoid ice formation which can introduce friction in the measuring system.

5.4 Temperature-measuring equipment, with a sensing element having appropriate precision. The temperature-sensing element shall be installed in such a way that it accurately measures the temperature of the test piece.

NOTE A Pt100 sensor has been found to be suitable for temperature measurement.

6 Calibration

The requirements for calibration of the test apparatus are given in Annex A.

7 Test piece

7.1 Type and preparation of test piece

7.1.1 General

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

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

7.1.2 Cylindrical test pieces

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

7.1.3 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 37:2011.

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

The sheets may be prepared by moulding or from finished articles by cutting and buffing.

Alternatively, an O-ring with an internal diameter of 14,0 mm and a cross-section diameter of 2,65 mm may be used as the standard test piece.

O-rings of other dimensions, together with seals or gaskets of other configuration, may be used as non-standard test pieces, where appropriate.

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, but for routine and screening tests two test pieces are acceptable.

7.4 Time interval between forming and testing

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

7.5 Conditioning of test pieces

7.5.1 Prior to testing, the test pieces shall undergo first thermal and then mechanical conditioning as detailed in 7.5.2 and 7.5.3. When using method A at elevated temperature, the thermal conditioning might not be required, as the preheating of the test piece before compression acts as thermal conditioning.

7.5.2 Thermal conditioning shall be carried out by heating the test pieces at 70 °C for 3 h. Following thermal conditioning, the test pieces shall be allowed to stand for a period of not less than 16 h and not more than 48 h at standard laboratory temperature prior to mechanical conditioning or testing.

NOTE Some test samples, especially of thermoplastic elastomers, might contain moulding stresses, and thermal conditioning to relieve these stresses might improve the reproducibility of the results.

7.5.3 Mechanical conditioning shall be carried out at one of the standard laboratory temperatures specified in ISO 23529, as follows:

Compress the test pieces to the same compression that will be used during the rest of the test and then immediately return them to zero stress; repeat this procedure to give a total of five cycles of deformation and immediate return.

Following mechanical conditioning, the test pieces shall be allowed to stand for a period of not less than 16 h and not more than 48 h at standard laboratory temperature prior to testing.

Mechanical conditioning has been found to improve test reproducibility, particularly for compounds containing substantial proportions of filler, but is not always appropriate for finished products and can, therefore, lead to results that are not typical of service. Such conditioning may be omitted provided thermal conditioning is still undertaken. This omission shall be mentioned in the test report.

8 Duration, temperature and test liquid

8.1 Duration of test

Unless otherwise specified, the duration of test shall be ($168^{\ 0}_{-2}$) h (i.e. one week) or two or more weeks.

The test period begins after the initial compression. If test times longer than one week are used, a logarithmic time-scale shall be employed.

8.2 Temperature of exposure

The temperature of exposure shall be chosen from the list of test temperatures in ISO 23529. Temperatures of exposure which cause rapid degradation or evaporation of the test liquid shall be avoided. The temperature shall be kept as constant as possible during the test, with the following tolerances (in accordance with ISO 23529): ±2 °C for standard laboratory temperature (23 ± 2) °C or (27 ± 2) °C, ±1 °C for temperatures up to 100 °C and ±2 °C for temperatures above 100 °C. For subzero temperatures, the tolerance is ±2 °C.

For the cyclic tests specified in this part of ISO 3384, one elevated temperature and one subzero temperature shall be chosen.

8.3 Immersion liquids

The test liquid shall be chosen according to the particular application, but should preferably be one of those listed in ISO 1817.

9 Procedure

9.1 Preparation

Carefully clean the operating surfaces of the compression device. When testing in a gas, apply a thin coating of a lubricant having substantially no action on the rubber.

NOTE A silicone or fluorosilicone fluid (having a kinematic viscosity of about 0,01 m^2/s) and molybdenum disulfide have been found to be suitable lubricants.

9.2 Thickness measurement

9.2.1 Cylindrical test pieces

Measure the thickness of each test piece at the central portion with an accuracy of 0,01 mm, after thermal conditioning and before mechanical conditioning, at the chosen standard laboratory temperature, as specified in ISO 23529.

9.2.2 Ring test pieces

Measure the axial thickness of each test piece with an accuracy of 0,01 mm at four points approximately 90° apart around the ring after thermal conditioning and before mechanical conditioning, at the chosen standard laboratory temperature, as specified in ISO 23529. Use the average of the measurements to calculate the necessary compression. Individual measurements, on a single test piece, shall not differ by more than 0,05 mm. If they do, discard the test piece.

9.3 Method A

9.3.1 In this method, the test piece is aged at a high temperature, and the temperature is lowered once a week to a low temperature, at which the remaining counterforce is measured. A material or product specification can specify the temperatures and the minimum remaining counterforce (or the minimum value of F_{tx}/F_0) (see Clause 10) required at each temperature.

An example of a temperature cycle is shown in Figure 1.

9.3.2 Bring the compression device and the test chamber to the high test temperature.

9.3.3 When testing in a liquid, the test piece and the operating surfaces of the compression device shall be gently lubricated with the test liquid. When testing in a gaseous medium, a thin coating of a lubricant having substantially no action on the rubber shall be applied (see 9.1).

9.3.4 Immediately after lubrication, condition the test piece at the test temperature for at least 30 min in accordance with ISO 23529. For temperatures upwards of 150 °C, longer times are necessary in accordance with ISO 23529.

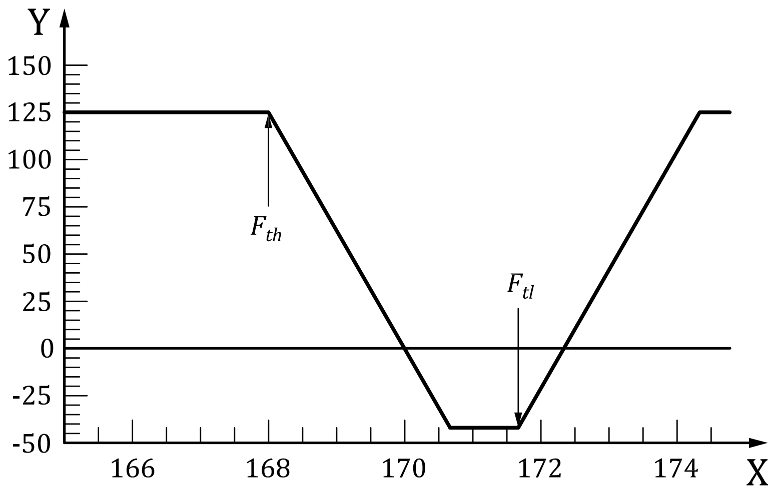
9.3.5 Place the preheated test piece in the preheated compression device (5.1) or, if the preheating is done in the compression device, place the test piece in the device and then preheat. In both cases, it is important to check that the test device and the test piece have reached the test temperature before compression is applied. Sometimes, it is necessary to increase the preheating time.

9.3.6 Compress the test piece by (25 ± 2) % in the compression device at the test temperature or, if a compression of 25 % cannot be obtained, use a compression of (15 ± 2) % or lower, in steps of 5 %. Compress the test piece in a time between 30 s and 120 s. When reached, the final compression shall be fixed and maintained during the entire test period.

9.3.7 Determine the initial counterforce F_0 with an accuracy of 1 % of the measured value, at the high test temperature, 30 min ± 1 min after completing the compression.

9.3.8 After 168 h, lower the temperature at a rate of 1 °C/min ± 0,5 °C/min to the low temperature. Keep the low temperature steady for 1 h. Then increase the temperature at a rate of 1 °C/min ± 0,5 °C/min to the high temperature again. Repeat this cycle weekly. The time for cooling down and the time at the low temperature are not included in the ageing time, but the time to increase the temperature is included.

9.3.9 Measure the counterforce F_{tx} continuously, starting with a measurement interval of e.g. 1 min on the first day and continuing with an interval of e.g. 10 min during the first week and 60 min during the remainder of the test.



Key

X time, h

Y temperature, °C

Figure 1 — Example of a temperature cycle used in method A

9.4 Method B

9.4.1 In this method, temperature cycling is used to introduce thermal stress, starting at standard laboratory temperature and cycling between a high and a subzero temperature. A material or product specification can specify the number of cycles, the temperatures and the minimum remaining counterforce (or the minimum value of F_{tx}/F_0) required at each temperature.

An example of a temperature cycle is shown in Figure 2.

9.4.2 Bring the compression device and the test chamber to standard laboratory temperature.

9.4.3 When testing in a liquid, the test piece and the operating surfaces of the compression device shall be gently lubricated with the test liquid. When testing in a gaseous medium, a thin coating of a lubricant having substantially no action on the rubber shall be applied (see 9.1).

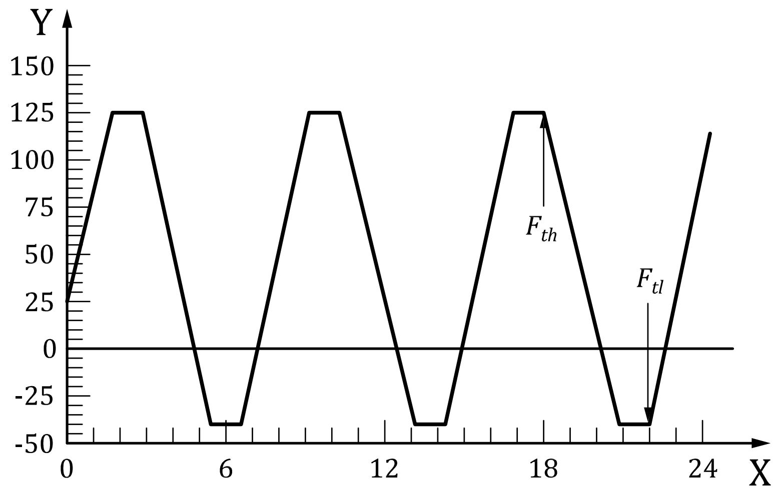
9.4.4 Place the conditioned test piece in the compression device (5.1) at standard laboratory temperature. It is important to check that the test device and the test piece have reached the test temperature before compression is applied.

9.4.5 Compress the test piece by (25 ± 2) % in the compression device or, if a compression of 25 % cannot be obtained, use a compression of (15 ± 2) % or lower, in steps of 5 %. Compress the test piece in a time between 30 s and 120 s. When reached, the final compression shall be fixed and maintained during the entire test period.

9.4.6 Determine the initial counterforce F_0 with an accuracy of 1 % of the measured value, at standard laboratory temperature, 30 min ± 1 min after completing the compression.

9.4.7 Increase the temperature at a rate of 1,0 °C/min ± 0,5 °C/min to the high temperature. Keep the test piece at this temperature for 1 h. Then decrease the temperature at a rate of 1,0 °C/min ± 0,5 °C/min to the subzero temperature and keep the test piece at this temperature for 1 h. Repeat this cycle 2 to 4 times daily, giving a total of 3 to 5 cycles. The precise number of cycles carried out per day will depend on the high and the low temperature used, and of the rate of increase and decrease of the temperature.

9.4.8 Measure the counterforce F_{tx} continuously, with a measurement interval of 5 min maximum.



Key

X time, h

Y temperature, °C

Figure 2 — Example of a temperature cycle used in method B

10 Expression of results

The compression stress relaxation $R(tx)$ after a specified duration of test t, expressed as a percentage of the initial counterforce, is given by the equation

$$R(tx) = \frac{F_0 - F_{tx}}{F_0} \times 100$$

where

F_0 is the initial counterforce, measured after 30 min;

F_{tx} is the counterforce measured after the specified duration of test t;

x is replaced by h for the high temperature and by l for the low temperature.

The median value of the results for the test pieces shall be taken. The individual values for the test pieces shall agree to within 10 % of the median value. If they do not, the test shall be repeated.

The counterforce at the high temperature, F_{th}, is determined after the specified time, e.g. 168 h, and the counterforce at the low temperature is determined, during the following low-temperature part of the cycle, after 1 h at the low temperature.

Stress relaxation values measured after different times of exposure shall be plotted as a function of time on a logarithmic scale to facilitate the interpretation of test data. For some applications, it is more useful to calculate compression stress ratio values, i.e. F_{tx}/F_0, after different times of exposure, rather than stress relaxation values. In this case, compression stress ratio values shall be presented graphically as a function of logarithmic time.

Key

X time, h

Y F_{tx}/F_0

Figure 3 — Force curve shown as F_{tx}/F_0 recorded during a method A test

11 Precision

An interlaboratory test programme has not yet been conducted.

12 Test report

The test report shall include the following information:

a) sample details:

   1) a full description of the sample and its origin,

   2) the compound details and cure conditions, where appropriate,

   3) the method of preparation of the test pieces from the sample, e.g. whether moulded or cut;

b) a full reference to the test method used, i.e. the number of this part of ISO 3384, its year of publication and the method utilized, e.g. ISO 3384-2:2012, method A;

c) test details:

   1) the type of test piece used,

   2) the number of test pieces tested,

   3) any special information concerning the apparatus, e.g. the method used for measuring the counterforce,

   4) the standard laboratory temperature used,

   5) the duration and temperature of conditioning of the test pieces prior to testing,

   6) the test duration and the high and low temperatures used,

   7) the number of cycles used,

   8) the compression used: 25 % or other (give details),

   9) the type of temperature chamber used,

   10) the lubricant used,

   11) any deviation, by agreement or otherwise, from the specified test procedure;

d) the individual test results and their median value for both the high temperature, i.e. for $R(th)$, and for the low temperature, i.e. for $R(tl)$;

e) the dates of the test.

Annex A
(normative)

Calibration schedule

A.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded on any calibration report or certificate. It shall be reported whether calibration is made in the “as-received” condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

A.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this part of ISO 3384. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table A.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code-letter. The code-letters used in the calibration schedule are:

C requirement to be confirmed but no measurement;

N initial verification only;

S standard interval as advised in ISO 18899.

Table A.1 — Calibration schedule

Parameter	Requirement(s)	Subclause in ISO 18899:2004	Verification frequency guide	Notes
Force measurement	Accurate to within 1 %	21.2	S	
Deformation of test piece (i.e. position of upper plate)	Kept constant to within ±0,01 mm during test	15.2	N	

Table A.1 *(continued)*

Parameter	Requirement(s)	Subclause in ISO 18899:2004	Verification frequency guide	Notes
Compression device plates	Parallel, i.e. 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 the test piece between the plates, neither compression plate shall bend by more than 0,01 mm. Flat. Highly polished. Made of corrosion-resistant material. Size sufficient 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.	C	N	
Temperature chamber	Meeting, for elevated temperatures, the requirements specified for one of the ovens used in ISO 188:2011, method A.	C	S	
Temperature sensor	Fitted in such a way that it accurately measures the temperature of the test piece.	C	N	

In addition to the items listed in Table A.1, use of the following is implied, which need calibrating in accordance with ISO 18899:

— a timer;

— an instrument for determining the dimensions of the test pieces.

Bibliography

[1] ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

[2] ISO 11346, *Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use*

ICS 83.060

Price based on 12 pages