
**Rubber, vulcanized — Determination of
adhesion to wire cord**

*Caoutchouc vulcanisé — Détermination de l'adhérence à un câble
métallique*



Reference number
ISO 5603:2011(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 5603 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 5603:2007), of which it constitutes a minor revision designed to add a calibration schedule (see Annex A).

Rubber, vulcanized — Determination of adhesion to wire cord

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 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 two methods for determining the adhesion strength of vulcanized rubber to wire cord which is embedded in the rubber.

The two methods do not necessarily give the same results.

They are applicable primarily to test pieces prepared in the laboratory under standard conditions and used for the development and control of materials and processes utilized in the manufacture of products reinforced with wire cord.

NOTE The methods can also be used for single wire, for example bead wire.

Method 1 reduces the dependence of the measured adhesion on the modulus and strength properties of the rubber.

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 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*

ISO 5893:2002, *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 strength is determined by measuring the force required to pull out a single cord from the surrounding rubber of a prepared test piece. The force is directed along the axis of the cord, using a test jig containing a suitably chosen circular- or square-shaped hole to ensure uniformity of stress.

Test pieces of cord and rubber are prepared together by vulcanizing them under pressure.

In method 1, the exterior surface of the rubber is suitably reinforced.

In method 2, such reinforcement is not employed.

4 Materials

4.1 Wire cord, conforming to the specification of the bonding system to be investigated. If no specification is given, brass-plated steel cord of construction $(1 \times 3 \times 0,15) \text{ mm} + (6 \times 0,27) \text{ mm}$ or of construction $(7 \times 4 \times 0,22) \text{ mm}$ shall be used.

NOTE The sizes of the cords are defined here in terms of their diameter and the number of component wires, starting from the centre core of the cord.

For quality control of cord, it shall be tested in the condition received, i.e. neither cleaned nor dried.

It is essential that the wire cord be stored in a dry atmosphere to prevent any surface deterioration. Storage can conveniently be carried out in an airtight container which also contains a desiccating material (for example silica, SiO_2). The container shall only be opened when removing wire cord and shall then be closed immediately. It is also essential that the cord not be contaminated with dust from the desiccating material.

4.2 Unvulcanized rubber compound, conforming to the specification of the bonding system to be investigated. Whenever possible, the rubber shall be freshly milled. If for any reason the rubber cannot be remilled, the surface shall be freshened by wiping with a solvent and allowed to dry. The preferred solvent is heptane, but a suitable alternative could be a petroleum solvent with a distillation range of about $65 \text{ }^\circ\text{C}$ to $125 \text{ }^\circ\text{C}$; these solvents shall possess a maximum residue on evaporation of $3 \text{ mg per } 100 \text{ cm}^3$ of solvent. The compound shall be stored at a standard laboratory temperature of $(23 \pm 2) \text{ }^\circ\text{C}$ or $(27 \pm 2) \text{ }^\circ\text{C}$ prior to use. It can be in the form of calendered sheet of suitable thickness and shall be protected by a dark-coloured polyethylene film.

4.3 Reinforcement material, to stiffen the rubber block. This applies only to method 1. The reinforcement can either be a sheet metal strip treated with adhesive promoter (when the thickness t is at least $0,5 \text{ mm}$) or a strip of rigid rubberized steel cord fabric. A suitable fabric is one containing steel cords of high bending stiffness, for example one of construction $(1 \times 3 \times 0,30) \text{ mm} + (6 \times 0,38) \text{ mm}$ [when the thickness t is $(2,5 \pm 0,1) \text{ mm}$ maximum].

5 Apparatus

5.1 Mould, capable of producing a test piece in which a multiplicity of cords is embedded equally spaced along its length. Different moulds are employed for the two methods.

a) Method 1

The mould is of a semi-follow-on type to obtain maximum consolidation of rubber around the cord during the pressing stage, but thereafter becomes a fixed-cavity mould.

One suitable mould designed to accommodate steel cord fabric reinforcement is shown in Figure 1. This mould produces test pieces of length 310 mm containing 21 cords, but similar moulds employing other numbers of cords greater than nine are acceptable. The mould dimension x will depend on the embedded length of test piece required (which is governed by the diameter of wire cord used, see 7.2). The mould shall be provided with pairs of inserts or spacers to obtain the different lengths of cord embedment. Dimension y is sufficiently great to allow the incorporation of excess rubber during moulding (see 7.3.1).

Another suitable mould designed to accommodate steel strip reinforcement is shown in Figure 2. A tensioning device for the cords can be used with this mould. Figure 3 details some mould dimensions and describes the exchangeable steel spacers which are required for different cord sizes for the moulds shown in Figures 1 and 2.

Any mould producing test pieces of the correct dimensions and applying the moulding force directly on to the rubber, for example by use of a suitable spring, is also acceptable.

If desired, the moulds can impart suitable features on to the moulded test piece to facilitate cord centring during subsequent testing.

b) Method 2

The mould is described in Figures 4 and 5. The mould is designed to produce four test blocks (each of length 200 mm) with the preferred dimensions (see 7.2), each containing 15 steel cords. When testing cords with a diameter equal to or less than 1,7 mm, the mould in Figures 4 and 5 shall be used to produce blocks and, when testing cords with a diameter greater than 1,7 mm, the mould shall be suitably modified. If more than one-third of the cords break at the 12,5 mm embedment, it is advisable to reduce the embedment by use of a suitable mould insert. Moulds designed to produce any other number of test blocks of the required dimensions are acceptable, and moulds designed to produce blocks with different embedded cord lengths are permitted.

5.2 Vulcanizing press, large enough to take the mould. It shall conform to the requirements of ISO 2393 and shall be capable of applying a force of at least 100 kN.

5.3 Tensile-testing machine, conforming to the requirements of class 2 as defined in ISO 5893:2002. It shall be capable of maintaining a rate of separation of the jaws at a constant value within the range 50 mm/min to 150 mm/min.

5.4 Fixture, for holding the test piece in the test machine. It shall possess a suitable slot to allow the test cord to reach the test hole: the hole dimensions shall depend on the test piece type (see 7.2). The fixture shall laterally support the test piece in a slide-fit and shall permit accurate centring of the applied load (i.e. cord position) during testing. If desired, the fixture can possess particular features to facilitate the centring of the test cord in the test hole. Suitable types of fixture are depicted in Figure 6.

5.5 Jaws, for gripping the cord to be extracted in the test machine. These can be of the wedge, pneumatic, pneumatic/hydraulic or bollard type, arranged so that the force applied to the cord during testing is normal to the face of the test piece.

6 Calibration

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

7 Test piece

7.1 Form

The test piece shall have one of the general forms shown in Figure 7.

7.2 Dimensions

Two types of test piece are specified for method 1, depending on the wire cord diameter. Their dimensions shall comply with Table 1. The dimensions of the test piece for method 2 shall comply with Table 2. For both methods, for cords with diameters below 0,5 mm or above 1,7 mm, or for smaller cords which experience cord breakages with rubber compounds showing very high adhesion strength, the same form of test piece shall be used, with the dimensions h and L suitably scaled.

Table 1 — Dimensions of test piece for method 1

Dimensions in millimetres

Type	Cord diameter, d	Embedded length, L		Min. width of rubber between reinforcing sheets, w_{\min}	Min. cord spacing, S^a	Test hole diameter or diagonal, h^a
		Using steel plate reinforcement	Using steel cord fabric reinforcement			
A	0,5 to 1,0	10,0	10,0	6,0 ^b	62,5 % of L	85 % of L
B	1,0 to 1,7	10,0	16,0	6,0 ^b	62,5 % of L	85 % of L

^a The minimum cord spacing S and the test hole diameter or diagonal h shall be read to the nearest 0,5 mm.

^b Greater widths are preferred when using steel cord fabric reinforcement.

Table 2 — Dimensions of test piece for method 2

Dimensions in millimetres

Cord diameter, d	Embedded length, L	Test piece width, W	Min. cord spacing, S	Test hole diameter, h
0,5 to 1,7	12,5	12,5	12,5	12,5

7.3 Preparation

7.3.1 General

Prepare all materials before commencing the build-up of the test piece, so that the mould can be filled quickly at the proper time. The operator shall wear clean gloves during the preparation of test pieces. The cords, rubber and moulded test piece shall be properly identified throughout.

NOTE An approximately 5 % excess of rubber compound is employed so that the test piece is formed under pressure with accompanying rubber flow and consolidation.

7.3.2 Method 1

7.3.2.1 For each multiple-corded test piece prepare the following:

- Two pieces of reinforcement material, cut to the correct size to fit the mould.
- Two sets of rubber compound, cut to the correct sizes to fit the mould above and below the cords: the exact sizes of the top and bottom set will depend on the mould used. Each set can consist of one thick strip or of several thinner strips to make up the correct thickness. Remove any protective polyethylene film used during storage and, if necessary, freshen the surfaces with solvent (see 4.2). If solvent is used, allow sufficient time for the surface to dry completely.
- Sufficient wire cords to occupy all stations in the mould. These shall be at least 300 mm long. Only touch the cords close to their ends, and not in the region to be moulded to the rubber. If required, the cord ends can be covered by a veneer or solder or cement to prevent fraying: if so, the solder shall be applied before cutting the wire cord off the spool. Alternatively, one endless cord can be used in conjunction with a suitable tensioning frame.

7.3.2.2 If required, preheat the mould, including all component parts, to about 100 °C.

7.3.3 Method 2

The test piece preparation procedure follows that of method 1, except that the mould spacers and the reinforcement strips are omitted, and that test piece construction prior to vulcanization is carried out on a special building rack similar to the mould (see Figure 4).

The unvulcanized formed test pieces shall be carefully removed from the building rack by pressing uniformly from the rear, placed in a desiccated container and stored at standard laboratory temperature (see ISO 23529) until vulcanization. Test pieces shall be vulcanized within a maximum of 12 h.

7.3.4 Both methods

Place the made-up mould in the press already set at the vulcanization temperature. Allow the mould to warm up so that the rubber temperature is about 100 °C and the rubber is ready to flow. Apply a force of at least 100 kN and maintain for the duration of the vulcanization.

NOTE In order to set the conditions to achieve a rubber temperature of 100 °C, it might be necessary to carry out a preliminary calibration using a thermocouple inserted into the rubber.

Either pass cooling water through the platens of the press for a suitable time, release the moulding force and remove the mould from the press, or allow the mould to cool after its removal from the press. Extract the test piece, using the relevant extraction tools, if appropriate, and avoiding any deformation of the test piece.

Examine the test piece to ensure that rubber has flowed completely along each cord-hole in the pairs of steel spacers for the method 1 test pieces.

Separate the blocks if appropriate. Trim off the shorter lengths of wire cord close to the surface of the rubber block and any spew or flash on the outer edges of the block. With test pieces for method 1, the flash near the cords need not be trimmed as it does not affect the test procedure or result; for method 2 test pieces, trim any flash with a razor blade or suitable clippers, taking care not to damage the cord or the body of the test piece.

The test piece shall be rested at standard laboratory temperature (see ISO 23529) for a minimum of 16 h before testing if no time is specified.

8 Procedure

Tests shall be performed at standard laboratory temperature (see ISO 23529) unless otherwise specified.

Mount the test piece in the tensile-testing machine using the relevant fixture shown in Figure 6. Carefully adjust the test piece so that the first wire cord is central in the hole, thus ensuring that the test tension will be uniformly distributed round the circumference of the cord (see Figures 6 and 8). This centring is facilitated by the use of the aligning features already mentioned in 5.1 a) and 5.4. Clamp the cord to be tested in the extraction jaws.

Apply tension by separating the extraction jaws from the fixture at a constant rate, between 50 mm/min and 150 mm/min, until the test piece breaks. Record the maximum force.

Repeat the procedure for each of the remaining cords in the test piece: at least 10 cords shall be tested.

9 Expression of results

For each test cord, calculate the adhesion strength by dividing the maximum force by the embedded length of the test piece, and express the result in newtons per millimetre or kilonewtons per metre to the nearest integer.

Determine the mean value and standard deviation for each set of test conditions.

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Examine each broken test piece and, if required, express the adhesion failure by one or the other of the following symbols:

R — indicating that the failure is in the rubber;

M — indicating that the failure is at the interface between the cord and the rubber and that the bare cord surface is visible.

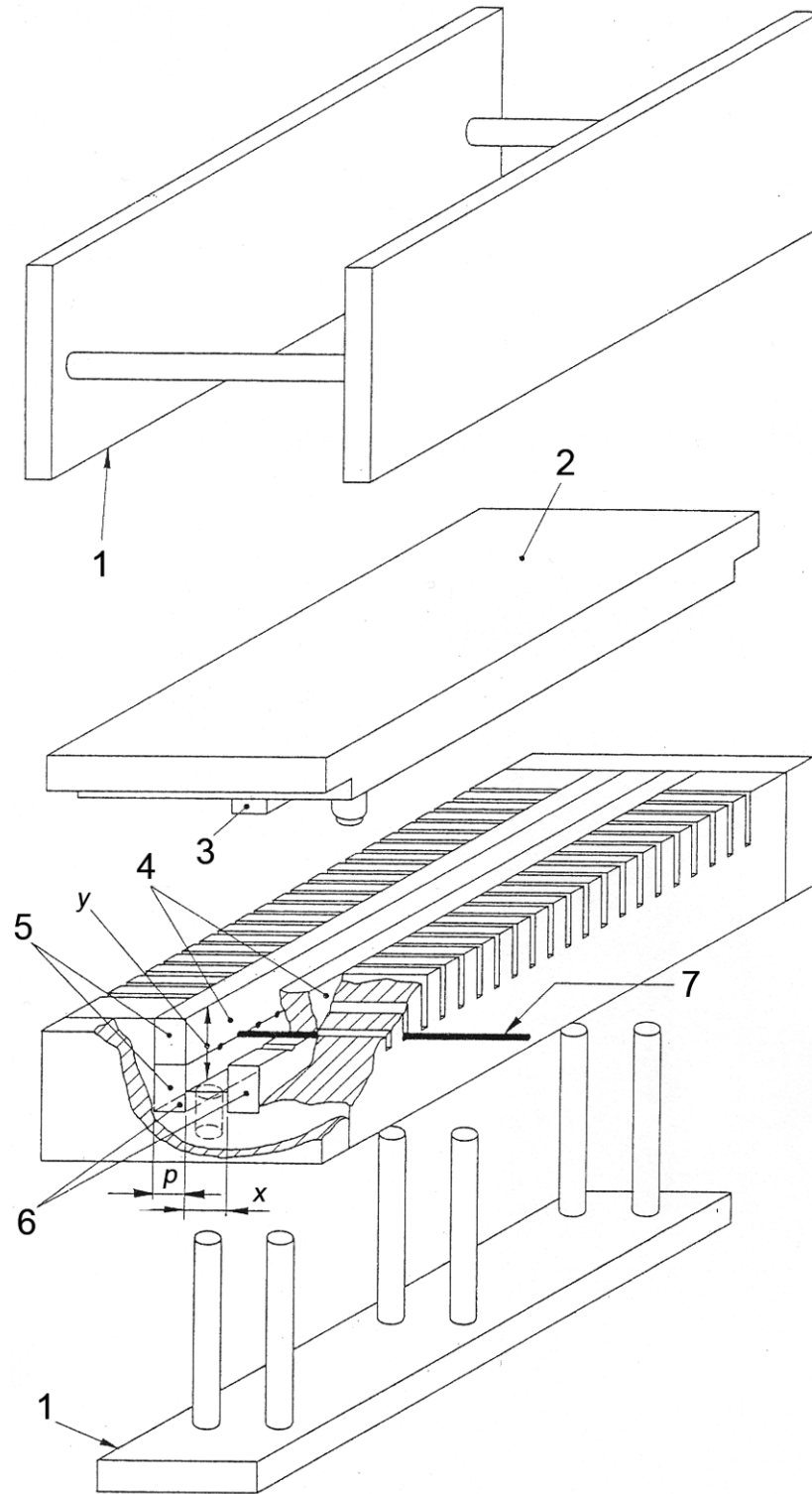
The symbols R and M shall be completed with percentages, the results being expressed as “percent coverage” in steps of 25 %.

EXAMPLE 25 R/75 M means that 75 % of the cord surface is visible.

10 Test report

The test report shall include the following information:

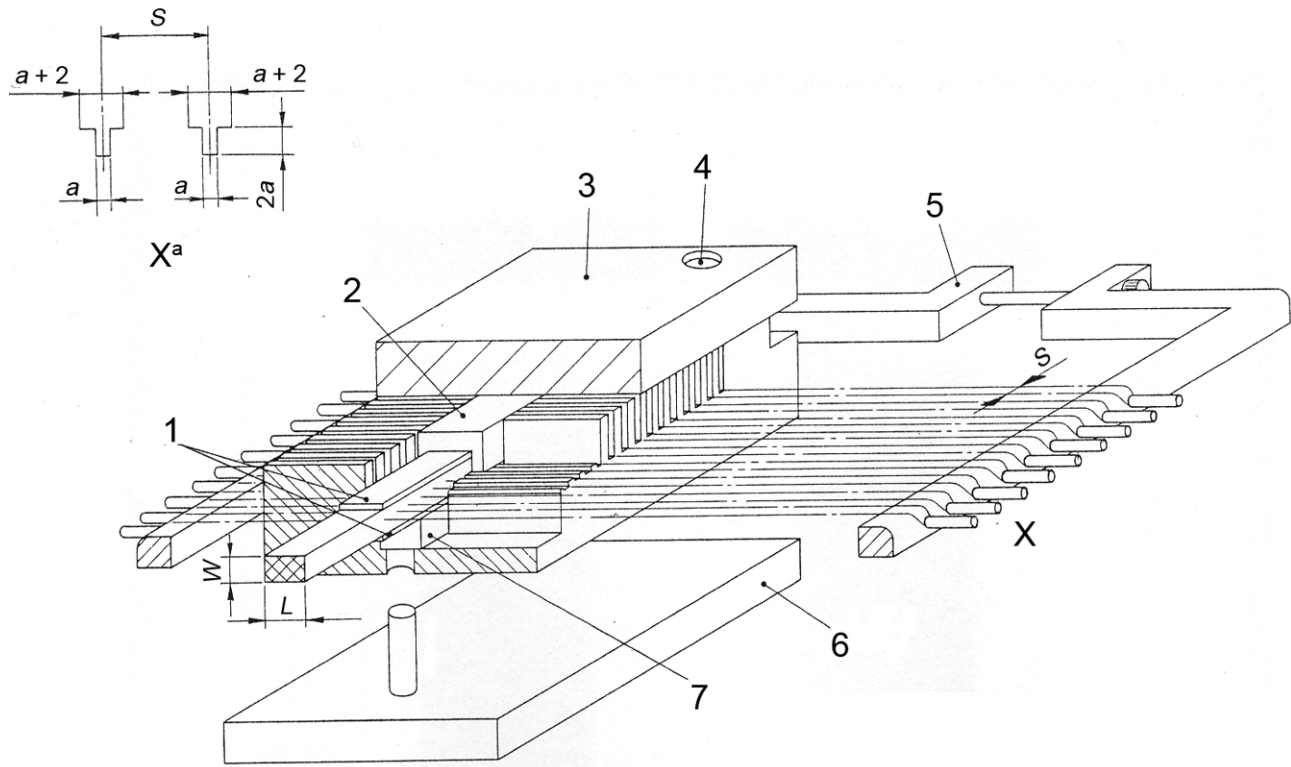
- a) a description and identification of the sample tested, including
 - 1) a description and identification of the wire cord,
 - 2) a description and identification of the rubber compound,
 - 3) whether solvent was used to wipe the surface of the rubber (see 4.2),
 - 4) the time, temperature and date of vulcanization;
- b) a reference to this International Standard;
- c) the method used (1 or 2);
- d) details of the conditions of test, including the temperature and humidity used for conditioning and testing;
- e) the results and the units in which they are expressed, including
 - 1) the number of cords tested,
 - 2) the individual test results,
 - 3) the mean value and standard deviation;
- f) details of any operation not included in this International Standard, or regarded as optional;
- g) the date of the test.



Key

- 1 extraction tool
- 2 semi-follow-on lid
- 3 ram
- 4 top spacers
- 5 pair of steel spacers
- 6 bottom spacers
- 7 steel cord

Figure 1 — Layout of one type of semi-follow-on mould and its extraction tools for method 1



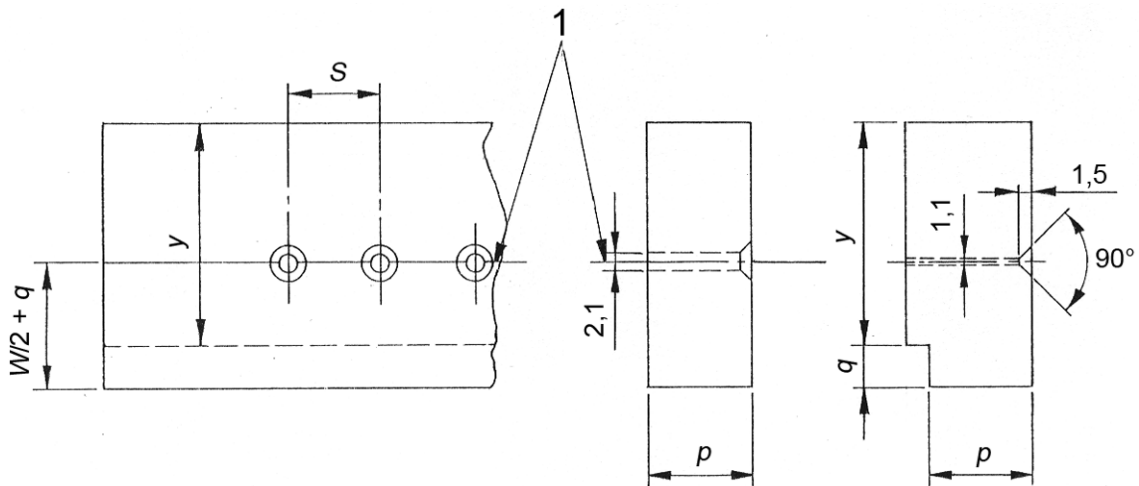
Key

- 1 metal reinforcement sheets (thickness t)
- 2 upper ledge/spacer
- 3 cover plate
- 4 centring bolts
- 5 tensioning frame
- 6 extraction tool
- 7 lower edge

^a Detail of cross-section of slot (for dimension a , see Figure 3).

Figure 2 — Layout of a second type of semi-follow-on mould and its extraction tool for method 1

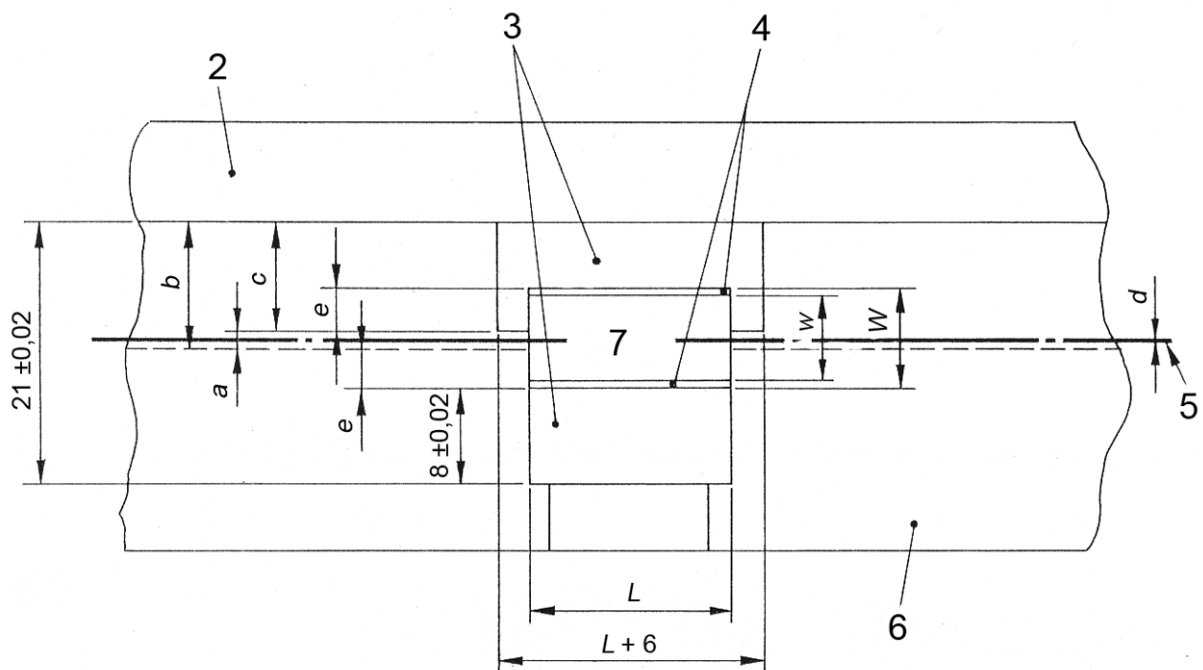
Dimensions in millimetres



a) Spacers for the mould in Figure 1

Cord diameter <i>d</i>	Details of ledge chosen			
	<i>a</i>	<i>b</i>	<i>c</i>	<i>e</i>
≥0,5 but <0,7	0,7 ± 0,02	9,85 ± 0,2	9,15 ± 0,02	3,15 ± 0,02
≥0,7 but <1,0	1,0 ± 0,02	10,0 ± 0,2	9,0 ± 0,02	3,0 ± 0,02
≥1,0 but <1,4	1,4 ± 0,02	10,2 ± 0,2	8,8 ± 0,02	2,8 ± 0,02
≥1,4 but <1,7	1,8 ± 0,02	10,4 ± 0,2	8,6 ± 0,02	2,6 ± 0,02

$a = b - c$ is the slot width
 b is the slot depth
 $2e + a = w + 2t = W$
 $e = 21 - (b + 8) = 13 - b$



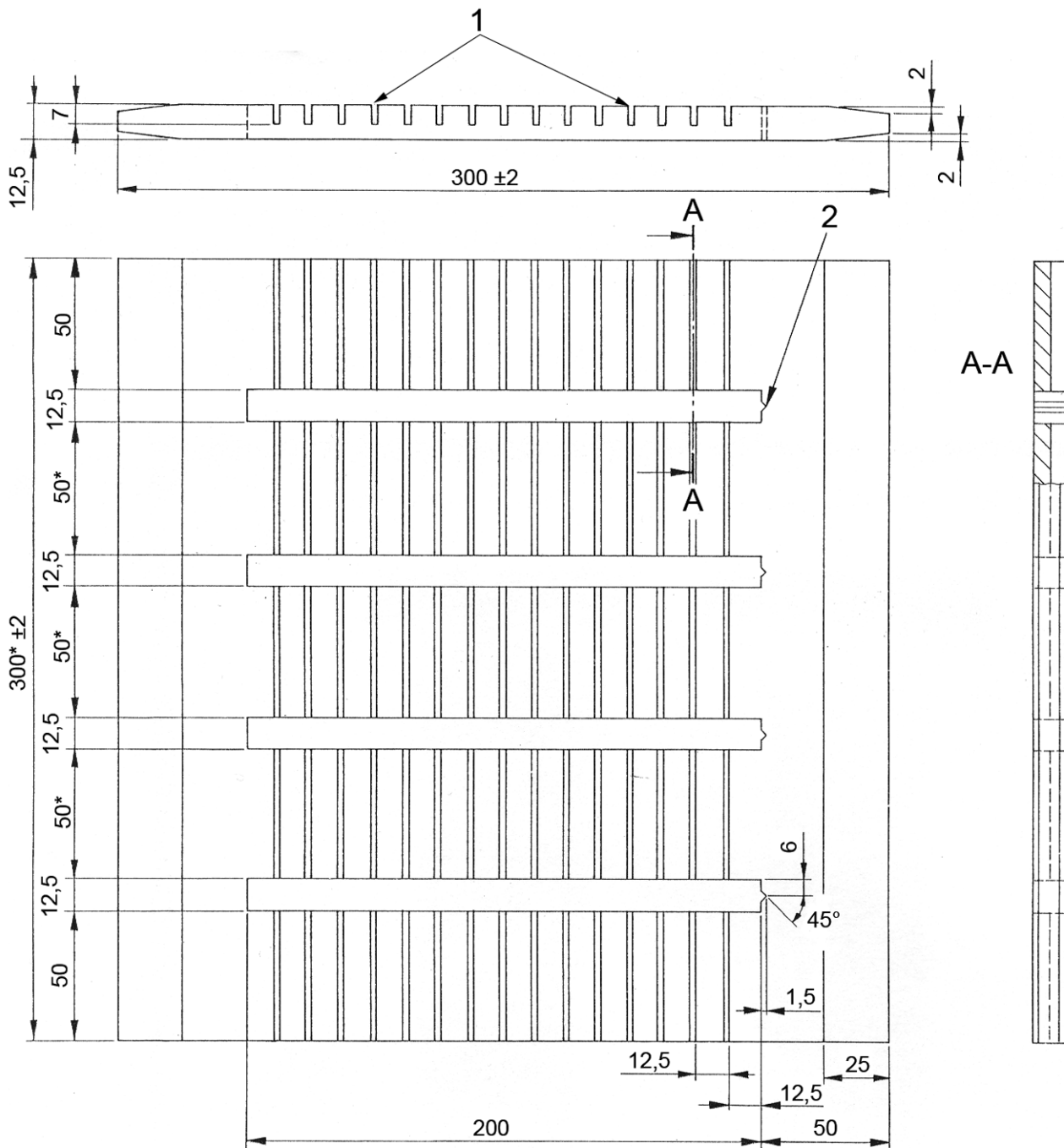
b) Exchange ledges/spacers for the mould in Figure 2

Key

- | | |
|--|-------------------|
| 1 split line | 5 cord |
| 2 cover plate | 6 main mould body |
| 3 ledges/spacers | 7 rubber |
| 4 metal reinforcement sheets (thickness <i>t</i>) | |

Figure 3 — Details of spacers used in the two types of semi-follow-on mould for method 1

Dimensions in millimetres



Key

- 1 15 slots (1,8 ± 0,1) mm wide
- 2 identification number

NOTE 1 Material — mild steel. Can be flash chrome-plated.

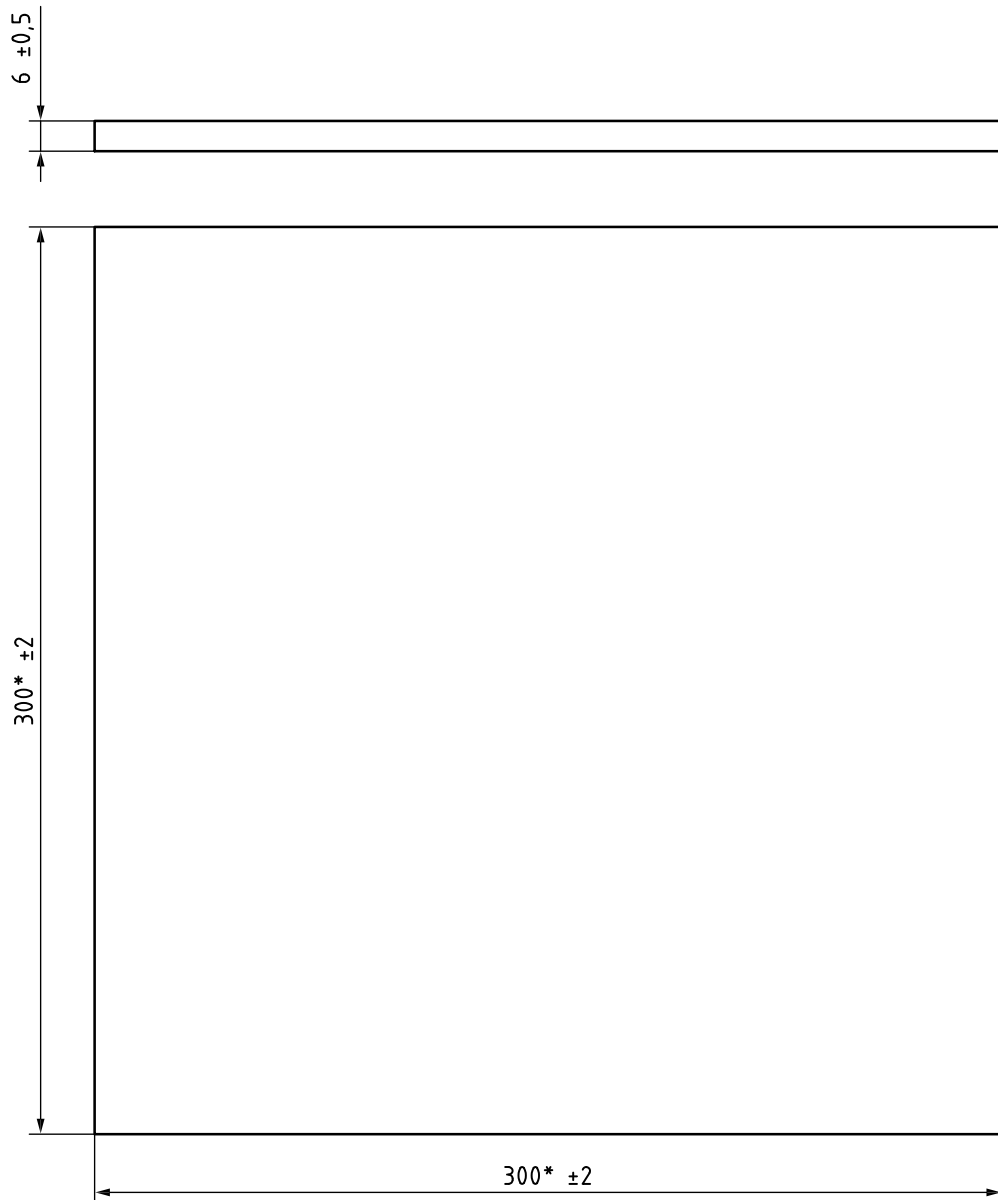
NOTE 2 Dimensions marked with an asterisk (*) can be altered to accommodate the test grips.

Tolerances (except where otherwise stated):

- dimensions ± 0,2;
- angles ± 2°.

Figure 4 — Mould for method 2

Dimensions in millimetres

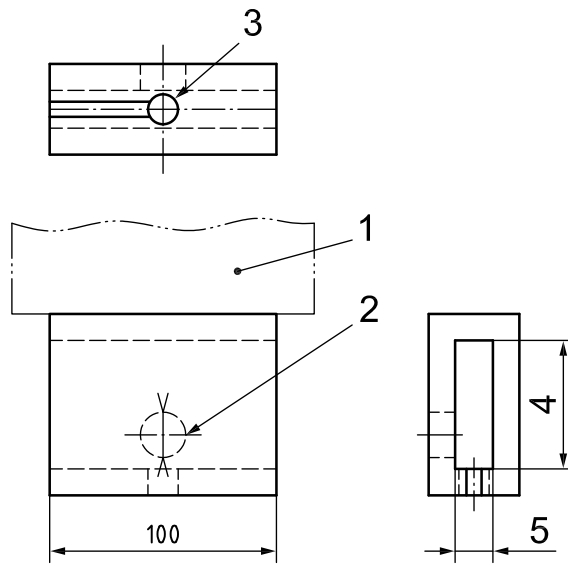


NOTE 1 Material — mild steel.

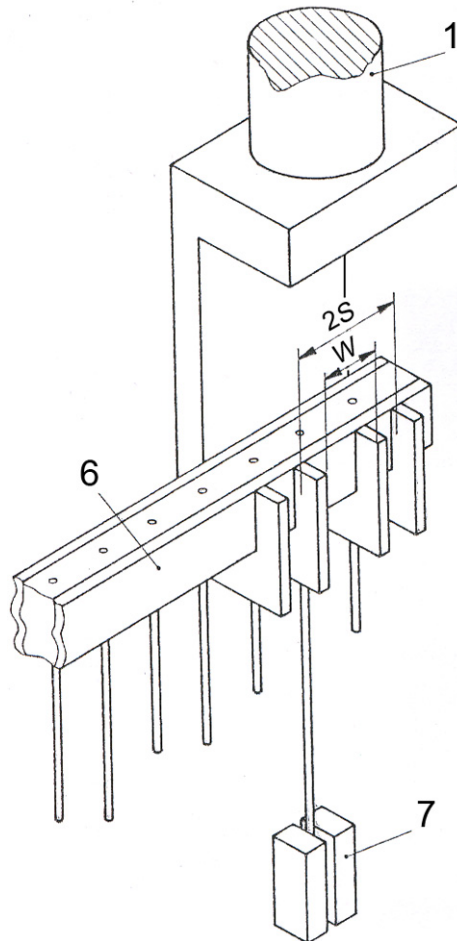
NOTE 2 Number required: 2.

NOTE 3 Dimensions marked with an asterisk (*) can be altered to accommodate the test grips.

Figure 5 — Mould for method 2



a) Fixture with circular hole

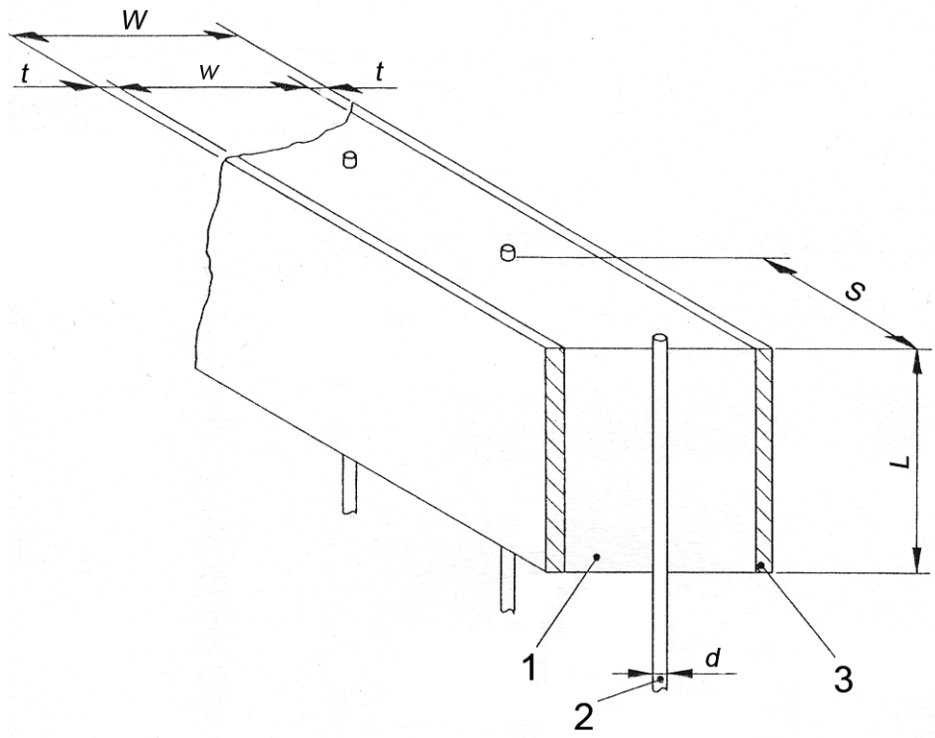


b) Fixture for testing with square hole, showing test arrangement

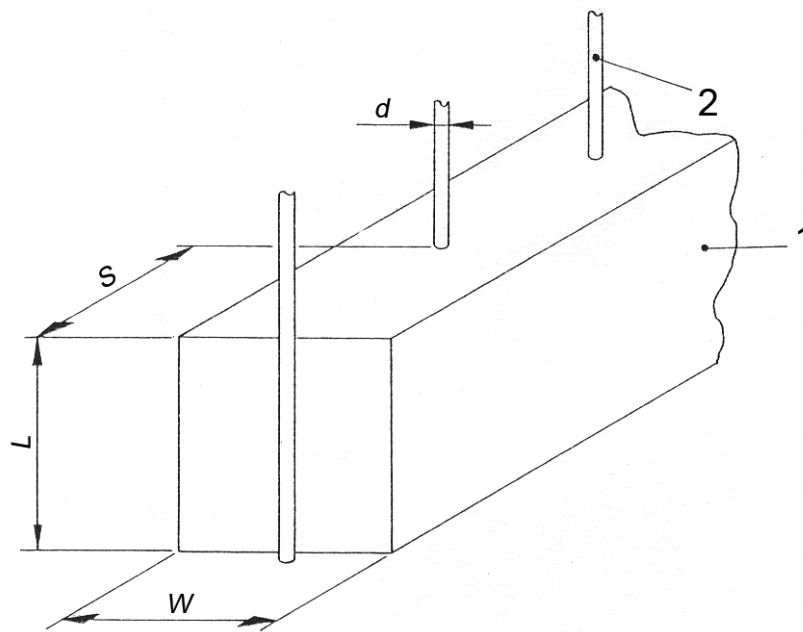
Key

- | | | |
|---|--------------------------------|--------------------------------|
| 1 to fit one jaw of tensile-testing machine | 4 to suit dimension L | 7 cord clamp to fit second jaw |
| 2 optional lateral hole with centring marks | 5 to suit test piece width W | |
| 3 slotted hole (diameter h) | 6 test piece | |

Figure 6 — Typical fixtures



a) Method 1

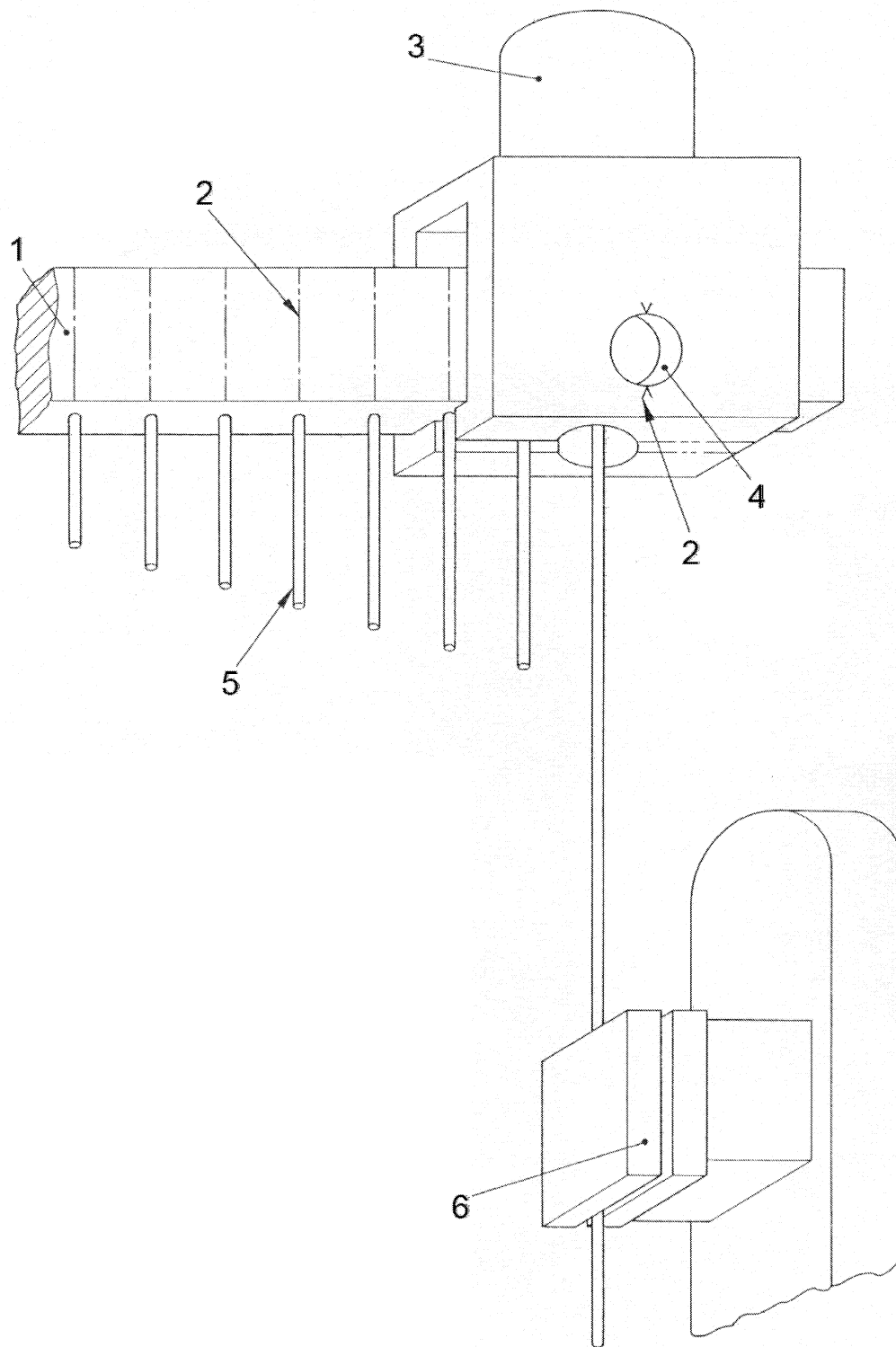


b) Method 2

Key

- 1 rubber
- 2 wire cord
- 3 reinforcement strip of sheet metal or steel cord fabric

Figure 7 — Test pieces



Key

- | | | | |
|---|---|---|------------------------------|
| 1 | test piece | 4 | optional lateral hole |
| 2 | optional centring lines | 5 | wire cords |
| 3 | to fit one jaw of tensile-testing machine | 6 | cord clamp to fit second jaw |

NOTE Test arrangement can be inverted if required.

Figure 8 — Test arrangement (circular hole)

Annex A (normative)

Calibration schedule

A.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 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.

A.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 A.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement might relate to the main test apparatus, to part of that apparatus or to an ancillary item of 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 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
- N Initial verification only
- S Standard interval as given in ISO 18899

Table A.1 — Calibration frequency schedule

Parameter	Requirement	Subclause in ISO 18899:2004	Verification frequency guide	Notes
Mould	As described in 5.1	15.2	N	
Vulcanizing press	As specified in ISO 2393	C	N	
Tensile-testing machine	Complying with ISO 5893	21.1	S	
Force measurement	Class 2 as defined in ISO 5893	21.2	S	
Rate of separation of jaws	Constant rate in the range 50 mm/min to 150 mm/min	23.4	S	See ISO 5893 for tolerances
Fixture for holding test piece	As specified in 5.4	C	N	
Jaws	As specified in 5.5	C	N	
Materials:				
— wire cord	As specified or brass-coated steel of construction (1 × 3 × 0,15) mm or (7 × 4 × 0,22) mm			
— unvulcanized rubber compound	As specified, preferably freshly milled			
— solvent, needed if rubber not freshly milled (see 4.2)	Heptane (preferred) or petroleum solvent (distillation range 65 °C to 125 °C), maximum residue 3 mg/100 ml of solvent			
— reinforcement material	Either metal strip, thickness ≥ 0,5 mm, treated with adhesive promoter, or fabric with steel cords as specified in 4.3			

In addition to the items listed in the table, use of the following is implied, all of which need calibrating in accordance with ISO 18899:

- a timer;
- a thermometer for monitoring the moulding and test temperatures;
- instruments for determining the dimensions of the test pieces.

ICS 83.060

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