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Rubber, vulcanized — Determination of static adhesion to textile cord — H-pull test

Caoutchouc vulcanisé — Détermination de l'adhérence statique au câblé textile — Essai d'arrachement en H

Reference number
ISO 4647:2010(E)

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Foreword

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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 4647 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 4647:1982), which has been technically revised primarily concerning updating the references, including a reference to ISO 5893 for the apparatus, updating the test report format and the addition of a calibration schedule.

Rubber, vulcanized — Determination of static adhesion to textile cord — H-pull test

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.

CAUTION — Certain procedures specified in this International Standard may 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 specifies a method for the determination of the static adhesion of textile cord to vulcanized rubber using the H-pull test. It is applicable to cords made from natural or man-made fibres.

The property levels obtained with this method are affected considerably by the history of the cord and the rubber compound. The method yields data, however, on which a judgement as to the service quality of the material can be based.

The method is primarily intended for use with tyre cord. However, it can be applied, if desired, to similar cords for use in other rubber products, but it is limited to cords of linear density not exceeding 800 mg/m (tex).

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 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 Principle

The adhesion between a rubber and textile cord is assessed by measuring the force required to pull a single cord from a block of vulcanized rubber, the force being applied along the longitudinal axis of the cord and the length of cord embedded in the rubber being fixed (see Figure 1).

The adhesion measured is essentially a shearing force acting at the cord-to-rubber interface. The two strips of rubber and the interconnecting cord form a test piece resembling the letter “H”, from which the test derives its name.

4 Materials

The materials comprise any combination of rubber compound, textile cord and adhesive agreed upon by both the cord user and the supplier. The vulcanizing conditions, both time and temperature, shall be exactly specified.

Square-woven, approximately 340 g/m^2 cotton fabric, or its equivalent, shall be used to support the rubber strips. This may be grey fabric or fabric that has been frictioned on one side. Alternatively, the rubber compound may be calendered to the frictioned side of the cotton fabric. The rubber surface which will be in contact with the cords shall be protected by a protective film, for example starch paper or polyethylene.

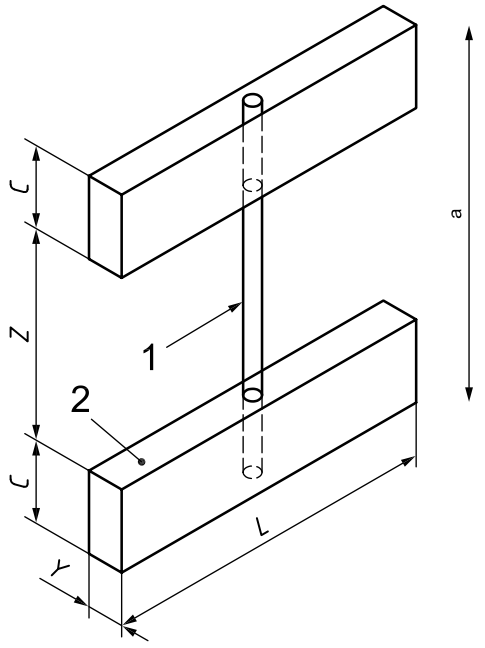
The thickness of the rubber compound required to fill the mould completely shall be determined by the supplier and the purchaser.

NOTE The decision as to which rubber compound to use is normally made by the cord user.

5 Apparatus

5.1 Mould.

The dimensions of the test pieces are controlled by the specifications and tolerances of the mould. The test pieces are prepared by laying strips of rubber, of thickness Y/2 (see Figure 1), spaced a distance Z apart, into cavities in a mould of width C.



Key

1 cord

2 rubber

a Direction of applied force.

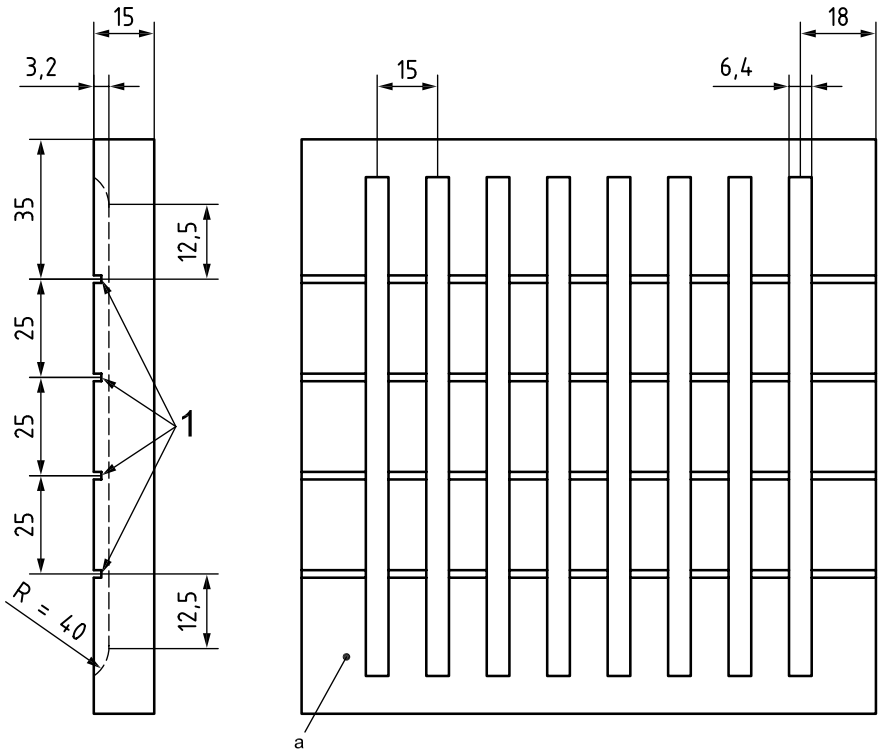
Figure 1 — Test piece

Cords are stretched over and perpendicular to the rubber strips, with a distance L between each cord. Two further strips of rubber are applied above the cord, the mould closed and put into a press, and the test pieces vulcanized.

It is common practice to use moulds which allow many identical test pieces to be produced simultaneously as a test pad.

One example of a suitable mould is shown in Figure 2. It is recommended that the width of the cord groove be 0,8 mm for cords of linear density 560 mg/m (tex) or less, and 1,2 mm for cords of linear density more than 560 mg/m (tex) and up to 800 mg/m (tex). Although this form of mould is simple to use, the moulding pressure tends to force excess rubber down the cord groove between the rubber strips, particularly when the cord is much narrower than the groove. This “flash” shall be removed from the cord by careful cutting before the test to improve the reproducibility of the results. The formation of this rubber flash can be almost completely eliminated by using a mould of the form shown in Figure 3, which shows two methods for preparing test pieces. The technique requires the cord length between the rubber strips to be held in position during vulcanization by a deformable surface, rather than a groove, so that there are no voids into which excess rubber can flow.

Dimensions in millimetres



Key

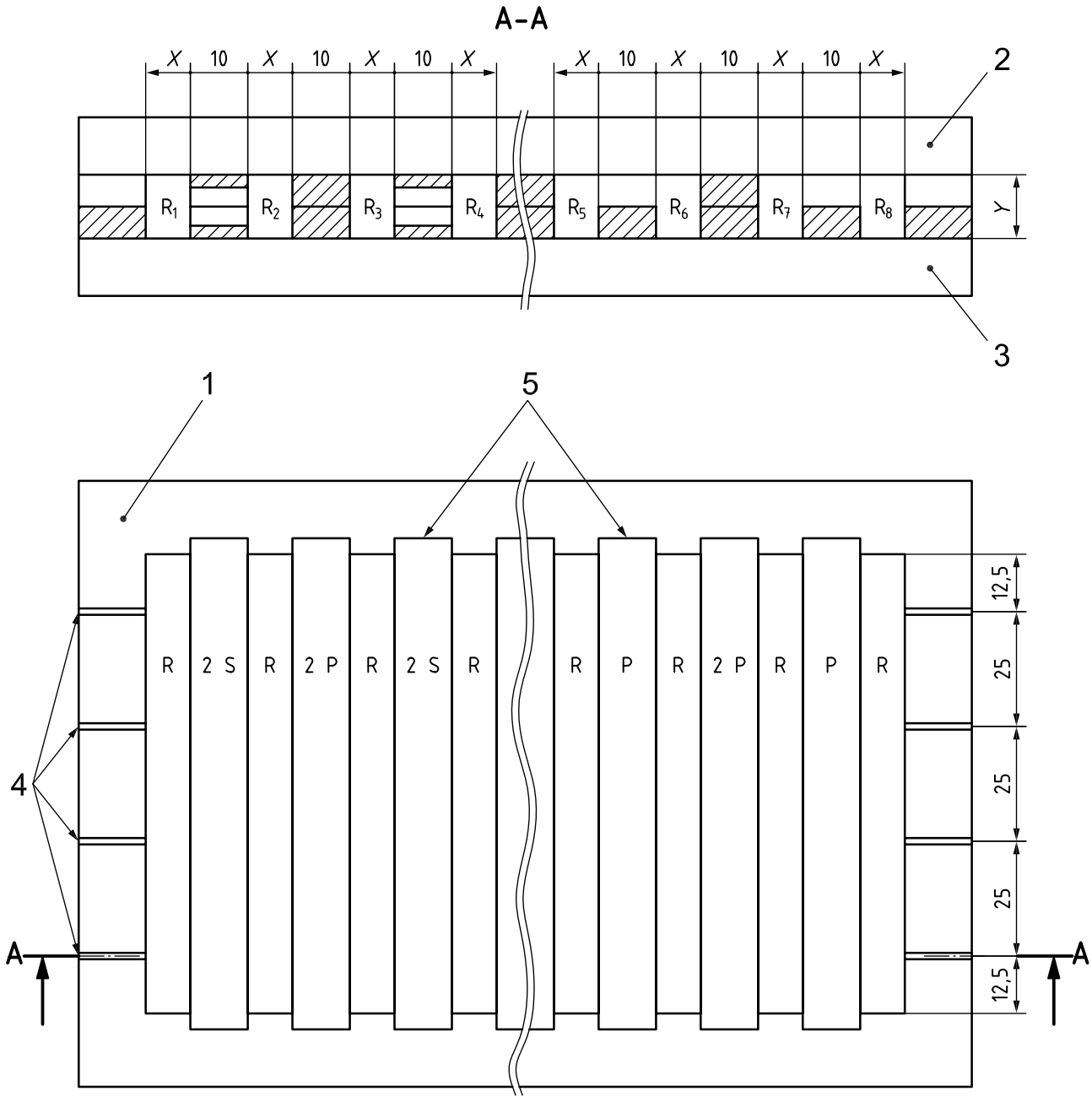
1 four slots 0,8 mm or 1,2 mm wide and 3,0 mm deep, with all burrs removed

The mould shown will produce 16 test pieces. It may be fabricated to produce a larger or smaller number, but the dimensions that govern the size of the test pieces may not be altered.

[a] Material: hot-rolled steel.

Figure 2 — Suitable mould for H-pull test

Dimensions in millimetres



a) Method A b) Method B

Key

1 mould frame
2 mould top plate
3 mould base plate
4 cord grooves, width 0,8 mm or 1,2 mm (see 5.1)
5 locating grooves for spacer bars
R rubber cavity, width X and depth Y (see 5.1 and 7.1)
S silicone-rubber-faced bar
P plain spacer bar

Figure 3 — Methods of preparation of test pieces

In method A, the cord between the rubber strips R_1 and R_2, and between R_3 and R_4, is held between specially prepared silicone-rubber-faced bars. A suitable method for the preparation of such bars is described in Annex A.

In method B, the upper strip of rubber is made sufficiently wide to cover the whole distance R_5 to R_6 (and R_7 to R_8), with the addition of a thin cellophane or polyester strip applied to the central portion of the rubber, which contacts the cords, to prevent the adherence of rubber to the cord in this region.

5.2 Tensioning device, capable of providing a tensioning force of 0,49 N ± 0,1 N. This can be achieved, for example, by suspending a mass of 50 g ± 1 g on one end of each cord during assembly of the test piece and removing it prior to placing the mould in the vulcanizing press. The masses can be of the hook type or designed in such a manner that they can be clamped to the cord. In any event, the total mass shall be 50 g ± 1 g.

5.3 Tensile-testing machine, complying with the requirements of ISO 5893, capable of measuring force with an accuracy corresponding to class 1 and with a rate of traverse of the moving grip of 100 mm/min ± 10 mm/min.

5.4 Test piece grips, of a design as shown in Figure 4 or Figure 5. Two grips are required.

NOTE The two types of grip do not necessarily give the same results.

6 Calibration

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

7 Test piece

7.1 Dimensions

The standard test piece shall be a length of cord embedded in rubber strips, nominally 6,4 mm wide and 3,2 mm thick (see 5.1).

Although this method specifies that the rubber strips shall be 3,2 mm thick, an interlaboratory test gave equivalent values for 3,2-mm-thick and 6,4-mm-thick test pieces. The embedded length of cord may be reduced to 5 mm or increased to 10 mm where the adhesion is very high or very low respectively, but the results obtained using different embedded lengths are not comparable.

7.2 Preparation

7.2.1 Cut the rubber compound into strips, 6 mm wide and of a suitable length, leaving the protective film attached. This can be done with scissors or with a clicker die cutter.

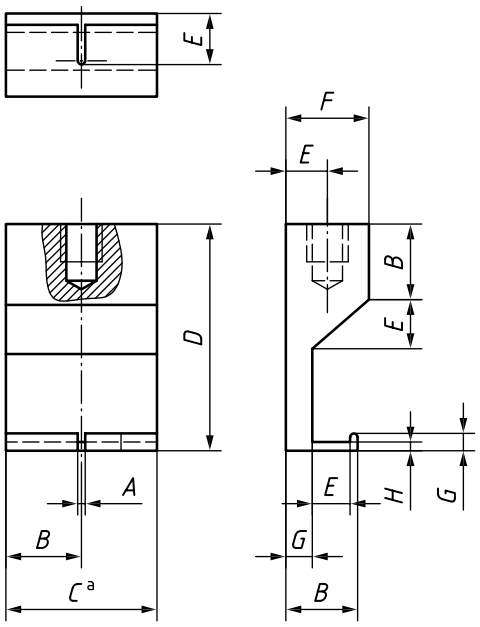
7.2.2 Cut strips of cotton fabric to the same dimensions as those of the rubber compound. (If the rubber compound is calendered onto the fabric, do not carry out this step.)

7.2.3 If necessary, place the bottom spacer bars in the mould (Figure 3 type mould).

7.2.4 Using a mould at room temperature, place the fabric strips in the bottom of the mould cavities (see 7.2.11, second paragraph).

7.2.5 Place the rubber strips in the mould cavities with the protective-film side on top. (If the rubber is calendered onto the fabric, the fabric side shall be on the bottom.)

Dimensions in millimetres



Dimension	Value in mm
A	1,6
B	12,5
C[a]	25,0
D	40,0
E	7,0
F	14,0
G	4,0
H	2,0

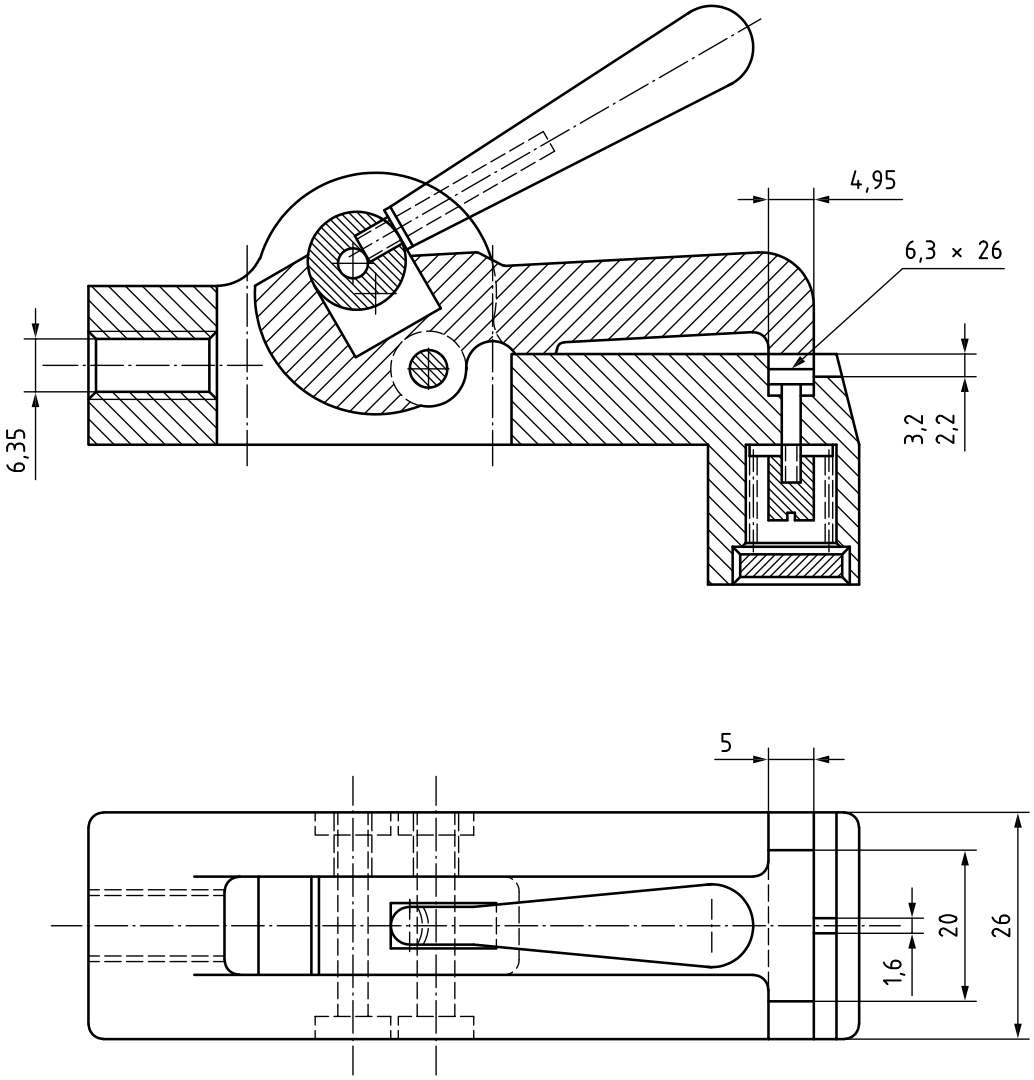
[a] This dimension is the most important one and shall not be altered. All other dimensions are included for guidance and may be altered if desired.

Break all sharp edges and corners with slight radii.

The threaded hole is drilled and tapped to facilitate attaching the grip to the tester.

Figure 4 — Test piece grip

Dimensions in millimetres



NOTE The lower part of the grip is spring loaded with a spring tension of between 5 N and 15 N to minimize deformation of the rubber.

Figure 5 — Alternative test piece grip

7.2.6 Remove the protective film from the rubber strips and immediately place the cords in the cord slots. The portion of the cord to be embedded in the rubber shall not be touched with bare hands. The procedure for handling calendered cords shall be agreed upon by the purchaser and the supplier. Knot each cord at one end so that it is secured firmly against the cord slot on one side of the mould. Take care to prevent the loss of cord twist. Attach a tensioning device to the other end of the cord.

7.2.7 If required, place the upper spacer bars in the mould.

7.2.8 Remove the protective film from additional strips of rubber and place them in the mould cavities on top of the cords. The side from which the protective film was removed shall be facing down. When preparing test pieces by method B, these additional strips of rubber shall be 22 mm wide, with a strip of protective film or similar material replaced over the central 10-mm-wide area.

7.2.9 Place strips of fabric on top of the rubber strips. (If the rubber is calendered onto the fabric, eliminate this step.)

7.2.10 Identify the test pieces in the mould and cover the mould with a smooth metal plate if the upper press platen is not smooth.

7.2.11 Remove the tensioning devices from the cords and place the mould in a press which has been preheated to the vulcanizing temperature. Adjust the pressure to a minimum of 3,5 MPa with reference to the mould surface. After vulcanizing for the specified time, immediately remove the test pad from the mould and allow to cool at room temperature.

A preheated mould may be used, but this will alter the vulcanizing conditions (time and temperature) of the rubber. If a preheated mould is used, the materials should exhibit sufficient tack to permit the pad to be prefabricated in a cold mould and then transferred to the preheated mould.

7.2.12 Cut the pads using scissors, a sharp knife or a clicker die cutter to produce "H" pieces consisting of a single cord with each end embedded in the centre of a rubber tab approximately 25 mm in length. If necessary, trim off all excess rubber flash. When using method B, the trimming shall be carried out with great care to avoid cutting into the test piece.

7.3 Number of test pieces

At least eight test pieces shall be used.

8 Time interval between vulcanization and testing

Unless otherwise specified for technical reasons, the following requirements for time intervals shall be observed.

For all test purposes, the minimum time between vulcanization and testing shall be 16 h.

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.

9 Conditioning of test pieces

9.1 When tests are performed at a standard laboratory temperature, the test piece shall be maintained at the conditions of test for at least 16 h immediately before testing.

9.2 When tests are made at higher or lower temperatures, the test pieces shall be maintained at the conditions of test for a period of time sufficient to reach temperature equilibrium with the test environment, or for the period of time required by the specification covering the material or product being tested, and shall then be tested immediately.

10 Test temperature

The test shall normally be carried out at a standard laboratory temperature specified in ISO 23529. When other temperatures are used, they shall be selected from those given in ISO 23529.

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

11 Procedure

11.1 Testing at standard laboratory temperature

Attach the test piece grips to the tensile machine and set them 1 mm apart. Take care to ensure axial alignment.

Adjust the speed of the movable grip to 100 mm/min ± 10 mm/min. Insert the test piece in the grips and start the machine. Record, to the nearest 1,0 N, the maximum force required to separate the cord from the rubber.

11.2 Testing at elevated temperature

Proceed as described in 11.1, but enclose the test piece grips in a temperature-controlled chamber attached to the tensile-testing machine. Heat the test pieces in the chamber, controlled at the test temperature, for not less than 15 min and not more than 60 min total elapsed time for testing of any one test piece. (Alternatively, a less precise procedure may be used whereby the test pieces are heated in an oven adjacent to the tensile-testing machine and then removed, one at a time, and tested within 15 s of removal.)

12 Expression of results

Record the cord adhesion value, in newtons, and calculate the arithmetic mean of the results. Describe the appearance of the cord, indicating whether the rubber has remained adhered to it or not.

13 Test report

The test report shall include the following information:

a) sample details:

   1) a full description of the sample and its origin, including complete identification of the cord and rubber,

   2) the method of preparation of the test pieces from the sample, including the date of moulding;

b) test method:

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

   2) the test procedure used (testing at standard laboratory temperature or testing at elevated temperature),

   3) the type of test piece used;

c) test details:

   1) the time and temperature of conditioning prior to testing,

   2) the test temperature,

   3) the type of grip used,

   4) details of any procedures not specified in this International Standard;

d) test results:

   1) the number of test pieces used,

   2) the individual cord adhesion values and the arithmetic mean of these values,

   3) the appearance of the cord after each test;

e) the date(s) of testing.

Annex A
(informative)

Preparation of silicone-rubber-faced bars

A.1 Silicone compound preparation

A.1.1 A self-bonding grade of silicone rubber of hardness about 60 IRHD has been found suitable.

A.1.2 Depending on the mould plate size available, press out, between sheets of polyester, a 50 g to 60 g piece of the compound, using two flat mould plates to obtain a thickness of 1,5 mm which is as even as possible. The pressing is best done in a hydraulic press at very low pressure (less than 175 kPa). A hand-pump press is preferable. The platens may be heated to 50 °C to 70 °C, if preferred.

A.1.3 After pressing for 2 min to 3 min, examine the silicone rubber to see if the required thickness has been reached. It may not be possible to attain 1,5 mm, but up to 2 mm is permissible.

A.1.4 Remove the sheet of silicone rubber and store it on a cool, flat surface, still protected by the polyester film.

A.2 Bar preparation

A.2.1 Remove as much old silicone rubber as possible from any previously used bars by scraping, mechanical wire-brushing of the bars, or similar means.

A.2.2 Degrease the bars in a vapour degreasing bath, using trichloroethylene or perchloroethylene, for 30 min to 60 min.

A.2.3 Abrade the bars on the bonding surfaces only, using fine emery cloth. Light shot or vapour blasting may also be used, but it is advisable to protect the non-bonding surfaces to prevent unnecessary roughening.

A.2.4 Finally, clean the bars by wiping liberally with a clean cloth soaked in a petroleum solvent, the solvent being allowed to evaporate. Apply the strip of silicone rubber (see A.3.1) as soon as possible after cleaning.

A.3 Bonding procedure

A.3.1 Cut 10-mm-wide strips from the polyester-film-protected silicone rubber to fit the bonding areas on the bars. If the silicone rubber is unavoidably thick, the width of the strips may be reduced by between 1 mm and 2 mm to prevent excessive spew.

A.3.2 Peel the polyester film from one face of the strip, place the exposed face on the freshly cleaned bonding area and, manually, lightly press into contact. Avoid contact with the exposed silicone rubber surface and the prepared bar surface to prevent contamination and hence poor bonding.

A.3.3 Prepare two bars at a time. These should be used as a pair, and preferably marked for easy identification.

A.3.4 Place the bars in a suitable mould, side by side, with the silicone rubber faces uppermost. The top layers of polyester film may now be removed if preferred, but to facilitate easy demoulding a piece of polyester film, sufficient to cover the mould cavity, should be inserted. Alternatively, the mould lid should be sprayed with a PTFE aerosol mould lubricant.

A.3.5 With the lid located by the matching holes and pegs of the bars, place the mould in a press while the platens are warming up and use a low pressure to spread the silicone rubber. When the full curing temperature of 160 °C is reached, apply the highest possible safe pressure and vulcanize for 15 min.

A.3.6 Cool the bars, preferably in the mould in the press still under pressure. If this is not possible, allow the complete mould to cool outside the press as at this stage (i.e. when hot) the silicone rubber is still weak and prone to damage.

A.3.7 When cool, carefully remove the faced bars from the mould. This may be facilitated by removing one of the screwed-in mould end stops, allowing a thin lever to be inserted under the bars.

A.3.8 Excess silicone spew, etc., may then be trimmed off, or this may be deferred until after the oven cure.

A.3.9 When all the bars to be re-faced have been press-cured, store them for 18 h to 24 h at 200 °C. Remove excess spew, etc., if this was not carried out at the previous stage.

NOTE 1 During use, the silicone rubber may protrude beyond the edge of the bar. If a trial pull-through test piece is moulded, the amount of excess silicone can be measured by examining the test piece cross-section, which will be an H-section if the silicone protrudes. The edge of the silicone can then be trimmed back from the edge of each bar by slightly less than the depth of the indentation caused in the pull-through test piece.

NOTE 2 With normal usage, a minimum of 500 test mouldings can be expected before the bars need to be refaced.

Annex B
(normative)

Calibration schedule

B.1 Inspection

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

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

B.2 Schedule

Verification/calibration of the test apparatus is a normative part of this International Standard. 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 B.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

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

The verification frequency for each parameter is given by the code-letter “S”, i.e. the standard interval as given in ISO 18899.

In addition to the items listed, some standards assume the use of “normal” laboratory apparatus, e.g. timers, beakers, etc. These may need calibrating in accordance with the appropriate procedure given in ISO 18899. Also, there may be items specified in the relevant cited standard which have a dimension, for example a 250 ml flask. Where the size has no particular significance, other than convenience of use, these items will not need to be calibrated.

Most standards for the testing of materials require the test piece to be conditioned. This implies the use of a thermometer and, possibly, a hygrometer, both of which will require calibration. Standard conditioning and test temperatures and humidities cited in the relevant standard have not been repeated in the schedule.

Test piece dimensions are usually specified. This implies the use of dimension-measuring instruments which will require calibration. Test piece dimensions cited in the relevant standard have not been repeated in the schedule.

Table B.1 — Calibration schedule

Parameter	Requirement	Clause or subclause in ISO 18899:2004	Verification frequency guide	Notes
Recommended mould	As in Figure 2 or Figure 3	15.2	S	
Cord groove width for linear densities				
$\leqslant$ 560 tex	0,8 mm	15.2	S	
$\geqslant$ 560 tex up to 800 tex	1,2 mm	15.2	S	
Tensioning force	0,49 N $\pm$ 0,1 N	21.2	S	E.g. 50 g $\pm$ 1 g on each cord
Tensioning machine	As in ISO 5893			
Force accuracy	Class 1	21.1	S	Low-inertia dynamometer preferred
Rate of separation of jaws	100 mm/min $\pm$ 10 mm/min	23.4	S	
Test piece grips	As in Figure 4 or Figure 5	15.2	S	The two types give the same result
Press	Preheated to vulcanization temperature	Clause 18	S	
Pressure	$\geqslant$ 3,5 MPa	16.6	S	
Materials	Trichloroethylene or perchloroethylene for degreasing Fine emery cloth, or materials for shot or vapour blasting Petroleum solvent			

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