INTERNATIONAL STANDARD

**ISO 3384**

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# Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression at ambient and at elevated temperatures

*Caoutchouc vulcanisé ou thermoplastique — Détermination de la relaxation de contrainte en compression à température ambiante et aux températures élevées*

Reference number
ISO 3384:2005(E)

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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.

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 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 3384:1999), of which it constitutes a minor revision designed to update the normative references. It also incorporates the Amendment ISO 3384:1999/Amd. 1:2001, *Precision data*.

# 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 may 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 whilst at high temperatures and/or long times chemical processes are dominant.

If the lifetime of a material is to be investigated, it can be determined using the air oven ageing test described in ISO 11346[1)].

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

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

1) ISO 11346, *Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use*.

# Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression at ambient and at elevated temperatures

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

## 1 Scope

This International Standard 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 maintained thus at a predetermined test temperature.

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.

Testing at temperatures below standard laboratory temperature is not specified. The methods have been used for low-temperature testing, but their reliability under these conditions is not proven.

## 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 37:2005, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

ISO 188:1998, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

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

ISO 3601-1:2002, *Fluid power systems — O-rings — Part 1: Inside diameters, cross-sections, tolerances and size identification code*

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

ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

ISO 23529:2004, *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

## 4 Principle

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

**In method A,** the compression is applied and all counterforce measurements are made at the test temperature.

**In method B,** the compression is applied and all counterforce measurements are made at standard laboratory temperature. The test pieces are stored at the test temperature.

NOTE 1 The two methods, A and B, of carrying out the measurement do not give the same values of stress relaxation, and comparison of values obtained from the two methods should be avoided. The method selected for use depends on the purpose of the test. Thus, for fundamental studies and in applications where sealing at elevated temperatures is a problem, method A may be preferred, and in applications where temperature cycling from normal to an elevated temperature is a problem, method B may be preferred.

NOTE 2 Other methods can be used for specific purposes, such as applying the compression at standard laboratory temperature and making all counterforce measurements at a different temperature.

## 5 Apparatus

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

When the apparatus is disassembled, the compression plates shall be flat to within 0,01 mm. The finish of the surface shall not be worse than $Ra$ 0,4 µm (see ISO 4287). When the apparatus is assembled without a test piece, the gap between the plates shall not vary by more than $\pm$ 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 an oven at the specified test temperature. 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 oven.

**5.2 Counterforce-measuring device,** capable of measuring compression forces in the desired range with an accuracy of 1 % of the measured value. The preferred device is one that monitors the test piece during the whole duration of the test, in which case continuous measurement of the change in counterforce with time is possible. The deformation of the test piece shall be kept within ± 0,01 mm for the duration of the test.

Alternatively, a compression-testing machine can be used to measure the counterforce at prescribed time intervals. In this case, the force necessary to cause a slight increase in the compression of the test piece is measured. This additional compression shall be as small as possible and in no case greater than a force of 1 N for balance-type machines, or greater than 0,05 mm for stress/strain-type machines, applied in either case without overshoot. The whole of the force exerted by the test piece as a result of the extra compression shall act on the force-measuring device. It shall also be possible to repeat the compression to within ± 0,01 mm from one measurement to another.

**5.3 Test chamber,** complying with the requirements of ISO 188:1998, method A.

For tests in air, a well designed, uniformly heated air oven shall be used, provided with adequate temperature control to maintain the specified air temperatures within the tolerance specified in 7.2. 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 an air oven as specified above.

**5.4 Temperature-measuring equipment,** with a sensing element, for example a PT100 element, class A or better. The temperature-sensing element shall be mounted so that it is located not more than 2 mm from a surface of the test piece, in one of the compression plates.

# 6 Test piece

## 6.1 Type and preparation of test piece

### 6.1.1 General

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

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

### 6.1.2 Cylindrical test pieces

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

### 6.1.3 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 37:2005.

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 can be prepared by moulding or from finished articles by cutting and buffing.

Alternatively, an O-ring, size code 14,0 × 2,65-G-N-ISO 3601-1, as specified in ISO 3601-1:2002 (internal diameter 14,0 mm and diameter of the cross-section 2,65 mm), can be used as the standard test piece.

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

NOTE Most test machines have jigs in which the test piece is compressed by screwing a compression plate down to stops. This gives a fixed strained thickness. Test pieces within the tolerances given above will not necessarily have the required compression strain when tested in such jigs. It is important that a compression strain within the limits given in 8.3.4 and 8.4.3 is achieved by careful matching of jig and test piece.

## 6.2 Measurement of dimensions of test pieces

The dimensions of test pieces shall be measured as specified in ISO 23529.

## 6.3 Number of test pieces

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

## 6.4 Time interval between vulcanization and testing

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

## 6.5 Conditioning of test pieces

**6.5.1** Prior to testing, the test pieces shall undergo first a thermal and then a mechanical conditioning as detailed in 6.5.2 and 6.5.3.

**6.5.2** 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, may contain moulding stresses, and thermal conditioning to relieve these stresses may improve the reproducibility of the results.

**6.5.3** 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 may 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.

# 7 Duration, temperature and test liquid

## 7.1 Duration of test

Unless otherwise specified, the duration of test shall be $(168\ ^{0}_{-2})$ h.

If intermediate times are used, 3 h $^{0}_{-10}$ min, 6 h $^{0}_{-20}$ min, $(24\ ^{0}_{-0,5})$ h and $(72\ ^{0}_{-1})$ h are preferred. The test period begins after the initial compression. If longer test times are used, a logarithmic time-scale shall be employed.

In method B, when compression is carried out at standard laboratory temperature, each time the test piece is conditioned for measurement at that temperature a conditioning period of 2 h (not included in the time of test) shall be allowed.

## 7.2 Temperature of exposure

The temperature of exposure shall be chosen from the list of standard 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 a tolerance of $\pm$ 1 °C for all temperatures, including standard laboratory temperature.

## 7.3 Immersion liquids

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

# 8 Procedure

## 8.1 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 fluoro-silicone fluid (having a kinematic viscosity of about 0,01 $m^2/s$) and molybdenum disulfide have been found to be suitable lubricants.

## 8.2 Thickness measurement

### 8.2.1 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 23529:2004, method A.

### 8.2.2 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.

## 8.3 Method A

**8.3.1** Bring the compression device and the test environment to the test temperature.

**8.3.2** 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 8.1).

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

**8.3.4** Place the conditioned 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. Compress the test piece by (25 ± 2) % in the compression device at the test temperature or use a compression of (15 ± 2) % or lower, in steps of 5 %, if a compression of 25 % cannot be obtained. 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 (apart from the further small compression which is used for measurement of the counterforce as mentioned in the alternative method in 5.2).

**8.3.5** Measure the counterforce with an accuracy of 1 % of the measured value, at the test temperature, 30 min ± 1 min after completing the compression.

**8.3.6** Repeat the measurement of the counterforce after the times specified in 7.1. Take all measurements at the test temperature.

NOTE 1 After the last measurement at the test temperature, the test piece may be allowed to cool down to standard laboratory temperature and a further measurement of the counterforce made.

NOTE 2 Valuable additional information can be obtained after the relaxation test has been finished. Research has shown that the amount of recovery (at the test temperature) is a measure of the permanent chemical reactions occurring alongside physical relaxation (see A.M. Prabhu, A.W. Birley and R.H. Sigley, *Polymer Testing*, 1991, **10**, 30).

## 8.4 Method B

**8.4.1** Bring the test environment to the test temperature.

**8.4.2** 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 gas, a thin coating of a lubricant having substantially no action on the rubber shall be applied (see 8.1).

**8.4.3** Compress the test piece by (25 ± 2) % in the compression device at standard laboratory temperature or use a compression of (15 ± 2) % or lower, in steps of 5 %, if a compression of 25 % cannot be obtained. 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 (apart from the further small compression which is used for measurement of the counterforce as mentioned in the alternative method in 5.2).

**8.4.4** Measure the counterforce with an accuracy of 1 % of the measured value, at standard laboratory temperature, 30 min ± 1 min after completing the compression.

**8.4.5** Immediately after measuring the counterforce, store the compressed test piece in the test environment (see 5.3) at the specified test temperature.

**8.4.6** When making measurements of counterforce after the times specified (see 7.1), remove the apparatus from the test environment, maintain it at the standard laboratory temperature for 2 h, determine the counterforce and then return it to the test environment for a further time. It is important that the apparatus and test piece reach thermal equilibrium within 2 h, and forced cooling can be necessary. The temperature shall be checked with the temperature sensor specified in 5.4.

## 9 Expression of results

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

$$R(t) = \frac{F_0 - F_t}{F_0} \times 100$$

where

$F_0$ is the initial counterforce, measured after 30 min;

$F_t$ is the counterforce measured after the specified duration of test $t$.

The median value of the results for the test pieces shall be chosen. 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.

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. $F_t/F_0$, 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.

## 10 Precision

### 10.1 General

An interlaboratory test programme (ITP) and the precision calculations to express the repeatability and reproducibility were performed in accordance with ISO/TR 9272. Consult this for precision concepts and nomenclature.

Annex A gives guidance on the use of repeatability and reproducibility results.

### 10.2 Precision details

**10.2.1** The ITP was conducted in 1998. One material, an IR/SBR blend rubber compound, was used. Testing using method A was conducted at 23 °C and 100 °C and using method B at 100 °C. A test result is taken as the average value, for two test pieces, of the percent decrease in the initial counterforce after 168 h of relaxation. Twelve laboratories participated in the 23 °C testing using method A, eleven laboratories in the 100 °C testing using method A and seven laboratories in the 100 °C testing using method B.

**10.2.2** The precision determined is a type 1 precision; fully prepared test pieces were submitted to the laboratories. The precision is also an intermediate-term precision with a span of 2 or 3 weeks between the two replications. This is required due to the relaxation-ageing period of 168 h for each replication of the test. This is in distinction to the more usual day 1/day 2 replication with a few days between replications.

**10.2.3** Analysis of the data from all the laboratories (all three tests) resulted in

— the results from three laboratories being declared outliers for method A at 23 °C;

— the results from two laboratories being declared outliers for method A at 100 °C;

— the result from one laboratory being declared an outlier for method B at 100 °C.

These results were rejected and the final analysis was conducted on the remaining data, viz:

— for method A at 23 °C: the results from nine laboratories;

— for method A at 100 °C: the results from nine laboratories;

— for method B at 100 °C: the results from six laboratories.

The revised database represents those laboratories that had good within-laboratory control of the testing (the results are in relatively good agreement).

### 10.3 Precision results

The precision data obtained from the final database are given in Table 1. The precision (both repeatability and reproducibility) of method B at 100 °C is substantially worse than that for method A. No relative precision, ($r$) and ($R$), is given for this International Standard.

The symbols used in Table 1 are as follows:

$s_r$ is the repeatability standard deviation, in measurement units;

$r$ is the repeatability, in measurement units (i.e. % relaxation);

$s_R$ is the reproducibility standard deviation, in measured units;

$R$ is the reproducibility, in measurement units (i.e. % relaxation).

**Table 1 — Precision results**

| **Method A, 168 h at 23 °C** | | | | | |
|---|---|---|---|---|---|
| Material | Mean % relaxation | $s_r$ | $r$ | $s_R$ | $R$ |
| A | 10,9 | 0,795 | 2,22 | 1,21 | 3,40 |
| **Method A, 168 h at 100 °C** | | | | | |
| Material | Mean % relaxation | $s_r$ | $r$ | $s_R$ | $R$ |
| A | 50,5 | 0,845 | 2,37 | 2,15 | 6,03 |
| **Method B, 168 h at 100 °C** | | | | | |
| Material | Mean % relaxation | $s_r$ | $r$ | $s_R$ | $R$ |
| A | 67,5 | 2,07 | 5,8 | 8,66 | 24,3 |

## 11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) sample details:

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

   2) compound details and cure conditions, where appropriate,

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

c) test details:

   1) the test method used (A or B),

   2) the type of test piece used,

   3) any special data 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 test pieces prior to testing,

   6) the test duration and temperature,

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

   8) the test environment used,

   9) the lubricant used,

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

d) test results:

   1) the number of test pieces used,

   2) the individual test results and/or their median value;

e) the date of the test.

# Annex A
(informative)

# Guidance for using precision results

**A.1** The general procedure for using precision results is as follows, with the symbol $|x_1 - x_2|$ designating a positive difference in any two measurement values (i.e. without regard to sign).

**A.2** Enter the appropriate precision table (for whatever test parameter is being considered) at an average value (of the measured parameter) nearest to the "test" data average under consideration. This line will give the applicable $r$, $(r)$, $R$ or $(R)$ for use in the decision process.

**A.3** With these $r$ and $(r)$ values, the following general repeatability statements may be used to make decisions.

**A.3.1** For an absolute difference: The difference $|x_1 - x_2|$ between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability $r$ on average not more than once in twenty cases.

**A.3.2** For a percentage difference between two test (value) averages: The percentage difference

$$\frac{|x_1 - x_2|}{0{,}5(x_1 + x_2)} \times 100$$

between two test values, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability $(r)$ on average not more than once in twenty cases.

**A.4** With these $R$ and $(R)$ values, the following general reproducibility statements may be used to make decisions.

**A.4.1** For an absolute difference: The absolute difference $|x_1 - x_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility $R$ not more than once in twenty cases.

**A.4.2** For a percentage difference between two test (value) averages: The percentage difference

$$\frac{|x_1 - x_2|}{0{,}5(x_1 + x_2)} \times 100$$

between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility $(R)$ not more than once in twenty cases.

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