INTERNATIONAL STANDARD

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Rubber, vulcanized or thermoplastic — Determination of compression stress-strain properties

Caoutchouc vulcanisé ou thermoplastique — Détermination des propriétés de contrainte/déformation en compression



ISO 7743:2011(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 7743 was prepared by Technical Committee ISO/TC 45, Rubber and rubber products, Subcommittee SC 2, Testing and analysis.

This fourth edition cancels and replaces the third edition (ISO 7743:2008), which has been technically revised.

Introduction

Knowledge of compression stress-strain properties is important in the design of, for instance, bridge bearings, anti-vibration mountings and O-rings. Measurement of compression stress-strain behaviour is also used for the quality control of small O-rings and other small products (i.e. those under 2 mm thick) where hardness cannot easily be measured. Compression tests are also used to detect the presence of porosity in products such as pipe sealing rings. Compression can be uniaxial or biaxial depending on test piece shape and experimental conditions. If there is no friction at the interface between the test piece and the compression device, compression is uniaxial. If friction is significant, the test piece shape affects the nature of the compression. When the thickness of the test piece is small, Saint Venant's principle is not applicable: the boundary condition at the interface influences the stress and strain fields and compression becomes biaxial (the thinner the test piece, the higher the biaxiality). The test piece behaves as if an additional radial compression were applied (friction hampers the radial expansion due to axial compression) and this phenomenon has to be taken into account when material properties such as moduli are to be derived from compression results.

Rubber, vulcanized or thermoplastic — Determination of compression stress-strain properties

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.

1 Scope

This International Standard specifies methods for the determination of the compression stress-strain properties of vulcanized or thermoplastic rubber using a standard test piece, a product or a part of a product.

Four procedures are given:

- using standard test piece A with the metal plates lubricated (method A);
- using standard test piece A with the metal plates bonded to the test piece (method B);
- using standard test piece B (method C);
- using a product or a part of a product with the metal plates lubricated (method D).

The four procedures do not give the same results. Method A (test piece A, lubricated) gives results which are dependent only on the modulus of the rubber and are independent of the test piece shape, provided that complete slip conditions are achieved. Effective lubrication is sometimes difficult to achieve, however, and it is prudent to inspect the variance in the test results from replicate test pieces for indications of erratic slip conditions. Method B (test piece A, bonded) gives results which are dependent on both the modulus of the rubber and the test piece shape. The dependence on test piece shape is strong and, consequently, the results are markedly different from those obtained with lubricated test pieces. Method C (test piece B) gives results which are independent of both the test piece shape and the lubrication conditions. This test piece is more appropriate and more convenient when intrinsic material properties are to be determined (see Annex A for details). For products (method D), the result is dependent on the shape, but as tests on products are mainly comparative, this is acceptable.

NOTE For well-specified product shapes, such as O-rings, the result can be correlated to the hardness value.

Provision is made for the use of test pieces of different size and/or shape from the specified test pieces, but extrapolation of the results obtained to other sizes and shapes can prove impossible.

Information on the effect of size and shape of test piece and of bonding or lubrication is given in Annex A.

The method is not suitable for materials that exhibit high set.

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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 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

stress applied so as to cause a deformation of the test piece in the direction of the applied stress, expressed as the force divided by the original area of cross-section perpendicular to the direction of application of the force

3.2

compression strain

deformation of the test piece in the direction of the applied stress divided by the original dimension in that direction

NOTE The compression strain is commonly expressed as a percentage of the original dimension of the test piece.

3.3

compression modulus

secant modulus

applied stress calculated on the original area of cross-section divided by the resultant strain in the direction of application of the stress

3.4

stiffness at 25 % compression

force which needs to be applied to a product or a part of a product to compress it by 25 %, expressed in newtons per metre or in newtons, depending on the shape of the test piece

4 Principle

A test piece (lubricated or bonded) is compressed at a constant speed between the compression plates until a pre-determined strain is reached.

5 Apparatus and materials

5.1 Flat metal plates, of uniform thickness and having lateral dimensions greater than or equal to those of test pieces for bonding or at least 20 mm greater than those of test pieces for lubrication.

For methods A and D, one surface of each plate shall be highly polished.

NOTE A surface finish not worse than Ra 0,4 μ m (see ISO 4287[2]) has been found to be suitable. Such an Ra can be obtained by a grinding or polishing operation.

For method B, one surface of each plate shall be suitably prepared for the bonding system to be used.

For method C, no specific preparation of the contact surfaces is required.

- **5.2** Dies and cutters (if required), for preparing test pieces, complying with the relevant requirements of ISO 23529.
- **5.3 Thickness gauge**, complying with the relevant requirements of ISO 23529.
- **5.4 Compression-testing machine**, complying with the requirements of ISO 5893^[4], equipped with means of autographic recording of the force-deformation relationship to an accuracy corresponding to grade 1 in respect of force.

When testing standard test pieces in methods A, B and C and larger test pieces in method D, it shall be possible to determine the displacement with an accuracy of ± 0.02 mm, including corrections for load cell and device stiffness.

When testing products with a height less than that of the standard test piece, it shall be possible to determine the displacement with an accuracy of ± 0.2 % of the height of the test piece, including corrections for load cell and device stiffness.

The machine shall be fitted with parallel compression platens at least as large as the metal plates (5.1), and shall be capable of operating at a speed of (10 \pm 2) mm/min.

NOTE 1 For methods A and D, the compression platens can be used directly without the metal plates, provided they have the required surface finish.

NOTE 2 For method C, the compression platens can be used directly, whatever the surface finish.

Machines with *y*-time recorders can give erroneous results because of:

- inertia effects;
- deformation caused by compliance in the load cell or machine frame.

Machines with x-y recorders are therefore preferred.

When testing lubricated test pieces, a suitable guard should be provided to avoid damage or injury should the rubber be ejected when strained.

5.5 Lubricant, having no significant effect on the rubber under test, for methods A, C and D.

NOTE For most purposes, a silicone or fluorosilicone fluid having a kinematic viscosity of 0,01 m²/s is suitable.

For method C, lubrication is recommended though it is not necessary (see Annex A).

6 Calibration

The test apparatus shall be calibrated in accordance with Annex C.

7 Test pieces

Standard test piece A: the standard test piece for both method A and method B is a cylinder of diameter (29 ± 0.5) mm and height (12.5 ± 0.5) mm.

Standard test piece B: the standard test piece for method C is a cylinder of diameter (17,8 \pm 0,15) mm and height (25 \pm 0,25) mm.

Test pieces can be cut or moulded. Cut test pieces shall be prepared in accordance with ISO 23529.

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Other test pieces can be used, but extrapolation of the results might not be possible (see Annex B).

For method B, test pieces can be directly moulded to the metal plates using a suitable mould and bonding system or adhered to the plates using suitable non-solvent adhesive systems.

It is essential to have test pieces with flat and parallel surfaces.

For method D, the test piece is a product, or a part of a product, or multiples thereof. For profiles, a length of 50 mm to 100 mm shall be used as the test piece (or two such lengths together if it is necessary to increase the force reading). For ring-shaped products with an inner diameter of 50 mm to 100 mm, the whole product shall be used. For small products, two or more products can be tested side by side, parallel to each other, to increase the force reading.

8 Number of test pieces

At least three test pieces, or sets of test pieces, shall be tested.

9 Time-lapse between vulcanization and testing

Unless otherwise specified for technical reasons, the following requirements shall be observed (see ISO 23529).

- For all test purposes, the minimum time between vulcanization and testing shall be 16 h.
- For non-product tests, the maximum time between vulcanization and testing shall be four weeks and, for evaluations intended to be comparable, the tests, as far as possible, shall be carried out after the same time interval.
- For product tests, whenever possible, the time between vulcanization and testing shall not exceed three
 months. In other cases, tests shall be made within two months of the date of receipt of the product by the
 customer.

10 Conditioning

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

Samples, after any necessary preparation, shall be conditioned at standard laboratory temperature (see ISO 23529) for at least 3 h before the test pieces are cut. The test pieces can be marked, if necessary, and measured and tested immediately. If not tested immediately, they shall be kept at the standard laboratory temperature until tested. If the preparation involves buffing, the interval between buffing and testing shall not exceed 72 h.

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

If the test is to be carried out at a temperature other than 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).

11 Temperature of test

The test shall normally be carried out at standard laboratory temperature (see ISO 23529). If another temperature is used, it shall preferably be one of the following:

$(-75\pm2)~^{\circ}\mathrm{C},$	$(-55\pm2)~^{\circ}\text{C},$	(-40 ± 2) °C,	(-25 ± 2) °C,	(-10 ± 2) °C,	$(0\pm2)~^{\circ}C,$
(40 ± 1) °C,	(55 ± 1) °C,	(70 ± 1) °C,	(85 ± 1) °C,	(100 ± 1) °C,	
$(125 \pm 2) ^{\circ}\text{C},$	$(150 \pm 2) ^{\circ}\text{C}$	$(175 \pm 2) ^{\circ}\text{C}$	$(200 \pm 2) ^{\circ}\text{C}$	$(225 \pm 2) ^{\circ}\text{C}$	$(250 \pm 2) ^{\circ}\text{C}.$

12 Procedure

12.1 Measurement of test pieces

Determine the dimensions of the test pieces by the appropriate methods specified in ISO 23529. For test pieces bonded by vulcanization, measure the thickness of the bonded assembly and determine the thickness of the rubber by subtracting the sum of the thicknesses of the metal plates from the thickness of the bonded assembly.

12.2 Determination of stress-strain properties

12.2.1 Method A

For lubricated test pieces, lightly coat the polished surfaces of the metal plates with a film of lubricant.

Insert the test piece centrally in the compression machine between the metal plates and operate the machine at a speed of 10 mm/min until a strain of 25 % is reached. Release the strain at the same speed of 10 mm/min and repeat the compression and release cycle three more times, the four compression cycles forming an uninterrupted sequence. Record the force-deformation curve.

12.2.2 Method B

Insert the bonded assembly centrally in the compression machine and operate the machine at a speed of 10 mm/min until a strain of 25 % is reached. Release the strain at the same speed of 10 mm/min and repeat the compression and release cycle three more times, the four compression cycles forming an uninterrupted sequence. Record the force-deformation curve.

12.2.3 Method C

Insert the assembly (lubricated or not) centrally in the compression machine and operate the machine at a speed of 10 mm/min until a strain of 25 % is reached. Release the strain at the same speed of 10 mm/min and repeat the compression and release cycle three more times, the four compression cycles forming an uninterrupted sequence. Record the force-deformation curve.

12.2.4 Method D

Place the test piece centrally on the lower lubricated compression platen. Compress the test piece at a speed of 10 mm/min until a strain of 30 % is reached and record the force-deformation curve.

This test is normally done without any mechanical conditioning. Mechanical conditioning as in methods A, B or C can also be used, but its use shall be mentioned in the test report.

Holes are needed in the compression platens when testing ring-shaped products, to let the air out during compression.

If a product includes bonded-on rigid components (e.g. an engine mount), it is tested without lubricated platens.

13 Expression of results

13.1 For methods A, B and C

The results shall be derived from the recorded force-deformation diagrams (see Figure 1) and shall be expressed in megapascals as the compression modulus at 10 % and 20 % strain, the strain being measured from the point at which the curve in the last cycle meets the strain (deformation) axis. Determine the stress-strain properties from the force-deformation measurements obtained during the compression part of the last cycle. Report the median and individual values at 10 % and 20 % compression strain for all the test pieces.

The compression modulus is given, in megapascals, by the formula

$$\frac{F}{A\varepsilon}$$

which is equal to

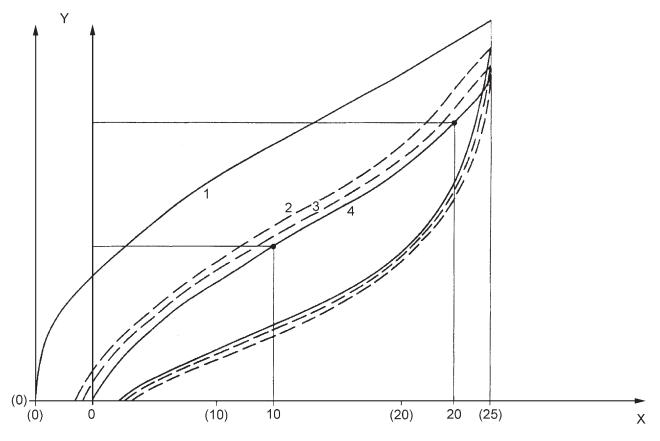
$$\frac{F_{0,1}}{A\varepsilon_{0,1}}$$

for the compression modulus at 10 % strain and

$$\frac{F_{0,2}}{A\varepsilon_{0,2}}$$

for the compression modulus at 20 % strain, where

- *F* is the force, in newtons, applied to produce the compression strain;
- A is the original cross-sectional area, in square millimetres, of the test piece;
- ε is the compression strain.



Key

X deformation, in %

Y force, F

1,2,3,4 compression cycles 1,2,3,4

Figure 1 — Calculation of compression modulus

13.2 For method D

The results shall be derived from the recorded force-deformation diagrams and shall be expressed in newtons per metre or in newtons. Read the values from the force-deformation curves at 25 % compression strain and calculate the stiffness at 25 % compression, S_{25} , from the equation:

$$S_{25} = \frac{F_{25}}{L}$$

or from the equation:

$$S_{25} = F_{25}$$

where

 F_{25} is the force, in newtons, at 25 % compression strain;

L is the length, in metres.

In the case of ring-shaped products, the length is taken along the average circumference, i.e. along the circle midway between the inner and outer surfaces of the ring.

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Report the median value for the test pieces tested, and the individual values.

NOTE Strains other than 25 % can be required by a product specification.

14 Test report

The test report shall include the following:

- a) sample details:
 - 1) full description of the sample and its origin,
 - 2) compound details and cure details, where appropriate,
 - 3) method of preparation of test piece from the sample, for example moulded or cut;
- a) test method:
 - 1) full reference to the test method used, i.e. the number of this International Standard (ISO 7743:2011),
 - 2) test procedure used (A, B,C or D),
 - 3) type of test piece used;
- b) test details:
 - 1) laboratory temperature,
 - 2) time and temperature of conditioning prior to test,
 - 3) temperature of test, if other than standard laboratory temperature and relative humidity, if necessary,
 - 4) type of lubrication or bonding agent used,
 - 5) details of any procedures not specified in this International Standard;
- c) test results:
 - 1) number of test pieces used,
 - 2) individual test results,
 - 3) median results, expressed in megapascals, of the compression modulus at 10 % and 20 % strain for methods A, B and C, and expressed in newtons per metre or newtons at 25 % strain for method D;
- d) date of test.

15 Precision for methods A and D

See Annex D.

Annex A (informative)

Influence of test piece geometry

The static or dynamic mechanical characterization of elastomeric materials involves well-defined loading conditions. It requires a test piece geometry which allows well-defined stress and strain fields to be maintained as uniformly as possible throughout the test.

In the case of a compression test piece, it is necessary to maximize the uniaxial stress component and to avoid shear and/or biaxial components. Ideally, a perfect compression test piece is a long cylinder with a small cross-section. Practically, such a test piece is not suitable for compression because of buckling. A series of tests performed on test pieces with various slenderness ratios together with finite element computations show that a uniaxial stress state can be created and preserved over a wide range of deformation when the slenderness ratio (length-to-diameter ratio) is greater than or equal to 1. If the test piece geometry is too flat, a correction factor is required to derive the compression properties from the test results.

NOTE Slenderness ratio is inversely related to shape factor (see Annex B).

Compression tests were made on an SBR compound filled with 60 phr of HAF N 330 carbon black. Four geometries of cylindical test pieces were considered.

- Test piece 1: diameter: 8 mm length: 14 mm (l/d = 1,75)
- Test piece 2: diameter: 18 mm length: 25 mm (l/d = 1,56) ISO 4666-3^[3]
- Test piece 3: diameter: 20 mm length: 20 mm (l/d = 1,00)
- Test piece 4: diameter: 29 mm length: 12,5 mm (l/d = 0,43) ISO $815-1^{[1]}$

The results are displayed in Figures A.1 and A.2; each curve presented is the mean of results obtained on three test pieces.

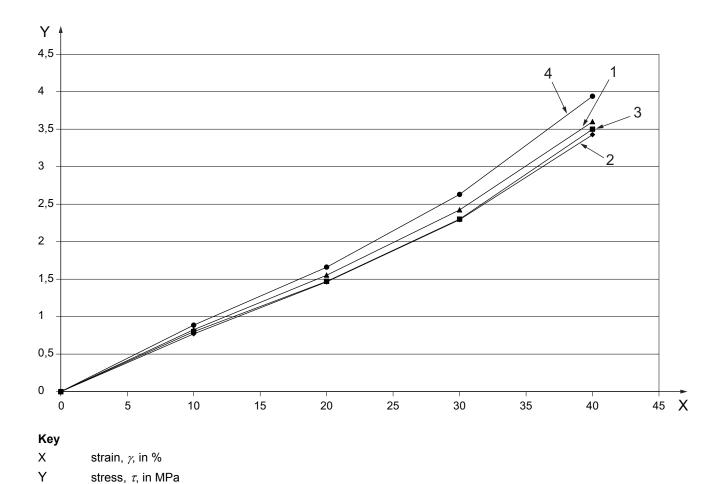


Figure A.1 — Static stress-strain properties in compression — Lubricated test pieces — Stresses presented without any correction

1,2,3,4

test pieces 1,2,3,4

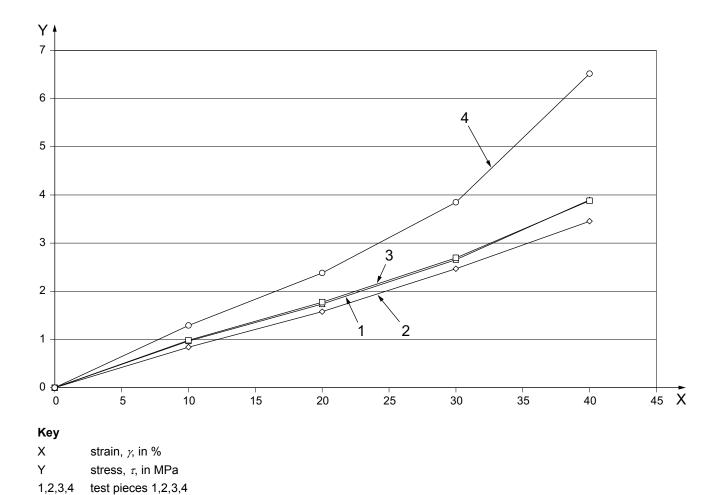


Figure A.2 — Static stress-strain properties in compression — Bonded test pieces — Stresses presented without any correction

The test piece geometry has little influence on the loading curve as long as the compression platens are well lubricated. However, if the test pieces are bonded, the effective stiffness increases when the slenderness ratio decreases. The results obtained show that the difference is particularly significant for the compression set test piece, test piece 4 (which was adopted as test piece A, as specified in Clause 7).

When intrinsic characteristics of a rubber are to be determined from a compression test, it is better to choose a test piece with a suitable slenderness ratio (h/d > 1). Test piece 2 was finally chosen to become test piece B because it is already used in International Standard methods.

A behaviour model was determined from mechanical tests conducted on the SBR compound mentioned in paragraph 3.

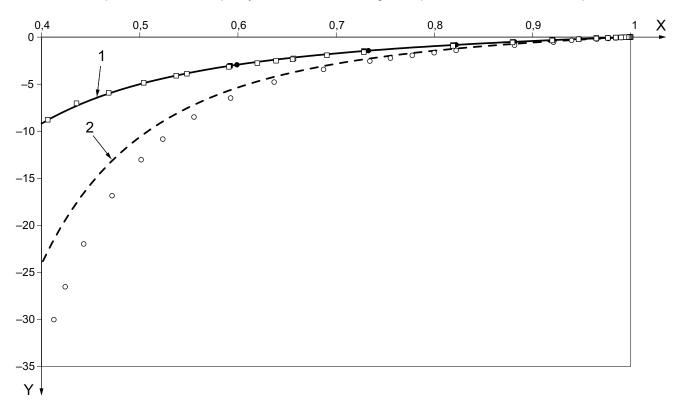
The Rivlin model so defined was used to derive the material behaviour in uniaxial compression and biaxial compression (pure shear in compression). The curves obtained are reported on Figure A.3. The results of several finite element computations are also plotted in Figure A.3. Those computations are:

- compression of test piece A without friction;
- compression of test piece A bonded;
- compression of test piece B without friction;
- compression of test piece B bonded.

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Figure A.3 shows that test piece B gives the required uniaxial compression result, whatever the friction level at the interface between the test piece and the compression platens. The response of test piece A to compression is highly dependent on the level of friction and varies between uniaxial and biaxial compression.

Moreover, test piece B allows the quality of the measure at high compression deformation to be preserved.



Key

- X stretch ratio
- Y stress, τ , in MPa
- 1 uniaxial compression behaviour
- 2 biaxial compression behaviour (pure shear in compression)
- test piece A, no friction
- o test piece A, bonded
- test piece B, no friction
- test piece B, bonded

Figure A.3 — Influence of the test piece shape on the mechanical response in compression

Annex B

(informative)

Extrapolation of results to non-standard test pieces

As shown in Annex A, the effects of shape factor and degree of slip at the compressed faces on the compression stress-strain properties of rubber are very complex and, normally, test results should be regarded as uniquely applicable to the specific shape of test piece and conditions used in the test.

However, this annex is intended to give some indication of the factors to be considered should any attempt be made to compare results obtained on different test pieces or to extrapolate from test pieces to products. It is emphasized that the relationships given are approximate and that any extrapolation of results using them should be confirmed by experimental means.

The following symbols are used throughout this annex:

- d diameter
- E Young modulus
- E_c effective compression modulus
- e thickness
- G shear modulus
- K bulk modulus
- k a factor depending on hardness^{[6][7]}
- S shape factor
- ε compression strain
- λ compression ratio ($\lambda = 1 \varepsilon$)
- σ average compression stress based on the original cross-section

Rubbers have a very high bulk modulus compared to their shear modulus and, for most purposes, can be regarded as incompressible.

Thus

$$E = 3G$$

Under lubricated conditions, assuming complete slip, the compression of test pieces A (method A) is homogeneous and the stress-strain relationship predicted by Gaussian theory is applicable:

$$\sigma = G\left(\lambda^{-2} - \lambda\right) = \frac{E\left(\lambda^{-2} - \lambda\right)}{3} \tag{B.1}$$

Since $\lambda = 1 - \varepsilon$, substitute for λ :

$$\sigma = \frac{E}{3} \left[\frac{1}{(1-\varepsilon)^2} - (1-\varepsilon) \right]$$

$$\sigma = \frac{E}{3} \left[\frac{1 - (1 - \varepsilon)^3}{(1 - \varepsilon)^2} \right]$$

$$\sigma = \frac{E}{3} \left[\frac{1 - 1 + 3\varepsilon - 3\varepsilon^2 + \varepsilon^3}{\left(1 - \varepsilon\right)^2} \right]$$

If the epsilon cubed term is ignored, this reduces to:

$$\sigma = \frac{E}{3} \left[\frac{3\varepsilon - 3\varepsilon^2}{\left(1 - \varepsilon\right)^2} \right] = E\varepsilon \left[\frac{1 - \varepsilon}{\left(1 - \varepsilon\right)^2} \right] = \frac{E\varepsilon}{1 - \varepsilon}$$
(B.2)

This approximation is satisfactory for strains up to about 30 %.

For very small strains, $1 - \varepsilon \approx 1$, Equation (B.2) reduces to:

$$\sigma = E\varepsilon$$
 (B.3)

This approximation is satisfactory for strains up to about 5 %.

In the bonded condition (test piece A, method B), non-uniform distribution of shear strain arises from the constraints at the bonded surfaces, and the compression behaviour becomes dependent on the shape and the hardness of the material.

To derive the Young modulus from the effective compression modulus, the literature [6][7] proposes Equation (B.4):

$$E_{\rm c} = E(A + BS^n) \tag{B.4}$$

where

S is the shape factor, i.e. the ratio of the area to which the force is applied to the force-free area, e.g. for a disc: S = d/4e;

A = 1 and B = 2k for discs;

 $1,0 \le A \le 1,3$ and $1,3 \le B \le 2,2$ for rectangles depending on hardness.

NOTE 1 In the case of natural rubber, n = 2.

NOTE 2 The value of E_c derived from Equation (B.4) can be substituted for E in Equation (B.1), (B.2) or (B.3), as appropriate, depending on the level of strain.

At very high strains, or when S becomes large, it can prove necessary to take into account the bulk modulus. An approximation is:

$$\frac{1}{E_{\mathsf{c}}} = \frac{1}{E\left(A + BS^n\right)} + \frac{1}{K} \tag{B.5}$$

A particular and simplified expression of the general equation with A = 1, B = 2 and n = 2 has been found to give accurate results even for high strains. It is convenient and very easy to use as it depends only on geometrical characteristics. It gives

$$E_c = E(1 + 2S^2)$$
 (B.6)

Rubber containing filler behaves non-linearly in shear and this can have a significant effect on the shape factor component of $E_{\rm c}$. The same applies in homogeneous compression.

When neither lubrication nor bonding is used, friction does not normally entirely prevent slip, and the extent to which slippage occurs is variable, depending on surface conditions, level of strain, etc. It can also be time dependent and can increase in the presence of vibration.

For design purposes, the Young modulus is of more value than the compression modulus (secant modulus). To determine the Young modulus from the experimental measurements at 10 % and 20 % strain, Equation (B.2) should be used, modified if required by Equation (B.4) or (B.6).

The compression modulus (secant modulus), $S_{\rm M}$, is then given by:

$$S_{\mathsf{M}} = \frac{\sigma}{\varepsilon}$$

for lubricated test pieces this is equal to

$$\frac{E}{1-\varepsilon}$$

for bonded test pieces this is equal to

$$\frac{E(A+BS^n)}{1-\varepsilon}$$

From these equations, the Young modulus is given, for lubricated test pieces, by

$$E = S_{M}(1 - \varepsilon)$$

and for bonded test pieces by

$$E = \frac{S_{\mathsf{M}} \left(1 - \varepsilon \right)}{A + BS^{n}}$$

The value to be reported is the median of E determined from the compression moduli (secant moduli) for 10 % and 20 % strain.

Annex C (normative)

Calibration schedule

C.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 carried out 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.

C.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. 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 C.1 has been compiled by listing all 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;
- S standard interval as given in ISO 18899;
- U in use.

Table C.1 — Calibration frequency schedule

Parameter	Requirement	Subclause in ISO 18899:2004	Verification frequency guide	Notes			
Compression testing machine	As per ISO 5893 ^[4] , with autographic recording of force-deformation curve	14.6	S	x-y recorders preferred to y-time recorders			
Accuracy, force	1 % of reading	21.2					
Accuracy, deformation	0,02 mm or 0,2 % of test piece height	15.4					
Speed	(10 ± 2) mm/min	23.4					
Compression platens	Parallel	С	S				
Size of platens	At least as large as the metal plates between them	С	U				
Metal plates:	Flat, of uniform thickness	С	U				
— for bonded test pieces	Lateral dimensions ≥ test piece dimensions	С	U				
for lubricated test pieces	At least 20 mm larger than test pieces	С	U				
Surface finish	ISO 4287 ^[2]	15.5	S				
— Method A	$Ra \leq 0.4 \mu m$						
— Method B	As required by bonding system						
Dies and cutters	ISO 23529	15.2	S				
Thickness gauge	As per ISO 23529	15.1	S				
Thermometer to monitor the conditioning and test temperatures ^a	As per ISO 23529	18	S	See Clause 10			
Lubricating fluids Suitable, having no significant effect on the rubber under test, e.g. silicone or fluorosilicone fluid							
a For tolerances of test temper	^a For tolerances of test temperature, see Clause 11.						

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

- a timer;
- a thermometer;
- an instrument for determining the dimensions of the test pieces.

Annex D

(informative)

Precision for methods A and D

D.1 Precision details

An interlaboratory test programme (ITP) for evaluating precision of compression modulus was conducted in 2008 using the precision procedures and guidelines described in ISO/TR 9272^[5].

See ISO/TR 9272^[5] for other details and terminology on precision evaluation.

Compression modulus measurement methods A and D were evaluated. A Type 1 precision was evaluated using cured test pieces prepared from nine different rubber-compound test piece combinations (with a range of values) supplied to each of the eight participating laboratories in the ITP. The nine compounds are designated as compounds A, B, C for Method A (for two different strain levels); D, E, F for Method D (profile) and G, H and I for method D (O-ring). On each of two test days, one week apart, the following test sequence was carried out. Three measurements for each compound test piece were conducted and a median of the three measurements was obtained. The median value is defined as a test result for that test day. All analysis was conducted on the basis of test result data (i.e. median of three for any day). The ISO/TR 9272^[5] Option 1 outlier treatment procedure, outlier deletion, was used.

The precision results as determined by this ITP may not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of this precision evaluation actually apply to the products or materials tested.

D.2 Precision results

- **D.2.1** General. The precision results for all methods and compounds for compression modulus are given in Table D.1. These results were obtained using the outlier deletion as noted above. General statements for the use of the precision results are cited below. These are given in terms of both the absolute precision, r or R, and also for relative precision (r) and (R).
- **D.2.2** Repeatability. The repeatability, or local domain precision, for each of the compression modulus test methods has been established for each compound or material and method by the values found in Table D.1. Two individual test results (obtained by the proper use of this International Standard) that differ by more than the tabulated values for r, in measurement units, and (r), as a percentage of the mean level, should be considered suspect, i.e. to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.
- **D.2.3 Reproducibility.** The reproducibility, or global domain precision, for each of the compression modulus test methods has been established for each compound or material and method by the values found in Table D.1. Two individual test results obtained in different laboratories (by the proper use of this International Standard) that differ by more than the tabulated values for R, in measurement units, and (R), as a percentage of the mean level, should be considered suspect, i.e. to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.

Table D.1 — Precision for compression modulus — Methods A (disc), D (profile) and D (O-ring)

Matarial		Within laboratory		Between laboratories			No.	
Material	Mean	S_{r}	r	(r)	s_R	R	(R)	laboratories ^b
	Part 1: Method A (disc) for modulus at 10 % strain, MPa							
Compound A	0,315	0,005 1	0,014 3	4,53	0,031 0	0,086 9	27,58	6
Compound B	0,489	0,008 7	0,024 2	4,96	0,500	0,140 0	28,63	6
Compound C	0,647	0,020 3	0,056 9	8,80	0,087 4	0,244 7	37,85	7
Pooled or average value ^a		0,011 4	0,031 8	6,10	0,156 2	0,157 2	31,35	
	Par	t 2: Method	d A (disc) fo	or modulus	at 20 % str	ain, MPa		
Compound A	0,641	0,006 6	0,046 4	7,25	0,040 3	0,112 8	17,60	6
Compound B	0,952	0,011 8	0,033 1	3,48	0,089 6	0,250 9	26,35	5
Compound C	1,348	0,033 3	0,093 2	6,91	0,247 0	0,691 7	51,32	7
Pooled or average value ^a		0,015 3	0,057 6	5,897	0,125 6	0,351 8	31,76	
		Part 3: Mo	ethod D (pr	ofile) at 25	% strain ^c , N	И Ра		
Compound D	2,76	0,078 0	0,218	7,93	0,415 2	1,163	42,20	3
Compound E	9,61	0,128 3	0,359	3,74	0,224 3	0,628	6,54	3
Compound F	3,00	0,031 4	0,088	2,93	0,232 2	0,650	21,66	3
Pooled or average value ^a		0,044 9	0,222	4,864	0,290 6	0,814	23,47	
Part 4: Method D (O-ring) at 25 % strain ^c , MPa								
Compound G	2,38	0,070 0	0,196 0	8,25	0,100 8	0,282 2	11,87	3
Compound H	3,57	0,091 8	0,257 1	7,21	0,398 1	1,114 6	31,23	3
Compound I	5,36	0,295 7	0,827 9	15,46	0,422 2	1,182 3	22,08	3
Pooled or average value ^a		0,152 5	0,427 0	10,304	0,307 0	0,859 7	21,73	

^a Simple averages calculated.

Notation used:

- s_r is the within-laboratory standard deviation, in measurement units
- r is the repeatability, in measurement units
- (r) is the repeatability, as percentage of mean level
- s_R is the between-laboratory standard deviation (for total between laboratory variation), in measurement units
- R is the reproducibility, in measurement units
- (R) is the reproducibility, as percentage of mean level

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b Number of laboratories after outliers deleted; final analysis; total laboratories = 8 for method A, 4 for method C.

^c The precision for Part 3 and Part 4 has been obtained for a very small number of laboratories; thus it is only a very approximate estimation of the true precision for these methods. Use it with extreme caution.

D.3 Additional analysis comments

The precision results for Method D, both profile and O-ring, are very approximate values since all eight laboratories did not participate in the testing. The results are based on only three laboratories. Such precision should be used with extreme caution.

D.4 Bias

Bias is the difference between a measured average test result and a reference or true value for the measurement in question. Reference values do not exist for this test method and therefore bias cannot be evaluated.

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- [1] ISO 815-1, Rubber, vulcanized or thermoplastic Determination of compression set Part 1: At ambient or elevated temperatures
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- [3] ISO 4666-3, Rubber, vulcanized Determination of temperature rise and resistance to fatigue in flexometer testing Part 3: Compression flexometer (constant-strain type)
- [4] ISO 5893, Rubber and plastics test equipment Tensile, flexural and compression types (constant rate of traverse) Specification
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