INTERNATIONAL STANDARD

ISO 37

Fifth edition
2011-12-15

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

Caoutchouc vulcanisé ou thermoplastique — Détermination des caractéristiques de contrainte-déformation en traction

Reference number
ISO 37:2011(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 37 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 37:2005), which has been technically revised. It also incorporates the Technical Corrigendum ISO 37:2005/Cor.1:2008.

The technical changes mainly concern the testing of ring test pieces, which is now described in more detail.

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

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.

IMPORTANT — Certain procedures specified in this International Standard 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 International Standard describes a method for the determination of the tensile stress-strain properties of vulcanized and thermoplastic rubbers.

The properties which can be determined are tensile strength, elongation at break, stress at a given elongation, elongation at a given stress, stress at yield and elongation at yield. The measurement of stress and strain at yield applies only to some thermoplastic rubbers and certain other compounds.

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 5893, *Rubber and plastics test equipment — Tensile, flexural and compression types (constant rate of traverse) — Specification*

ISO 23529:2010, *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
tensile stress

S

stress applied so as to extend the test piece

NOTE It is calculated as the applied force per unit area of the original cross-section of the test length.

3.2
elongation

E

tensile strain, expressed as a percentage of the test length, produced in the test piece by a tensile stress

3.3
tensile strength

TS

maximum tensile stress recorded in extending the test piece to breaking point

NOTE See Figures 1a) to 1c).

3.4
tensile strength at break

$\mathbf{TS_b}$

tensile stress recorded at the moment of rupture

NOTE 1 See Figures 1a) to 1c).

NOTE 2 The values of TS and $\mathrm{TS_b}$ might be different if, after yield at S_y, the elongation continues and is accompanied by a drop in stress, resulting in $\mathrm{TS_b}$ being lower than TS [see Figure 1c)].

3.5
elongation at break

E_b

tensile strain in the test length at breaking point

NOTE See Figures 1a) to 1c).

3.6
elongation at a given stress

E_s

tensile strain in the test length when the test piece is subjected to a given tensile stress

3.7
stress at a given elongation

S_e

tensile stress in the test length required to produce a given elongation

NOTE In the rubber industry, this definition is widely identified with the term "modulus" and care should be taken to avoid confusion with the other use of "modulus" to denote the slope of the stress-strain curve at a given elongation.

3.8
tensile stress at yield

S_y

tensile stress at the first point on the stress-strain curve where some further increase in strain occurs without any increase in stress

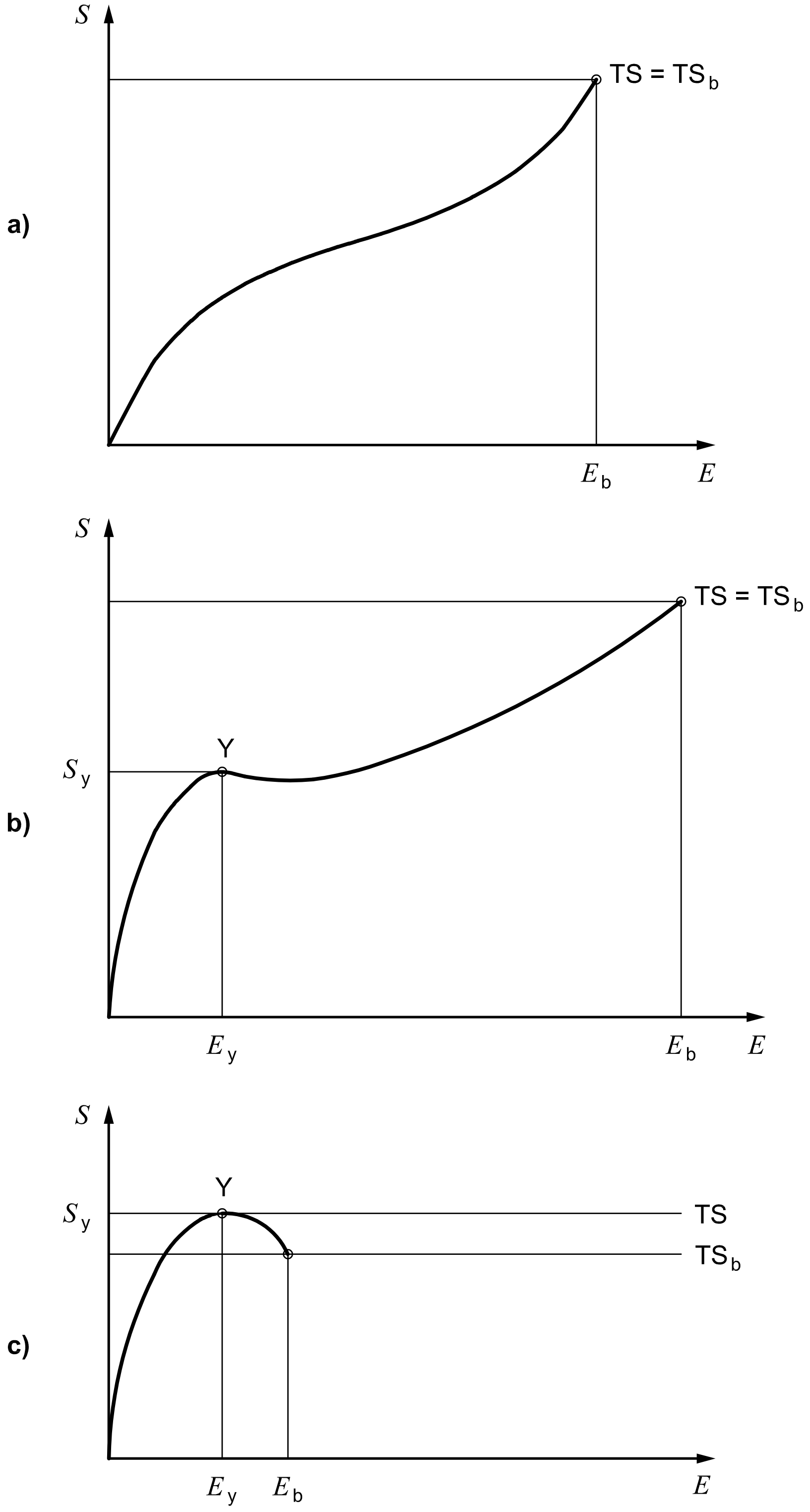
NOTE This might correspond either to a point of inflection [see Figure 1b)] or to a maximum [see Figure 1c)].

3.9
elongation at yield

E_y

tensile strain at the first point on the stress-strain curve where some further increase in strain is not accompanied by an increase in stress

NOTE See Figures 1b) and 1c).



Key

E	elongation	S_y	stress at yield
E_b	elongation at break	TS	tensile strength
E_y	elongation at yield	TS_b	tensile strength at break
S	stress	Y	yield point

Figure 1 — Illustration of tensile terms

3.10
test length of a dumb-bell
initial distance between reference points within the length of the narrow portion of a dumb-bell test piece used to measure elongation

NOTE See Figure 2.

4 Principle

Standard test pieces, either dumb-bells or rings, are stretched in a tensile-testing machine at a constant rate of traverse of the driven grip or pulley. Readings of force and elongation are taken as required during the uninterrupted stretching of the test piece and when it breaks.

5 General

Dumb-bell and ring test pieces do not necessarily give the same values for their respective stress-strain properties. This is mainly because in stretched rings the stress is not uniform over the cross-section. A second factor is in the existence of “grain” which might cause dumb-bells to give different values depending on whether their length is parallel or at right angles to the grain.

The main points to be noted in choosing between rings and dumb-bells are as follows:

a) Tensile strength

Dumb-bells are preferable for determination of tensile strength. Rings give lower, sometimes much lower, values than dumb-bells.

b) Elongation at break

Rings give approximately the same values as dumb-bells, provided that

1) the elongation of rings is calculated as a percentage of the initial internal circumference and

2) dumb-bells are cut at right angles to the grain if this is present to a significant degree.

Dumb-bells shall be used if it is required to study grain effects, as rings are not suitable for this purpose.

c) Elongation at a given stress and stress at a given elongation

The larger dumb-bells (types 1, 2 and 1A) are generally preferred.

Rings and dumb-bells give approximately the same values provided that

1) the elongation of rings is calculated as a percentage of the initial mean circumference and

2) the average value is taken for dumb-bells cut parallel and at right angles to the grain if this is present to a significant degree.

Rings might be preferred in automated testing, due to the ease of handling of the test pieces, and in the determination of stress at a given strain.

6 Test pieces

6.1 General

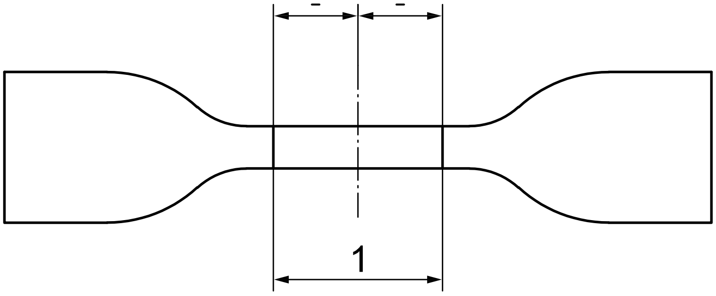
Miniature test pieces might give somewhat different, usually higher, values for tensile strength and elongation at break than the larger test pieces.

Seven types of test piece are provided, i.e. dumb-bell-shaped types 1, 2, 3, 4 and 1A and ring-shaped types A (normal) and B (miniature). The results obtained for a given material are likely to vary according to the type of test piece used, and the results obtained for different materials should therefore not be regarded as comparable unless the same type of test piece has been used.

When preparation of test pieces requires buffing or thickness adjustment, results might be affected.

6.2 Dumb-bells

Dumb-bell test pieces shall have the outline shown in Figure 2.



Key

1 test length (see Table 1)

Figure 2 — Shape of dumb-bell test pieces

The standard thickness of the narrow portion shall be 2,0 mm ± 0,2 mm for types 1, 2, 3 and 1A and 1,0 mm ± 0,1 mm for type 4.

The test length shall be in accordance with Table 1.

The other dimensions of the dumb-bells shall be as produced by the appropriate die (see Table 2).

For non-standard test pieces, e.g. those taken from finished products, the maximum thickness of the narrow portion shall be 3,0 mm for types 1 and 1A, 2,5 mm for types 2 and 3, and 2,0 mm for type 4.

Table 1 — Test length of dumb-bells

Type of test piece	Type 1	Type 1A	Type 2	Type 3	Type 4
Test length (mm)	25 ± 0,5	20 ± 0,5[a]	20 ± 0,5	10 ± 0,5	10 ± 0,5

[a] The test length shall not exceed the length of the narrow portion of the test piece (dimension C in Table 2).

Type 3 and 4 dumb-bell test pieces shall only be used where insufficient material is available for the larger test pieces. These test pieces are particularly suitable for testing products and are used in certain product standards, e.g. type 3 dumb-bells have been used for testing pipe sealing rings and cable coverings.

6.3 Rings

The standard type A ring test piece shall have an internal diameter of 44,6 mm ± 0,2 mm. The median axial thickness and median radial width shall be 4 mm ± 0,2 mm. The radial width of any ring shall nowhere deviate from the median by more than 0,2 mm and the axial thickness of the ring shall nowhere deviate from the median by more than 2 %.

The standard type B ring test piece shall have an internal diameter of 8 mm ± 0,1 mm. The median axial thickness and median radial width shall be 1 mm ± 0,1 mm. The radial width of any ring shall nowhere deviate from the median by more than 0,1 mm. This test piece shall be used only where insufficient material is available for the larger type A test piece.

7 Apparatus

7.1 Dies and cutters

All dies and cutters used shall be in accordance with ISO 23529. Dies for preparation of dumb-bells shall have the dimensions given in Table 2 and Figure 3 except for the cutting edge for which Figure 3 only indicates a suitable geometry. The departure from parallelism at any point along the width of the narrow portion of the die shall nowhere exceed 0,05 mm.

For a method of cutting type B ring test pieces, see Annex A.

Table 2 — Dimensions of dies for dumb-bell test pieces

Dimension		Type 1	Type 1A	Type 2	Type 3	Type 4
A	Overall length (minimum)[a] (mm)	115	100	75	50	35
B	Width of ends (mm)	25 ± 1	25 ± 1	12,5 ± 1	8,5 ± 0,5	6 ± 0,5
C	Length of narrow portion (mm)	33 ± 2	21 ± 1	25 ± 1	16 ± 1	12 ± 0,5
D	Width of narrow portion (mm)	6,2 ± 0,2	5 ± 0,1	4 ± 0,1	4 ± 0,1	2 ± 0,1
E	Transition radius outside (mm)	14 ± 1	11 ± 1	8 ± 0,5	7,5 ± 0,5	3 ± 0,1
F	Transition radius inside (mm)	25 ± 2	25 ± 2	12,5 ± 1	10 ± 0,5	3 ± 0,1

[a] A greater overall length might be necessary to ensure that only the wide end tabs come into contact with the machine grips, thus avoiding “shoulder breaks”.

7.2 Thickness gauge

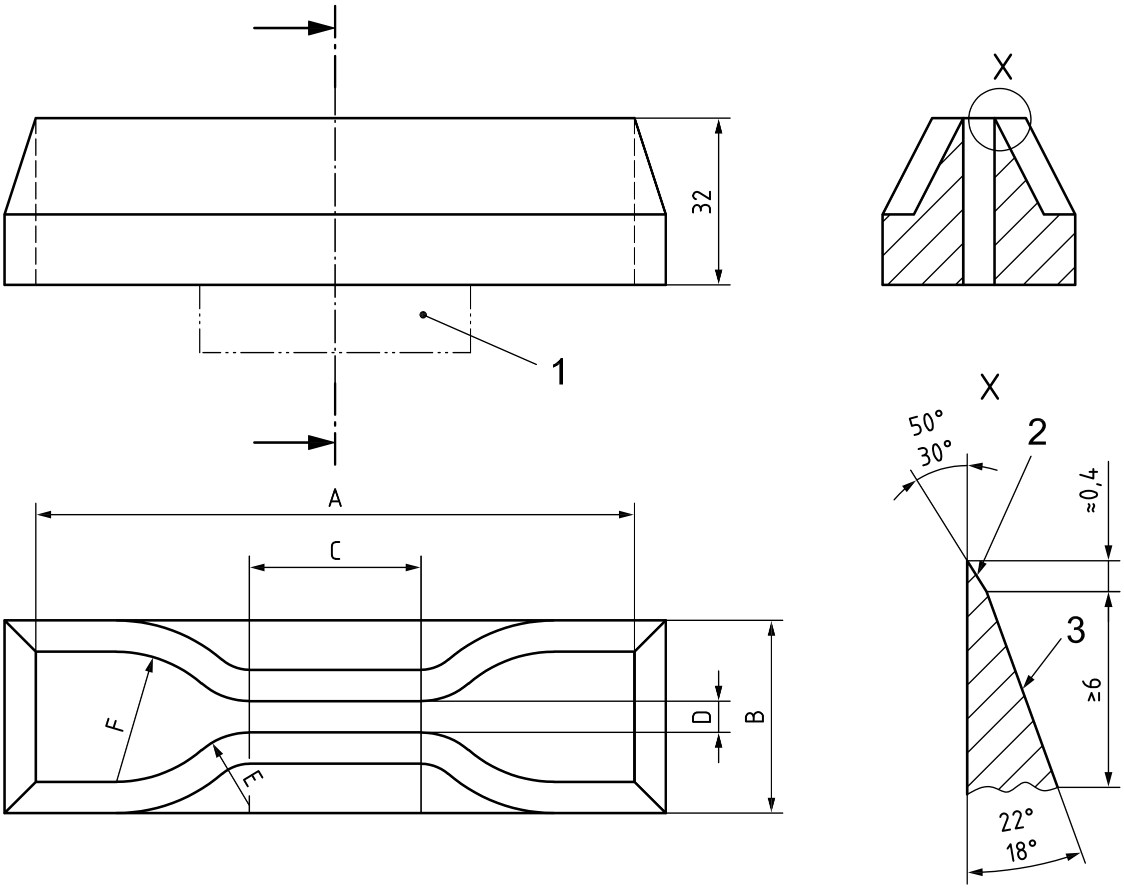
The instrument for measuring the thickness of dumb-bell test pieces and the axial thickness of ring test pieces shall be in accordance with that used in method A of ISO 23529:2010.

The instrument for measuring the radial width of ring test pieces shall be similar to the above, except that the contact and base plate shall be shaped to fit the curvature of the ring.

7.3 Cone gauge

A calibrated cone gauge or other suitable equipment shall be used to measure the internal diameter of ring test pieces. The equipment shall be capable of measuring the diameter with an error of not more than 0,01 mm. The means of supporting the ring test piece to be measured shall be such as to avoid any significant change in the dimension being measured.

Dimensions in millimetres



Key

1 method of fixing to suit machine

2 ground smooth

3 ground

NOTE 1 For dimensions A to F, see Table 2.

NOTE 2 The diagrams on the right show sections of typical fixed blades.

Figure 3 — Die for dumb-bell test pieces

7.4 Tensile-testing machine

7.4.1 The tensile-testing machine shall comply with the requirements of ISO 5893, having an accuracy of force measurement complying with class 2. An extensometer, where used, shall have an accuracy complying with class D for type 1, 1A and 2 dumb-bell test pieces and class E for type 3 and 4 dumb-bell test pieces. The machine shall, as a minimum, be capable of operating at rates of traverse of 100 mm/min, 200 mm/min and 500 mm/min.

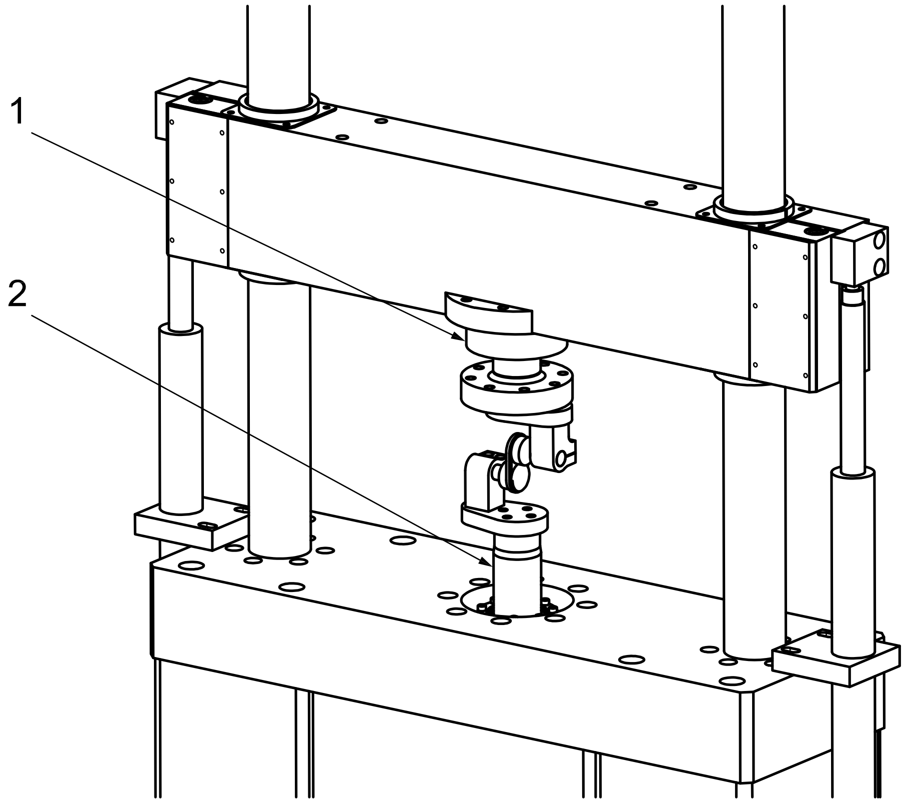
When testing dumb-bells, the method of measuring the extension might require the test machine to apply a small prestress to the test piece to avoid it bending. In this case, the machine shall be capable of applying the necessary prestress.

7.4.2 For tests at temperatures other than a standard laboratory temperature, a suitable thermostatically controlled chamber shall be fitted to the tensile-testing machine. Guidance for achieving elevated or subnormal temperatures is given in ISO 23529.

7.5 Test rig for ring test pieces

An example of a test rig using pulleys for testing rings is shown in Figure 4. For rings of types A and B, the pulley dimensions shall be as specified in Table 3 and Figure 5.

One of the pulleys shall be free to turn with very low friction and the other shall be driven to rotate the ring. It shall run at a speed between 10 rpm and 15 rpm.



Key

1 load cell

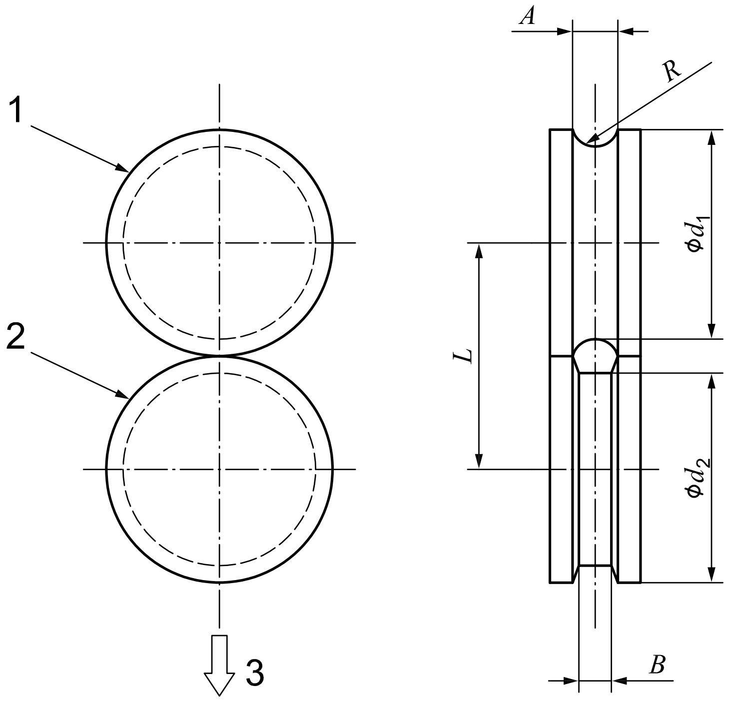
2 actuator

Figure 4 — Example of rig for tensile tests on rings

Table 3 — Pulley dimensions

Dimensions in millimetres

Pulleys	L	$\varnothing\, d_1$	A	R	$\varnothing\, d_2$	B
Standard pulleys for type A rings	$30^{+0,5}_{0}$	25 ± 0,05	6,0	3,0	25 ± 0,05	4,3
Alternative pulleys for type A rings	$35^{+0,5}_{0}$	22,3	—	—	22,3	5,0
Standard pulles for type B rings	$5,3^{+0,2}_{0}$	4,50 ± 0,02	1,5	0,75	4,50 ± 0,02	1,0



Key

1 freely rotating pulley

2 fixed pulley

3 direction of extension

Figure 5 — Pulley arrangement for tensile testing of type A and B rings

8 Number of test pieces

A minimum of three test pieces shall be tested.

The number of test pieces should preferably be decided in advance, bearing in mind that the use of five test pieces will give a lower uncertainty than a test with three test pieces.

9 Preparation of test pieces

9.1 Dumb-bells

Dumb-bell test pieces shall be prepared by the appropriate methods described in ISO 23529. Dumb-bells shall, wherever possible, be cut parallel to the grain of the material unless grain effects are to be studied, in which case a set of dumb-bells shall also be cut perpendicular to the grain.

9.2 Rings

Ring test pieces shall be prepared by cutting or punching, using the appropriate methods described in ISO 23529, or by moulding.

10 Conditioning of sample and test pieces

10.1 Time between vulcanization and testing

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 4 weeks and, for evaluations intended to be comparable, the tests shall, as far as possible, be carried out after the same time interval.

For product tests, whenever possible, the time between vulcanization and testing shall not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt of the product by the customer.

10.2 Protection of samples and test pieces

Samples and test pieces shall be protected as completely as possible from all external influences likely to cause damage during the interval between vulcanization and testing, e.g. they shall be protected from light and heat.

10.3 Conditioning of samples

Condition all samples, other than those from latex, in accordance with ISO 23529 at a standard laboratory temperature, without humidity control, for not less than 3 h prior to cutting out the test pieces.

Condition all prepared latex samples in accordance with ISO 23529 at a standard laboratory temperature, with humidity control, for not less than 96 h prior to cutting out the test pieces.

10.4 Conditioning of test pieces

Condition all test pieces in accordance with ISO 23529. If the preparation of test pieces involves buffing, the interval between buffing and testing shall be not less than 16 h and not greater than 72 h.

For tests at a standard laboratory temperature, test pieces that do not require further preparation may be tested immediately, if cut from conditioned test samples. Where additional preparation is involved, a minimum conditioning period of 3 h at standard laboratory temperature shall be allowed.

For tests at temperatures other than a standard laboratory temperature, condition the test pieces at the temperature at which the test is to be conducted for a period sufficient to enable the test pieces to attain substantial equilibrium in accordance with ISO 23529 (see also 7.4.2).

11 Marking of dumb-bell test pieces

If using a non-contact extensometer, mark the dumb-bell test pieces with two reference marks to define the test length as specified in Table 1, using a suitable marker. The test piece shall be unstrained when it is marked.

The lines shall be marked on the narrow part of the test piece, as shown in Figure 2, i.e. equidistant from the centre of the test piece and at right angles to its longitudinal axis.

12 Measurement of test pieces

12.1 Dumb-bells

Measure the thickness at the centre and at each end of the test length with the thickness gauge. Use the median value of the three measurements to calculate the area of the cross-section. In any one dumb-bell, none of the three thickness measurements of the narrow portion shall differ by more than 2 % from the median thickness. The width of the test piece shall be taken as the distance between the cutting edges of the die in the narrow part, and this distance shall be measured in accordance with ISO 23529 to the nearest 0,05 mm. Test pieces cut from products might have a non-rectangular cross-section, in which case the width shall be measured directly on the test piece.

12.2 Rings

Measure the radial width and axial thickness at six approximately equally spaced positions around the ring. The median value of each set of measurements shall be used in calculating the area of the cross-section. The internal diameter shall be measured to the nearest 0,1 mm. Calculate the internal circumference and the mean circumference as follows:

$$\text{Internal circumference} = \pi \times \text{internal diameter}$$

$$\text{Mean circumference} = \pi \times (\text{internal diameter} + \text{radial width})$$

12.3 Comparison of groups of test pieces

If two groups of test pieces (either dumb-bells or rings) are being compared, the median thickness for each group shall be within 7,5 % of the grand median thickness for the two groups.

13 Procedure

13.1 Dumb-bell test pieces

Insert the test piece into the tensile-testing machine, ensuring that the end tabs are gripped symmetrically so that the tension is distributed uniformly over the cross-section. It is strongly recommended that the load cell be reset to zero before each test. If necessary, apply a prestress of 0,1 MPa so that the test piece is not bent when the initial test length (see Figure 2) is measured. If necessary, set up the extensometry device. Start the machine and monitor continuously the change in test length and force throughout the test to an accuracy of ± 2 % or as required for the purposes of Clause 15.

The nominal rate of traverse of the moving grip shall be 500 mm/min for type 1, type 1A and type 2 test pieces and 200 mm/min for type 3 and type 4 test pieces.

Any test piece that breaks outside the narrow portion or yields outside the test length shall be discarded and a repeat test conducted on an additional test piece.

NOTE In making visual measurements, care should be taken to avoid inaccuracies due to parallax.

13.2 Ring test pieces

Set the correct initial distance between the pulleys, as follows:

— $30^{+0,5}_{\ \ 0}$ mm between the centres of the standard pulleys for type A rings;

— $35^{+0,5}_{\ \ 0}$ mm between the centres of the alternative pulleys for type A rings;

— $5{,}3^{+0,2}_{\ \ 0}$ mm between the centres of the standard pulleys for type B rings.

Set the load to zero before mounting the test piece on the pulleys.

Place the test piece on the pulleys and start the machine, monitoring continuously the distance between the pulleys and the increase in stress throughout the test.

The nominal rate of traverse of the moving pulley shall be 500 mm/min for type A test pieces and 100 mm/min for type B test pieces. The driven pulley shall rotate at the correct speed from the start to the end of the test.

14 Temperature of test

The test shall normally be carried out at one of the standard laboratory temperatures specified in ISO 23529. When other temperatures are required, these shall be selected from the list of preferred temperatures given in ISO 23529.

The same temperature shall be used throughout any one test or series of tests intended to be comparable.

15 Calculation of results

15.1 Dumb-bells

Calculate the tensile strength, TS, expressed in megapascals, using the equation

$$\mathrm{TS} = \frac{F_\mathrm{m}}{Wt}$$

Calculate the tensile strength at break, TS_b, expressed in megapascals, using the equation

$$\mathrm{TS}_\mathrm{b} = \frac{F_\mathrm{b}}{Wt}$$

Calculate the elongation at break, E_b, expressed as a percentage, using the equation

$$E_\mathrm{b} = \frac{100(L_\mathrm{b} - L_0)}{L_0}$$

Calculate the stress at a given elongation, S_e, expressed in megapascals, using the equation

$$S_\mathrm{e} = \frac{F_\mathrm{e}}{Wt}$$

Calculate the elongation at a given stress, E_s, expressed as a percentage, using the equation

$$E_\mathrm{s} = \frac{100(L_\mathrm{s} - L_0)}{L_0}$$

The value, in newtons, of the force, F_e, corresponding to a given stress is calculated using the equation

$$F_\mathrm{e} = S_\mathrm{e} W t$$

Calculate the tensile stress at yield, S_y, expressed in megapascals, from the force recorded at the yield point, using the equation

$$S_\mathrm{y} = \frac{F_\mathrm{y}}{Wt}$$

Calculate the elongation at yield, E_y, expressed as a percentage, using the equation

$$E_\mathrm{y} = \frac{100(L_\mathrm{y} - L_0)}{L_0}$$

In the equations given above, the symbols used have the following meanings:

F_b	is the force recorded at break, in newtons;
F_m	is the maximum force recorded, in newtons;
F_y	is the force recorded at yield, in newtons;
L_0	is the initial test length, in millimetres;
L_b	is the test length at break, in millimetres;
L_s	is the test length at a given stress, in millimetres;
L_y	is the test length at yield, in millimetres;
t	is the thickness of the test piece over the test length, in millimetres;
W	is the width determined as specified in 12.1, in millimetres.

15.2 Ring test pieces

Calculate the tensile strength, TS, expressed in megapascals, using the equation

$$\mathrm{TS} = \frac{F_\mathrm{m}}{2Wt}$$

Calculate the tensile strength at break, TS_b, expressed in megapascals, using the equation

$$\mathrm{TS}_\mathrm{b} = \frac{F_\mathrm{b}}{2Wt}$$

Calculate the elongation at break, E_b, expressed as a percentage, using the equation

$$E_\mathrm{b} = \frac{100(\pi d + 2L_\mathrm{b} - C_\mathrm{i})}{C_\mathrm{i}}$$

Calculate the stress at a given elongation, S_e, expressed in megapascals, using the equation

$$S_e = \frac{F_e}{2Wt}$$

The distance, in millimetres, between the pulley centres corresponding to a given elongation, L_e, also in millimetres, is calculated using the equation

$$L_e = \frac{C_m E_s}{200} + \frac{C_i - \pi d}{2}$$

Calculate the elongation at a given stress, E_s, expressed as a percentage, using the equation

$$E_s = \frac{100(\pi d + 2L_s - C_i)}{C_m}$$

The value, in newtons, of the force, F_e, corresponding to the required stress is calculated using the equation

$$F_e = 2S_e Wt$$

Calculate the tensile stress at yield, S_y, expressed in megapascals, using the equation

$$S_y = \frac{F_y}{2Wt}$$

Calculate the elongation at yield, E_y, expressed as a percentage, using the equation

$$E_y = \frac{100(\pi d + 2L_y - C_i)}{C_m}$$

In the equations given above, the symbols used have the following meanings:

C_i is the initial internal circumference of the ring, in millimetres;

C_m is the initial mean circumference of the ring, in millimetres;

d is the diameter of the pulleys, in millimetres;

F_b is the force recorded at break, in newtons;

F_m is the maximum force recorded, in newtons;

F_y is the force recorded at yield, in newtons;

L_b is the distance between the pulley centres at break, in millimetres;

L_s is the distance between the pulley centres at a given stress, in millimetres;

L_y is the distance between the pulley centres at yield, in millimetres;

t is the axial thickness of the ring, in millimetres;

W is the radial width of the ring, in millimetres.

16 Expression of results

When more than one tensile stress-strain property is being determined on the same test pieces, the test data shall be treated as if they had been obtained independently for each property and the result calculated as described for each property separately.

In all cases, the median for each property shall be reported.

17 Precision

See Annex B.

NOTE Annex C analyses, on the basis of the precision data, the performance of the different types of dumb-bell test piece.

18 Test report

The test report shall include the following particulars:

a) details of sample and test pieces:

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

    2) compound details and cure condition, if known,

    3) the method of preparation of the test pieces (e.g buffing),

    4) the type of test piece used,

    5) the median thickness of the test piece,

    6) the direction relative to any grain in which dumb-bell test pieces were cut;

b) a full reference to the test method, i.e. the number of this International Standard;

c) test details:

    1) the temperature of test and the relative humidity, if necessary,

    2) the number of test pieces tested,

    3) any deviations from the procedure specified;

d) test results:

    1) the individual test results,

    2) the median values of the properties determined, calculated in accordance with Clause 15;

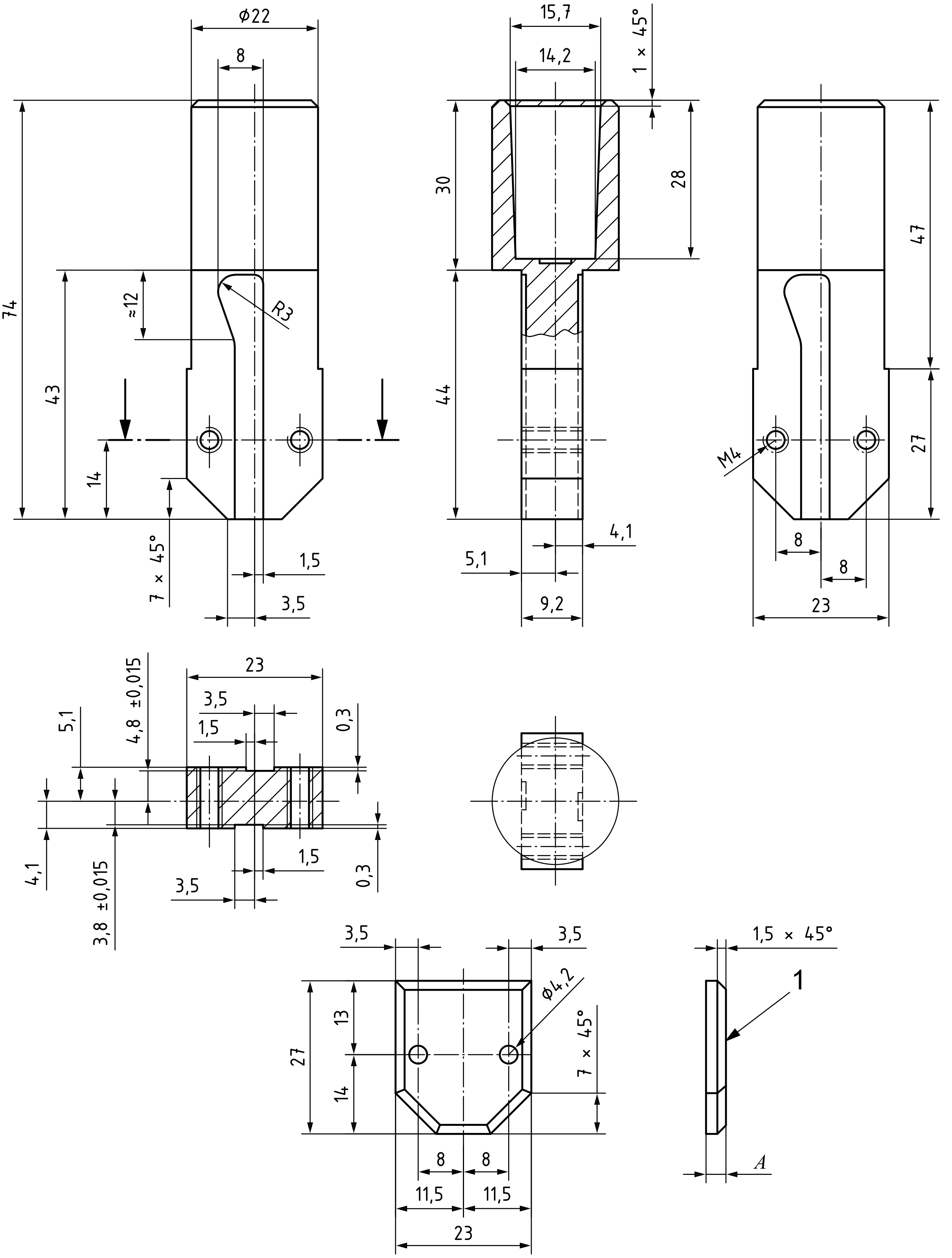
e) the date of testing.

Annex A
(informative)

Preparation of type B ring test pieces

Type B rings may be cut out using a rotary cutting machine operated at 400 rpm and fitted with a special holder for blades (see Figure A.1). The blades should be lubricated with soap solution and inspected frequently for sharpness, damage, etc. The sample should be secured during cutting with the device shown in Figure A.2.

Dimensions in millimetres

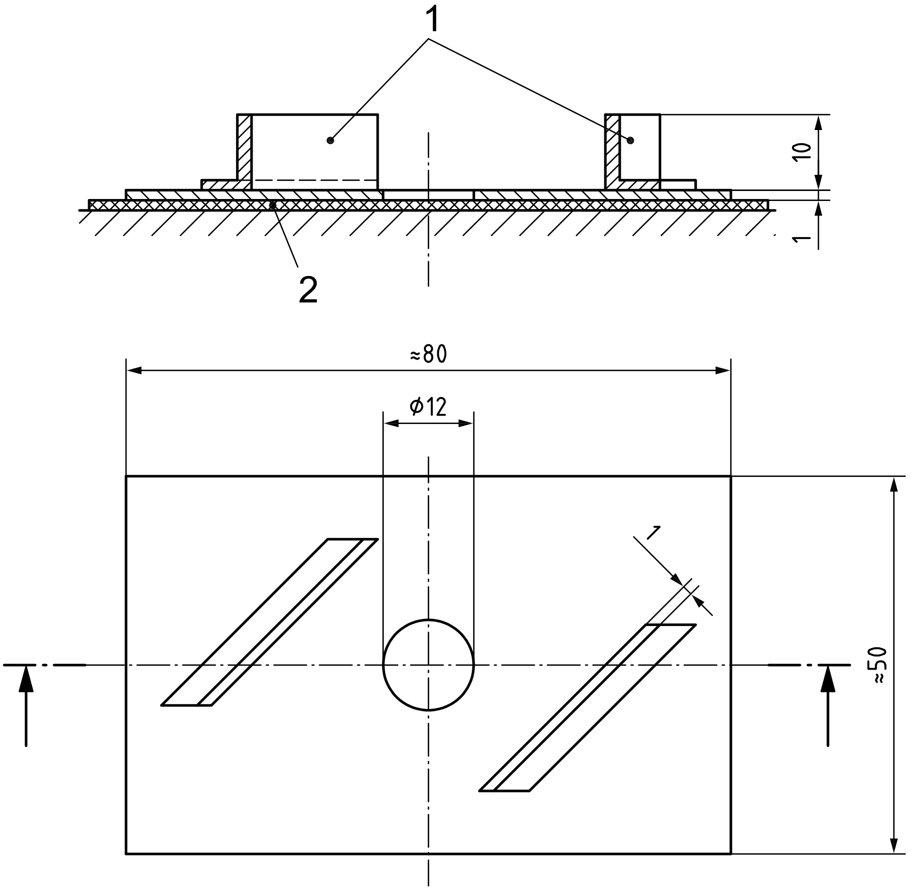


Key

1 side-clamp for blade (dimension *A* is not critical)

Figure A.1 — Special tool holding removable blades

Dimensions in millimetres



Key

1 protection for operator's fingers

2 sheet to be cut

Figure A.2 — Tool for holding the rubber sheet

Annex B
(informative)

Precision

B.1 General

The repeatability and reproducibility of the method were calculated on the basis of ISO/TR 9272:2005. Original data were treated for outliers at the 5 % and 2 % significance levels on the basis of the procedures described in ISO/TR 9272.

B.2 Details of test programmes

B.2.1 Two inter-laboratory test programmes (ITPs) were organized.

The first ITP in 2001 was as follows:

Three different compounds of NR, SBR and EPDM were used for tensile tests. A test result for this test method was the average or mean of five separate measurements of each of the properties as indicated below.

A total of 23 laboratories in eight countries participated in the programme.

The second ITP in 2002 was as follows:

One NR compound was used for tensile testing. The compound formulation was the same as the NR compound used in the first ITP.

A total of 17 laboratories in six countries participated in the programme.

The fully prepared rubber test pieces were sent to each laboratory for evaluation in both ITPs, thus giving a type 1 precision.

B.2.2 The test properties to be measured were tensile strength at break ($\mathrm{TS_b}$), elongation at break (E_b), stress at 100 % elongation (S_{100}) and stress at 200 % elongation (S_{200}).

B.2.3 Three types of dumb-bell, type 1, type 2 and type 1A, were tested.

The type 1 was tested with two test lengths of 20 mm and 25 mm marked on them in the first ITP, but for the second ITP only test pieces with a test length of 25 mm were tested.

B.3 Precision results

The results calculated for the precision are given in Tables B.1, B.2, B.3 and B.4. Tables B.1, B.2 and B.3 show the results of the first ITP for the NR, SBR and EPDM compounds, respectively. Table B.4 shows the results of the second ITP for NR.

The symbols used in these tables are defined as follows:

r = repeatability, in measurement units;

(r) = repeatability, in percent (relative);

R = reproducibility, in measurement units;

(R) = reproducibility, in percent (relative).

Table B.1 — Precision for NR compound (first ITP)

Property	Dumb-bell type/ test length	Mean value $N = 23 \times 2 = 46$	Within-laboratory repeatability		Inter-laboratory reproducibility	
			r	(r)	R	(R)
TS_b	Type 1/20 mm	34,25	1,10	3,20	3,35	9,79
	Type 1/25 mm	34,17	1,53	4,47	2,49	7,29
	Type 2/20 mm	31,93	1,25	3,93	2,85	8,94
	Type 1A/20 mm	34,88	0,67	1,91	2,63	7,54
E_b	Type 1/20 mm	671	42,1	6,28	57,2	8,52
	Type 1/25 mm	670	66,3	9,89	63,1	9,41
	Type 2/20 mm	651	29,9	4,60	60,5	9,29
	Type 1A/20 mm	687	29,9	4,35	57,8	8,41
S_{100}	Type 1/20 mm	1,83	0,18	10,00	0,36	19,50
	Type 1/25 mm	1,86	0,12	6,73	0,32	17,24
	Type 2/20 mm	1,84	0,15	8,33	0,40	21,95
	Type 1A/20 mm	1,89	0,07	3,90	0,28	14,81
S_{200}	Type 1/20 mm	4,49	0,45	10,08	0,85	18,97
	Type 1/25 mm	4,42	0,52	11,82	0,77	17,36
	Type 2/20 mm	4,39	0,39	8,79	0,87	19,85
	Type 1A/20 mm	4,58	0,38	8,25	0,70	15,26

Table B.2 — Precision for SBR compound (first ITP)

Property	Dumb-bell type/ test length	Mean value $N = 23 \times 2 = 46$	Within-laboratory repeatability		Inter-laboratory reproducibility	
			r	(r)	R	(R)
TS_b	Type 1/20 mm	24,87	1,48	5,94	2,12	8,53
	Type 1/25 mm	24,60	1,17	4,74	2,58	10,47
	Type 2/20 mm	24,38	1,52	6,22	2,84	11,65
	Type 1A/20 mm	24,70	1,01	4,11	2,38	9,65
E_b	Type 1/20 mm	457	29,3	6,40	39,0	8,53
	Type 1/25 mm	458	31,4	6,85	31,6	6,90
	Type 2/20 mm	462	32,9	7,12	48,2	10,43
	Type 1A/20 mm	459	13,9	3,04	41,1	8,96
S_{100}	Type 1/20 mm	2,64	0,20	7,46	0,51	19,47
	Type 1/25 mm	2,61	0,20	7,52	0,41	15,75
	Type 2/20 mm	2,66	0,24	9,11	0,57	21,30
	Type 1A/20 mm	2,65	0,10	3,87	0,43	16,15
S_{200}	Type 1/20 mm	7,76	0,59	7,62	1,28	16,52
	Type 1/25 mm	7,74	0,47	6,08	0,94	12,15
	Type 2/20 mm	7,68	0,56	7,31	1,48	19,25
	Type 1A/20 mm	7,81	0,45	5,74	1,00	12,79

Table B.3 — Precision for EPDM compound (first ITP)

Property	Dumb-bell type/ test length	Mean value $N = 23 \times 2 = 46$	Within-laboratory repeatability		Inter-laboratory reproducibility	
			r	(r)	R	(R)
TS_b	Type 1/20 mm	14,51	1,13	7,78	2,01	13,83
	Type 1/25 mm	14,59	1,57	10,76	2,22	15,20
	Type 2/20 mm	14,50	1,20	8,26	2,14	14,74
	Type 1A/20 mm	14,77	0,65	4,39	1,87	12,65
E_b	Type 1/20 mm	470	22,2	4,71	32,4	6,90
	Type 1/25 mm	474	33,8	7,13	44,5	9,38
	Type 2/20 mm	475	21,9	4,60	42,4	8,93
	Type 1A/20 mm	471	20,2	4,28	39,2	8,34
S_{100}	Type 1/20 mm	2,33	0,21	8,99	0,36	15,32
	Type 1/25 mm	2,30	0,18	7,61	0,32	13,94
	Type 2/20 mm	2,39	0,17	7,21	0,32	13,52
	Type 1A/20 mm	2,40	0,09	3,87	0,29	12,04
S_{200}	Type 1/20 mm	5,11	0,35	6,87	0,65	12,80
	Type 1/25 mm	5,05	0,25	4,88	0,62	12,35
	Type 2/20 mm	5,08	0,27	5,24	0,71	14,04
	Type 1A/20 mm	5,20	0,22	4,22	0,46	8,84

Table B.4 — Precision for NR compound (second ITP)

Property	Dumb-bell type/ test length	Mean value $N = 17 \times 2 = 34$	Within-laboratory repeatability		Inter-laboratory reproducibility	
			r	(r)	R	(R)
TS_b	Type 1/25 mm	32,26	1,86	5,76	2,21	6,84
	Type 2/20 mm	34,75	1,53	4,41	4,04	11,63
	Type 1A/20 mm	33,13	1,19	3,60	2,71	8,17
E_b	Type 1/25 mm	640	27,26	4,26	54,44	8,50
	Type 2/20 mm	683	30,80	4,51	94,49	13,83
	Type 1A/20 mm	665	22,94	3,45	83,52	12,56
S_{100}	Type 1/25 mm	1,74	0,13	7,29	0,32	18,17
	Type 2/20 mm	1,83	0,20	11,08	0,30	16,18
	Type 1A/20 mm	1,78	0,13	7,06	0,22	12,19
S_{200}	Type 1/25 mm	4,27	0,32	7,42	1,10	25,81
	Type 2/20 mm	4,31	0,44	10,31	1,03	23,91
	Type 1A/20 mm	4,35	0,21	4,78	0,87	20,11

Annex C
(informative)

Analysis of ITP data and dumb-bell shape

C.1 General

This annex considers the performance of the different dumb-bell shapes, including the type 1A, that was measured through the ITP programmes. The type 1A dumb-bell is a relatively new addition to this International Standard, but it had been in use in Japan and other countries for many years.

Inter-laboratory tests showed that the type 1A dumb-bell has advantages over types 1 and 2 of better repeatability and, particularly, lower incidence of breaks outside the test length. Finite-element analysis demonstrated that the strain distribution in the type 1A is more uniform, which probably accounts for its improved performance.

The values of the tensile properties determined with the type 1A dumb-bell are very similar to those obtained with the type 1, but they cannot be expected to be identical in all cases.

The type 1A dumb-bell has similar overall dimensions to the type 1 and can be considered as an alternative. It has not replaced the type 1 because of the huge bank of data obtained and the long tradition with the latter dumb-bell.

C.2 Three variances for three-factor fully-nested experiments

In the comparison of the precision calculated in accordance with ISO/TR 9272, R is an indicator of the variance between laboratories (σ_L^2), and the value of r is an indicator of the total variance ($\sigma_D^2 + \sigma_M^2$) for a particular laboratory, made up of the variance between the days (σ_D^2) and the variance due to measurement errors (σ_M^2). In order to analyse σ_D^2 and σ_M^2 separately, it is enough to make an estimate of each component of the variance by the so-called three-factor fully-nested experiment described in ISO 5725-3.

The estimate was made for each component of the total variance in the measurements in the second ITP. The results are shown in Tables C.1 and C.2.

Table C.1 — Estimate of each component of the variance by means of "three-factor fully-nested experiments" for tensile strength in the second ITP

	Type 1	Type 2	Type 1A
σ_L^2	$(0{,}60)^2$	$(1{,}80)^2$	$(0{,}80)^2$
σ_D^2	$(0{,}67)^2$	$(0{,}54)^2$	$(0{,}17)^2$
σ_M^2	$(1{,}60)^2$	$(1{,}08)^2$	$(1{,}04)^2$

Table C.2 — Estimate of each component of the variance by means of "three-factor fully-nested experiments" for elongation in the second ITP

	Type 1	Type 2	Type 1A
σ_L^2	$(20{,}4)^2$	$(43{,}7)^2$	$(24{,}3)^2$
σ_D^2	$(13{,}6)^2$	$(21{,}9)^2$	$(28{,}6)^2$
σ_M^2	$(28{,}1)^2$	$(19{,}3)^2$	$(19{,}3)^2$

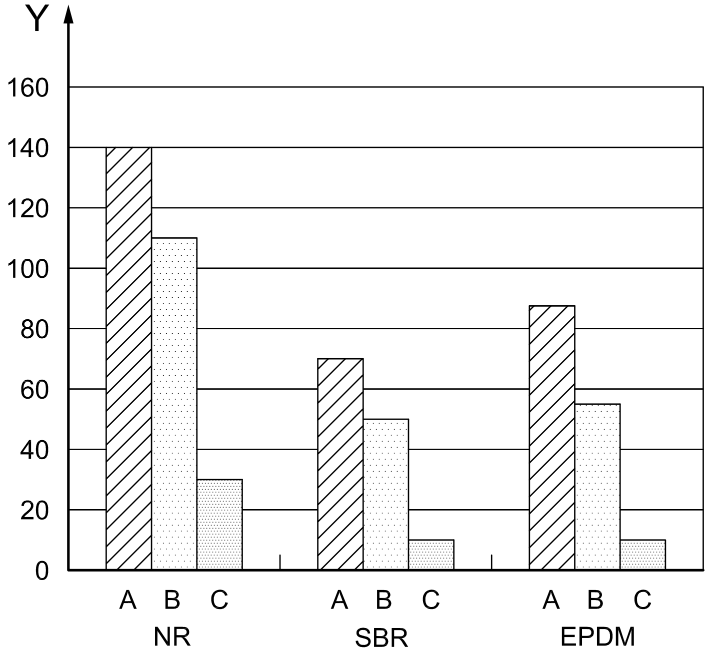
Of the three variances, the variance due to measurement errors (σ_M^2) is the most important for the dumb-bell shape. Other variances (σ_L^2 and σ_D^2) are influenced by many factors other than the dumb-bell shape.

It is shown that σ_M^2 is smallest for the type 1A dumb-bell, which means that the measurement precision is best with this type.

C.3 Analysis of test pieces that broke

C.3.1 Numbers of test pieces that broke outside the test length

Figure C.1 shows the numbers of test pieces that broke outside the test length (outside the reference marks). For each dumb-bell type, 230 test pieces were tested, as 23 laboratories each tested five test pieces on two test days.



Key

Y number of test pieces that broke outside test length

A type 1 dumb-bell (with a 25 mm test length)
B type 2 dumb-bell
C type 1A dumb-bell

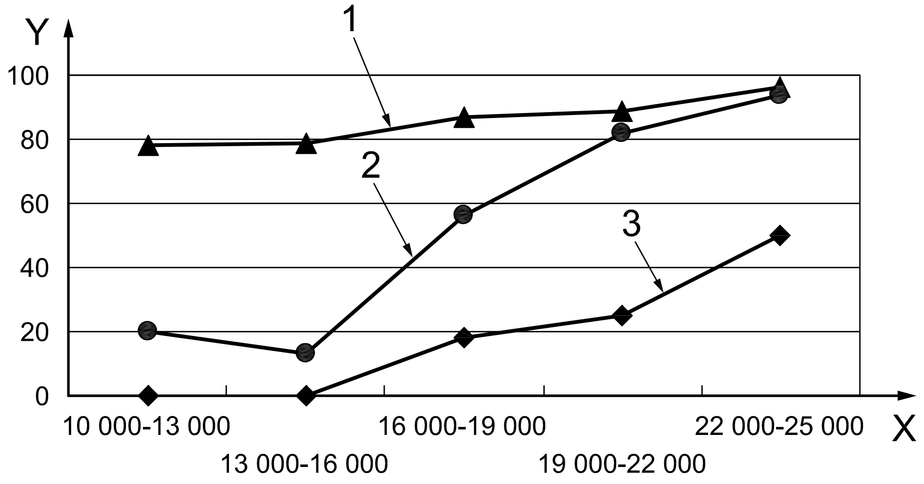
Figure C.1 — Numbers of test pieces that broke outside the test length
(first ITP — total of 230 of each type of test piece)

In the case of the type 1 dumb-bells made of an NR compound with a 20 mm test length, 159 test pieces broke outside the test length, which is about 70 %. In the case of type 1 with a 25 mm test length, it was about 60 % of the test pieces. In the case of type 2, it was 47 %. However, in the case of type 1A, only 13 % of the test pieces broke outside the test length.

With SBR and EPDM, the probability of breaking outside the test length for type 1A was also considerably smaller than for the other dumb-bells.

C.3.2 Relationship between the proportion of test pieces that broke outside the test length and tensile energy

The relationship between the percentage of test pieces that broke outside the test length and the tensile energy (tensile strength multiplied by elongation at break) was also investigated. NR compounds differing in the volume of carbon black they contained were prepared, and their TS_b and E_b measured. The percentage of test pieces that broke outside the test length was observed. Figure C.2 shows the results of this experiment.



Key

X $TS_b \times E_b$ (MPa %)
Y percentage of test pieces that broke outside test length

1 type 1 dumb-bell
2 type 2 dumb-bell
3 type 1A dumb-bell

Figure C.2 — Percentage of test pieces that broke outside the test length vs $TS_b \times E_b$ (tensile energy)

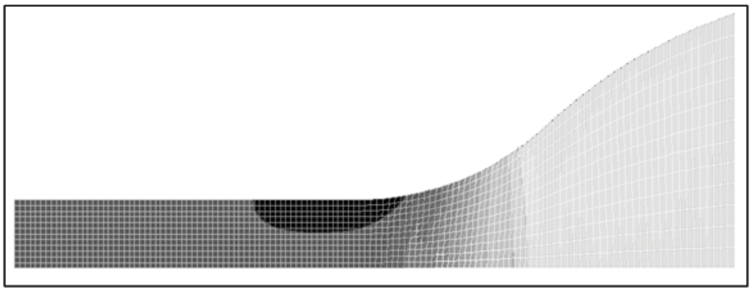
As the value of the tensile energy increased, the percentage of test pieces that broke outside the test length increased. At values of the tensile energy below 20 000 MPa %, most of the type 1A test pieces broke inside the test length.

C.4 Finite-element analysis

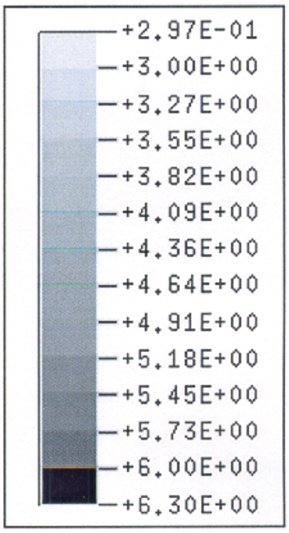
A finite-element analysis (FEA) was conducted on part of the test piece. Figure C.3 shows the distribution of the strain, obtained using “ABAQUS” software.

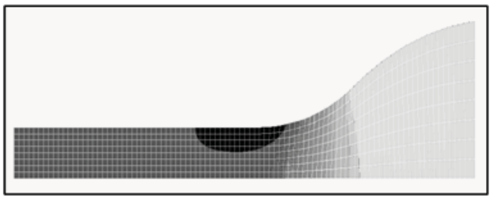
The analysis of the strain distribution shows that the highest strain area appears near the edge of the pieces for type 1 and type 2. This observation coincides with the results of the tensile testing described in Clause C.3.

On the other hand, for type 1A, the strain near the edge is at the same level as in the central area, which means type 1A has a relatively uniform strain distribution.

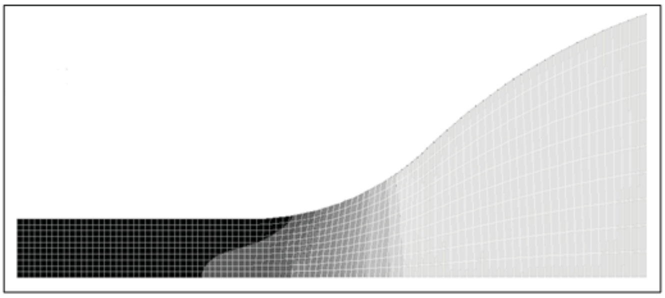


a) Type 1 dumb-bell





b) Type 2 dumb-bell



c) Type 1A dumb-bell

Figure C.3 — Example of strain distribution obtained using “ABAQUS”

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[2] ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

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