

International Standard



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Rubber, vulcanized — Determination of compression stress relaxation (rings)

Caoutchoucs vulcanisés — Détermination de la relaxation de contrainte en compression (anneaux)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6056 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in November 1978.

It has been approved by the member bodies of the following countries :

Austria	Germany, F. R.	Poland
Belgium	Greece	Romania
Brazil	Hungary	South Africa, Rep. of
Bulgaria	India	Sweden
Canada	Ireland	Thailand
China	Italy	Turkey
Czechoslovakia	Korea, Rep. of	United Kingdom
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The member body of the following country expressed disapproval of the document on technical grounds :

USA

Rubber, vulcanized — Determination of compression stress relaxation (rings)

1 Scope and field of application

This International Standard specifies three methods for determining the decrease in counterforce exerted by a ring-shaped test piece of vulcanized rubber which has been compressed to a constant deformation and maintained thus at a predetermined test temperature. The methods are particularly suitable for the determination of stress relaxation in liquid environments.

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.

In method C, the compression is applied at standard laboratory temperature and all counterforce measurements are made at the test temperature.

An alternative method, using cylindrical test pieces, is specified in ISO 3384.

NOTES

1 The three methods, A, B, and C, of carrying out the measurement do not give the same values of stress relaxation and comparison of values obtained from the three methods must be avoided. The method selected for use depends on the purpose of the test.

2 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 References

ISO 37, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 468, *Surface roughness — Parameters, their values and general rules for specifying surfaces.*¹⁾

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO/R 1078, *Dimensions of elastomeric toroidal sealing rings for aircraft (Inch series — Class 1 tolerances).*

ISO 1817, *Vulcanized rubbers — Resistance to liquids — Methods of test.*

ISO 1826, *Rubbers — Time-lapse between vulcanization and testing.*

ISO 3383, *Rubber — General directions for achieving elevated or sub-normal temperatures for tests.*

ISO 3384, *Vulcanized rubbers — Determination of stress relaxation in compression at normal and at elevated temperatures.*

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

ISO 4648, *Rubber, vulcanized — Determination of dimensions of test pieces and products for test purposes.*

ISO 4661, *Rubber — Preparation of test pieces.*

3 Definition

compression stress relaxation: The 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 Apparatus

4.1 Compression device, consisting of two parallel, flat, highly polished, chromium plated or stainless steel plates, between the faces of which the test pieces are compressed.

The finish of the surface of the compression plates shall not be worse than 0,2 μm centre line average (see ISO 468). The plates shall be sufficiently rigid to withstand the stress without bending and of size sufficient to ensure that the whole of the compressed test piece is within the area of the plates.

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.

The compression device shall be connected to suitable equipment for compressing the test piece to the specified compression within 30 s and for measuring the counterforce exerted by the compressed test piece with an accuracy of 2 % of the measured value. It shall be capable of setting and maintaining the compression during the whole duration of the test and shall be such that it should be possible to keep it in an oven at the

1) At present at the stage of draft. (Revision of ISO/R 468-1966.)

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.

4.2 Counterforce measuring device, capable of measuring compression forces in the desired range with an accuracy of 2 % of the measured value. The device may be such as to contain the test pieces during the whole duration of the test, in which case continuous measurements are possible. Alternatively, a testing machine may be used in which the counterforce is measured at prescribed time intervals, on test pieces compressed in a suitable jig, by increasing the compression by no more than 0,05 mm.

4.3 Test chamber, complying with the requirements of ISO 3383.

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 tolerances specified in 7.2. Satisfactory circulation of the air shall be secured by means of a fan.

For tests in liquids, the compression device shall be totally immersed in the liquid in a bath, or a closed vessel for volatile and 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.

4.4 Temperature measuring equipment, with a sensing element, for example a thermocouple. The element shall be mounted in a sealed hole drilled in one of the compression plates so that it is located no more than 2 mm from the surface contacting the test piece.

5 Test piece

5.1 Type and preparation of test piece

5.1.1 General

Test pieces shall be prepared as specified in ISO 4661. The type 1 test piece is preferred.

5.1.2 Type 1

The type 1 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 the annex of ISO 37.

The dimensions of test pieces shall be :

- thickness : $2,0 \pm 0,2$ mm
- inner diameter : $15,0 \pm 0,2$ mm
- radial width : $2,0 \pm 0,2$ mm

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

5.1.3 Type 2

Alternatively, an O-ring, size code 26501400 as specified in ISO 3601/1 (cross-section 2,65 mm and internal diameter 14,0 mm), or ring size 113 as specified in ISO/R 1078 (cross-section $2,616 \pm 0,076$ mm and internal diameter $13,4 \pm 0,1$ mm) may be used as the standard test piece.

NOTES

- 1 The results obtained from the different types of test piece cannot be compared.
- 2 O-rings of other dimensions, together with seals or gaskets of other configuration, may be used as non-standard test pieces where appropriate.

5.2 Measurement of dimensions of test pieces

The dimensions of the test pieces shall be measured as specified in ISO 4648.

5.3 Number of test pieces

At least three tests using separate test pieces shall be carried out.

5.4 Time-interval between vulcanization and testing

The interval between vulcanization and testing shall be in accordance with ISO 1826.

5.5 Conditioning of test pieces

Test pieces shall be conditioned at standard laboratory temperature in accordance with ISO 471 before testing.

6 Procedure

6.1 Carefully clean the operating surfaces of the compression device. The test piece surface shall be substantially free from mould release agents or dusting powder.

6.2 Measure the axial thickness of each test piece with an accuracy of 0,01 mm at four points approximately 90° apart around the ring, at standard laboratory temperature, as specified in ISO 4648. The average of the measurements shall be used for calculation of the necessary compression. Individual measurements on a single test piece should not differ by more than 0,05 mm; if they do discard the test piece.

6.3 Method A

6.3.1 Bring the compression device and the test environment to the test temperature.

6.3.2 When testing in a liquid environment, 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 fluorosilicone fluid, having a kinematic viscosity of about 0,01 m²/s, is a suitable lubricant.

6.3.3 Immediately after lubrication, condition the test piece at the test temperature in accordance with ISO 3383. Conditioning for 15 min is recommended.

6.3.4 Compress the conditioned test piece in the compression device (see 4.1). The compression should be between 15 and 30 % with a tolerance of ± 1 % on the nominal value to correspond to the product specification. For general testing, a compression of 25 ± 1 % is preferred. Apply the compression within 30 s. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which may be used for measurement of counterforce, mentioned in 4.2).

6.3.5 Within 5 min of completing the compression, immerse the part of the compression device containing the test piece in the test environment at the test temperature.

6.3.6 Measure the counterforce with an accuracy of 2 % of the measured value, at the test temperature, 30 ± 1 min after applying the counterforce.

6.3.7 Repeat the measurement of the counterforce after different times according to 7.1. Take all measurements at the test temperature.

6.4 Method B

6.4.1 Bring the test environment to the test temperature.

6.4.2 When testing in a liquid environment, 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 fluorosilicone fluid, having a kinematic viscosity of about 0,01 m²/s, is a suitable lubricant.

6.4.3 Within 10 min of lubrication, compress the test piece in the compression device at standard laboratory temperature (see 4.1). The compression should be between 15 and 30 % with a tolerance of ± 1 % on the nominal value to correspond to the product specification. For general testing, a compression of 25 ± 1 % should be used. Apply the compression within 30 s.

6.4.4 Measure the counterforce with an accuracy of 2 % of the measured value, at standard laboratory temperature, 30 ± 1 min after applying the counterforce.

6.4.5 Immediately after measuring the counterforce, store the compressed test piece in the test environment (see 4.3) at the specified test temperature.

6.4.6 When making measurements of counterforce after the times specified in 7.1, first bring the compression device with the compressed test piece to standard laboratory temperature and test 2 h after removal from the test environment.

6.5 Method C

6.5.1 Bring the test environment to the test temperature.

6.5.2 When testing in a liquid environment, 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 fluorosilicone fluid, having a kinematic viscosity of about 0,01 m²/s, is a suitable lubricant.

6.5.3 Within 10 min of lubrication, compress the test piece at standard laboratory temperature. Apply the compression within 30 s.

6.5.4 Within 5 min of completing the compression, immerse the part of the compression device containing the test piece in the test environment at the test temperature.

6.5.5 Two hours after placing the test piece in the test environment, measure the counterforce with an accuracy of 2 % of the measured value at the test temperature.

6.5.6 Repeat the measurement of the counterforce after different times according to 7.1. Take all measurements at the test temperature.

7 Duration, temperature and test liquid

7.1 Duration of test

Unless otherwise agreed between the interested parties, the test period shall be $168 \pm \frac{0}{2}$ h. If longer testing times are needed, equal increments on a logarithmic time scale should be used.

In method B, when compression is effected 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 one of the following list of preferred temperatures :

23 $\pm$ 2 °C	150 $\pm$ 2 °C
27 $\pm$ 2 °C	175 $\pm$ 2 °C
70 $\pm$ 1 °C	200 $\pm$ 2 °C
85 $\pm$ 1 °C	225 $\pm$ 2 °C
100 $\pm$ 1 °C	250 $\pm$ 2 °C
125 $\pm$ 2 °C	

If there is no technical reason for choosing a particular temperature, 100 °C should be used.

Temperatures of exposure which cause rapid degradation or evaporation of the test liquid shall be avoided.

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 Expression of results

The stress relaxation R , expressed as a percentage of the initial counterforce, is given by the formula

$$\frac{F_0 - F_1}{F_0} \times 100$$

where

F_0 is the initial counterforce measured 30 min or 2 h, as appropriate, after compression of the test piece;

F_1 is the counterforce measured after the specified time.

The median value of the results for the test pieces shall be taken. Individual values for the test pieces shall be within 10 % of the median value. If they are not, the test shall be repeated using at least three further test pieces and the median value of the results for all test pieces shall be quoted.

Stress relaxation values measured after different times of exposure may be plotted as a function of time on a logarithmic scale to give a better understanding of the combined influence of the swelling and relaxation processes.

NOTE — For some applications, it may be more useful to calculate compression stress ratio values, i.e. F_1/F_0 , after different times of exposure, rather than stress relaxation values. The compression stress ratio values may be presented graphically as a function of time.

9 Test report

The test report shall include the following particulars :

a) sample details :

- 1) a full description of the sample and its origin;
- 2) compound and cure details, where appropriate;
- 3) the method of preparation of test pieces from samples, for example whether moulded or cut;

b) test method :

- 1) a full reference to the test method, i.e. the number of this International Standard;
- 2) the method used, A, B or C;
- 3) the type of test piece used;
- 4) any special data concerning the apparatus, for example the method used for measuring the counterforce;

c) test details :

- 1) the standard laboratory temperature;
- 2) the duration and temperature of conditioning of test pieces prior to testing;
- 3) the test duration and temperature;
- 4) the test environment used;
- 5) the nature of the lubricant;
- 6) 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 median value;

e) date of test.