

BS ISO 4649:2010



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# Rubber, vulcanized or thermoplastic — Determination of abrasion resistance using a rotating cylindrical drum device

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# INTERNATIONAL STANDARD

BS ISO 4649:2010

**ISO**  
**4649**

Third edition  
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## **Rubber, vulcanized or thermoplastic — Determination of abrasion resistance using a rotating cylindrical drum device**

*Caoutchouc vulcanisé ou thermoplastique — Détermination de la  
résistance à l'abrasion à l'aide d'un dispositif à tambour tournant*



Reference number  
ISO 4649:2010(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 4649 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 4649:2002), which has been technically revised. The main changes concern the addition of a calibration schedule (see Annex C), updating the normative references and moving the precision statement to Annex D.

# Rubber, vulcanized or thermoplastic — Determination of abrasion resistance using a rotating cylindrical drum device

**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 two methods for the determination of the resistance of rubber to abrasion by means of a rotating cylindrical drum device.

The methods involve determination of the volume loss due to the abrasive action of rubbing a test piece over a specified grade of abrasive sheet. Method A is for a non-rotating test piece and method B for a rotating test piece. For each method, the result can be reported as a relative volume loss or an abrasion resistance index.

Because factors such as the grade of abrasive sheet, the type of adhesive used in the manufacture of the sheet and contamination and wear caused by previous testing lead to variations in the absolute values of abrasion loss, all tests are comparative. Runs with a reference compound are included so that the results can be expressed either as a relative volume loss compared to a calibrated abrasive sheet or as an abrasion resistance index compared to a reference compound.

**NOTE** The abrasion loss is often more uniform using the rotating test piece because the whole surface of the test piece is in contact with the abrasive sheet over the duration of the test. However, there is considerable experience using the non-rotating test piece.

These test methods are suitable for comparative testing, quality control, specification compliance testing, referee purposes, and research and development work. No close relation between the results of this abrasion test and service performance can be inferred.

## 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 2230, *Rubber products — Guidelines for storage*

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

ISO 2781, *Rubber, vulcanized or thermoplastic — Determination of density*

ISO 7619-1, *Rubber, vulcanized or thermoplastic — Determination of indentation hardness — Part 1: Durometer method (Shore hardness)*

ISO 9298:1995, *Rubber compounding ingredients — Zinc oxide — Test methods*

ISO 18899:2004, *Rubber — Guide to the calibration of test equipment*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

#### 3.1 abrasion resistance

resistance to wear by mechanical action upon a surface

NOTE For the purposes of this International Standard, the abrasion resistance is expressed either as a relative volume loss compared to an abrasive sheet calibrated using a standard reference compound, or as an abrasion resistance index compared to a reference compound.

#### 3.2 relative volume loss

$\Delta V_{\text{rel}}$

volume loss of the test rubber after being subjected to abrasion by an abrasive sheet which will cause a reference compound to lose a defined mass under the same specified conditions of test

NOTE 1 A value of 200 mg has been established as the mid-point of the calibration range (see B.2.4.3) for the abrasive sheet using method A with standard reference compound No. 1 (see B.2) and considerable experience has been accumulated using the relative volume loss calculation in 9.2. A relative volume loss can be calculated for method B (rotating test piece), or with either test method with another reference compound, if the defined mass loss is known. [150 mg has been indicated as a possible value for method B with standard reference compound No. 2 (see B.3), but its accuracy has not been documented to the degree of the value 200 mg using method A with standard reference compound No. 1 (see B.2).]

NOTE 2 Relative volume loss is expressed in cubic millimetres.

#### 3.3 abrasion resistance index ARI

$I_{\text{AR}}$

ratio of the volume loss of a reference compound to the volume loss of the test rubber, measured under the same specified conditions of test and expressed as a percentage

NOTE A smaller number indicates a lower abrasion resistance.

### 4 Principle

A cylindrical rubber test piece is made to slide over an abrasive sheet of specified abrasive grade at a specified contact pressure over a given distance. The test piece may be non-rotating or rotating during the test.

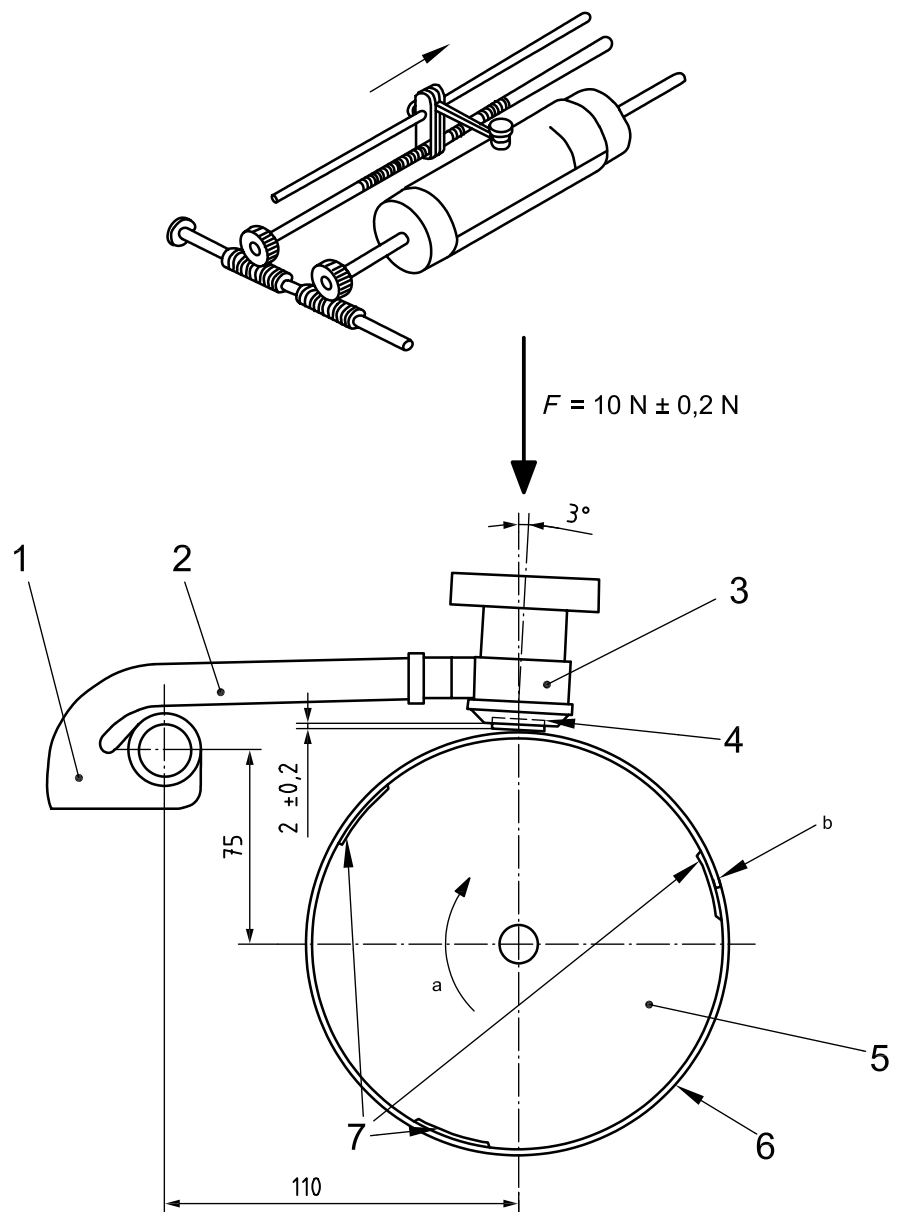
Abrasion takes place over one of the end surfaces of the cylindrical test piece (see Figure 1). The abrasive sheet is attached to the surface of a rotating cylindrical drum against which the test piece is held and across which it is traversed.



The loss in mass of the test piece is determined and the volume loss is calculated from the density of the material used for the test piece. The volume loss of the test piece is compared to that of a reference compound tested under the same conditions.

A very important part of this method is the preparation of the abrasive sheet and its calibration using standard reference compound No. 1 (see B.2) with a non-rotating test piece.

Dimensions in millimetres



**Key**

- |   |   |
|---|---|
| 1 sledge  | 6 abrasive sheet                              |
| 2 swivel arm  | 7 double-sided adhesive tape                  |
| 3 test piece holder                                 | <i>F</i> vertical force                       |
| 4 test piece  | <i>a</i> Rotational speed 40 r/min ± 1 r/min. |
| 5 cylinder, diameter 150 mm ± 0,2 mm, length 500 mm | <i>b</i> Gap of size ≤ 2 mm.                  |

**Figure 1 — Schematic illustration of apparatus**

## 5 Apparatus and materials

### 5.1 Abrasion machine

The test apparatus (see Figure 1) consists of a laterally movable test piece holder and a rotatable cylinder to which the abrasive sheet (5.2) is fixed.

The cylinder shall have a diameter of  $150 \text{ mm} \pm 0,2 \text{ mm}$  and a length of about 500 mm and shall be rotated at a speed of  $40 \text{ r/min} \pm 1 \text{ r/min}$ , the direction of rotation being as indicated in Figure 1.

The test piece holder shall have a cylindrical opening, the diameter of which can be adjusted from 15,5 mm to 16,3 mm, and a device for adjusting the length of the test piece protruding from the opening to  $2 \text{ mm} \pm 0,2 \text{ mm}$ . The holder shall be mounted on a swivel arm that, in turn, is attached to a sledge that can be moved laterally on a spindle. The lateral displacement of the holder shall be  $4,20 \text{ mm} \pm 0,04 \text{ mm}$  per revolution of the drum (see Note 1). Suitable attachments may be provided to rotate the test piece during the test run by rotation of the test piece holder (method B), preferably at the rate of 1 revolution per 50 revolutions of the drum.

NOTE 1 With this lateral movement, the test piece passes over any one area of the abrasive sheet four times.

The central axis of the holder shall have an inclination of  $3^\circ$  to the perpendicular in the direction of rotation (see Figure 1), and shall be placed directly above the longitudinal axis of the cylinder to within  $\pm 1 \text{ mm}$ .

The swivel arm and test piece holder shall be free from vibration during operation, and so disposed that the test piece is pressed against the drum with a vertical force of  $10 \text{ N} \pm 0,2 \text{ N}$ . For investigation of extremely soft or hard materials, the force may be changed to  $5 \text{ N} \pm 0,1 \text{ N}$  or  $20 \text{ N} \pm 0,4 \text{ N}$  respectively (see Note 2). The force is generated by adding masses to the top of the test piece holder.

NOTE 2 A force of 5 N is typically used for rubbers softer than approximately 40 IRHD and a force of 20 N is used for hard rubbers of 80 IRHD and above.

The abrasive sheet shall be attached to the drum using three evenly spaced strips of double-sided adhesive tape extending along the complete length of the drum. The width of the margins that are not touched by the test piece shall be equal. Care shall be taken to ensure that the abrasive sheet is firmly held so as to present a uniform abrasive surface over the whole area of the cylinder. One of the strips shall be placed where the ends of the abrasive sheet meet. Ideally, the ends should meet exactly, but any gap left between them shall not exceed 2 mm. The adhesive tape shall be about 50 mm wide and not more than 0,2 mm thick.

The abrasion run starts by bringing the test piece into contact with the abrasion sheet.

Placement of the test piece on the sheet at the beginning of a test run, and its removal after an abrasion run of  $40 \text{ m} \pm 0,2 \text{ m}$  (equivalent to 84 revolutions), shall be automatic. In special cases of very high volume loss of the test piece, the abrasion distance may be reduced to  $20 \text{ m} \pm 0,1 \text{ m}$  (equivalent to 42 revolutions). In that case, a revolution counter or automatic stopping device should preferably be used.

NOTE 3 For rubbers with very high mass loss, a distance of 10 m has been used.

To protect the abrasive sheet from damage by the test piece holder, a device for switching off the apparatus just before the lower edge of the test piece holder touches the sheet is recommended.

The test machine may be equipped with a vacuum hose and a brush to aid in the removal of debris from the machine.

### 5.2 Abrasive sheet

Abrasive sheet made with aluminium oxide of grain size 60, at least 400 mm wide,  $474 \text{ mm} \pm 1 \text{ mm}$  long, and 1 mm average thickness, shall be used as the abrasive medium.

In a test using a non-rotating test piece of standard reference compound No. 1 (see B.2), this abrasive sheet shall cause a mass loss of between 180 mg and 220 mg for an abrasion distance of 40 m.

When each new sheet is first used, the direction of motion shall be indicated on the sheet, as it is important that the same direction be used for all subsequent test runs.

Notes on a suitable abrasive sheet are given in Annex A.

### 5.3 Hollow drill (see Figure 2)

The drill shown in Figure 2 is an example of a tool suitable for the preparation of test pieces that are not moulded (see 7.1). The speed of rotation of the drill needs to be at least 1 000 r/min for most rubbers, and even higher for rubbers with a hardness of less than 50 IRHD. A sufficient cooling of the tool is necessary to prevent an undesired heating of the drill.

Dimensions in millimetres

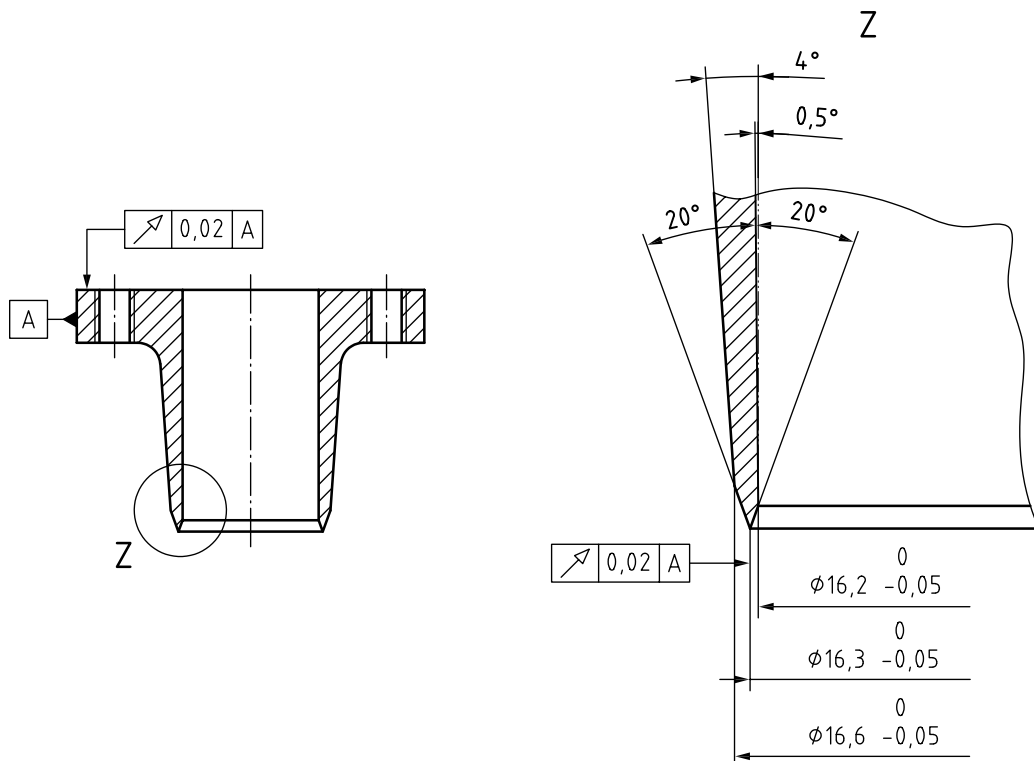


Figure 2 — Example of a hollow drill for test piece preparation

### 5.4 Balance

The balance shall be of sufficient accuracy to enable the mass loss of a test piece to be determined to  $\pm 1$  mg.

### 5.5 Standard reference compounds

Specifications for standard reference compounds are given in detail in Annex B.

## 6 Calibration

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

## 7 Test pieces

### 7.1 Type and preparation

The test pieces shall be cylindrical in shape, of diameter  $16 \text{ mm} \pm 0,2 \text{ mm}$ , with a minimum height of 6 mm.

Test pieces are normally prepared from moulded sheet using the hollow drill (5.3) or other rotating cutting tool. During cutting, the cutting edge shall be lubricated with water to which a wetting agent has been added. Punching of the test pieces is not permitted.

NOTE If the cut sample is not satisfactory, cylinder vibration occurs.

Alternatively, test pieces may be vulcanized or formed in a mould.

If test pieces of the required thickness are not available, the necessary thickness may be obtained by bonding a piece of the test rubber to a base element of hardness not less than 80 IRHD. The thickness of the test rubber shall be not less than 2 mm.

### 7.2 Number

A minimum of three test runs shall be carried out for each rubber being tested. A new test piece shall be used for each test run. For referee purposes, use 10 test pieces.

In the case of the standard reference compounds No. 1 (see B.2) and No. 2 (see B.3), three test runs may be carried out on one test piece in order to reduce wastage.

### 7.3 Time interval between vulcanization or forming and testing

For all test purposes, the minimum time between vulcanization or forming 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, as far as possible, shall be carried out after the same time interval. For product tests, whenever possible, the time between vulcanization or forming 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.

### 7.4 Conditioning

Condition all test pieces at standard laboratory temperature, in accordance with ISO 23529, for a minimum period of 16 h immediately before testing.

For rubbers which are sensitive to moisture, the humidity should also be controlled.

## 8 Test temperature

The test shall be carried out at standard laboratory temperature (see ISO 23529).

During a test run, there may be a considerable increase in temperature at the abrading interface, which may lead to temperature rises within the test piece. For the purposes of this International Standard, such temperature rises are to be disregarded, the temperature of test being that of the ambient atmosphere and of the test piece before commencing the test.

## 9 Procedure

### 9.1 General test procedure

Before each test, any rubber debris left on the abrasive sheet from a previous abrasion test shall be removed with a brush. A brush of about 55 mm diameter with hard nylon or similar bristles (see Note 1) and about 70 mm in length is recommended for this purpose. In some cases, a blank test with a reference compound (see Note 1) will effectively clean the abrasive sheet (see Note 2).

Brushes with metal bristles are not recommended as they shorten the life of the abrasive sheet.

NOTE 1 Reference compound used only for cleaning purposes does not necessarily have to meet the stringent requirements of the reference compound used for test purposes.

NOTE 2 Some laboratories have found that blowing removes the debris left by some test rubbers better than brushing. Safety blow guns which give a maximum pressure of 0,2 MPa at the nozzle when the nozzle is blocked off, used with a supply air pressure between 0,5 MPa and 0,9 MPa, have given good results.

For method A, the non-rotating test piece shall be used. For method B, the rotating test piece shall be used. Standard reference compound No. 1 (see B.2) or No. 2 (see B.3) or a user-defined reference compound shall be used as the reference compound. The method and the reference compound used shall be stated in the test report, because the results obtained can differ. For measurements intended to be comparable, the same conditions shall be used for all test rubbers and the reference compound.

Weigh the test piece to the nearest 1 mg. Fix the test piece in the test piece holder in such a way that a length of  $2,0 \text{ mm} \pm 0,2 \text{ mm}$  protrudes from the opening. This length shall be checked by means of a gauge.

The test piece shall normally be pressed against the drum with a vertical force of  $10 \text{ N} \pm 0,2 \text{ N}$ . If, for special cases, the vertical force is reduced to  $5 \text{ N} \pm 0,1 \text{ N}$  or increased to  $20 \text{ N} \pm 0,4 \text{ N}$ , this shall be stated in the test report.

Turn on the suction, if it is provided. Move the test piece holder and sledge to the starting point and start the automatically controlled test run. Check for vibration in the test piece holder. This test method does not yield meaningful results if there is abnormal vibration in the test piece holder. The test run is stopped automatically after an abrasion distance of 40 m. When relatively large mass losses (usually more than 400 mg in 40 m) occur, the test run may be stopped after 20 m, and the length of exposed test piece reset to  $2,0 \text{ mm} \pm 0,2 \text{ mm}$  so that the remaining 20 m of the run may be completed. At no time shall the height of the test piece be less than 5 mm. If the mass loss is greater than 600 mg in 40 m, the abrasion distance shall be reduced to 20 m and this shall be stated in the test report. The results shall be multiplied by 2 so that the mass loss can still be given for an abrasion distance of 40 m.

For non-rotating test pieces that are removed during the test, care shall be taken to ensure that the test piece is always replaced in the test piece holder in the same way.

For bonded test pieces, care shall be taken that the test pieces are not abraded down to the bond line or if a sample with a fabric reinforcement is used in fabric line (if necessary, use a 20 m run).

Weigh the test piece to the nearest 1 mg after the test run. Sometimes a small edge hanging from the test piece has to be pulled off before weighing, especially if a non-rotating test piece is used.

Carry out all test runs on the same rubber consecutively.

### 9.2 Comparison against standard reference compounds or user-defined reference compounds

The test rubbers are compared against one of the two standard reference compounds specified in Annex B or a user-defined reference compound.

The mass loss of the reference compound shall be determined by carrying out a minimum of three test runs both before and after each test series with the test rubber following the procedure in 9.1. There shall be a maximum of 10 runs of test rubber test pieces in each test series. Do not split the runs on a test rubber between series. When repeat runs are made on the same test piece of one of the standard reference compounds, allow sufficient time between such runs for the temperature of the whole of the test piece to return to standard laboratory temperature.

For rubbers which have a tendency to smear, determine the mass loss of the reference compound after each run with the test rubber. In extreme cases of smearing, there is a considerable reduction in mass loss of the reference compound measured after the test run compared to that measured before the test run. This is due to the fact that the abrasive sheet is being "cleaned" by the reference compound, as opposed to the reference compound being abraded by the sheet. If the reduction in mass loss of the reference compound is greater than 10 %, then the method is not valid.

Variations in the test method have been proposed to overcome this problem, including the use of a 40 grit abrasive sheet. Any such variations used shall be agreed upon by all parties involved and carefully noted in the test report [see Clause 12, item c) 3)].

### 9.3 Density

Determine the density of the test material by one of the methods specified in ISO 2781.

## 10 Expression of results

### 10.1 General

The results may be expressed either as a relative volume loss or as an abrasion resistance index.

Calculate the mean value of the mass losses of the test rubber,  $\Delta m_t$ , and of the reference compound,  $\Delta m_r$ , from the separate determinations.

### 10.2 Relative volume loss, $\Delta V_{rel}$

The relative volume loss (see 3.2),  $\Delta V_{rel}$ , in cubic millimetres, is given by the equation:

$$\Delta V_{rel} = \frac{\Delta m_t \Delta m_{const}}{\rho_t \Delta m_r}$$

where

$\Delta m_t$  is the mass loss, in milligrams, of the test rubber test piece;

$\Delta m_{const}$  is the defined value of the mass loss, in milligrams, of the reference compound test piece (see Note 1 to 3.2);

$\rho_t$  is the density, in milligrams per cubic millimetre, of the test rubber;

$\Delta m_r$  is the mass loss, in milligrams, of the reference compound test piece.

NOTE Standard reference compound No. 1 is usually used with this method of expression of results.

Calculate the mean value of the relative volume loss.

### 10.3 Abrasion resistance index

The ARI (see 3.3),  $I_{AR}$ , as a percentage, is given by the equation:

$$I_{AR} = \frac{\Delta m_r \rho_t}{\Delta m_t \rho_r} \times 100$$

where

$\Delta m_r$  is the mass loss, in milligrams, of the reference compound test piece;

$\rho_r$  is the density, in milligrams per cubic millimetre, of the reference compound;

$\Delta m_t$  is the mass loss, in milligrams, of the test rubber test piece;

$\rho_t$  is the density, in milligrams per cubic millimetre, of the test rubber.

Calculate the mean value of the abrasion index.

## 11 Precision

See Annex D.

## 12 Test report

The test report shall include at least the following information:

- a) sample details:
  - 1) full description of the sample and its origin,
  - 2) compound details and curing or forming conditions, if known,
  - 3) method of preparation of the test pieces from the sample, i.e. whether cut or moulded;
- b) test method
  - 1) full reference to the test method used, i.e. reference to this International Standard (ISO 4649:2010),
  - 2) method used (A or B);
- c) test details:
  - 1) standard laboratory temperature used,
  - 2) whether standard reference compound No. 1 (see B.2) or No. 2 (see B.3) or a user-defined reference compound was used,
  - 3) any deviations from the normal test procedure, especially if the test run comprised only half the abrasion distance or if half the vertical force was used (see last paragraph in 9.2);
- d) test result:
  - 1) mean value of the relative volume loss and the abrasion resistance index,
  - 2) densities of the reference compound and of the test rubber;
- e) date of the test.

## Annex A (normative)

### Notes on a suitable abrasive sheet

A suitable abrasive sheet comprises corundum particles of grain size 60, i.e. passing through a 60 mesh sieve, bonded to a twill sheet with a phenolic resin. As produced, the abrasive sheet causes an abrasion loss of more than 300 mg when standard reference compound No. 1 specified in B.2 is tested using a non-rotating test piece. It is necessary to perform one or two runs with a steel test piece to reduce the abrasion loss to between 200 mg and 220 mg. This is checked by single runs with two test pieces. The direction of motion shall be indicated on the sheet, as it is important that the same direction be used for all subsequent test runs.

Experience has shown that a minimum of a few hundred runs with standard reference compound No. 1 (see B.2) can be carried out with this type of sheet before the abrasion loss comes down to 180 mg, after which the sheet shall be discarded.

Abrasive sheet produced and standardized in this manner is available commercially.

NOTE Abrasive paper can be used instead of cloth, if it provides comparable results.



## Annex B (normative)

### Standard and user-defined reference compounds

#### B.1 Purpose

Reference compounds are necessary because these abrasion tests are comparative tests. The quality of the reference compounds significantly influences the repeatability and reproducibility of the tests.

The standard reference compound described in B.2 shall be used for the calibration of the abrasive sheet (5.2) using method A because of the considerable experience already gained using it for this purpose. It may also be used as the comparative standard reference compound in test methods A and B. Standard reference compound No. 1 may be obtained commercially.

The compound described in B.3 is representative of a simple tyre tread compound and has had some previous use as a standard reference compound, including use in the international interlaboratory test programme described in Annex D. Standard reference compound No. 2 would normally be prepared by the user. There is a possibility of obtaining it commercially for those users who do not have their own facilities for preparing it.

Users may define other reference compounds as needed for their own use, noting the degree of care in preparation needed for repeatable and reproducible results. Care shall also be taken not to confuse user-defined reference compounds with standard reference compound No. 1 or No. 2 in test reports.

#### B.2 Standard reference compound No. 1 for the calibration of the abrasive sheet and for use as a comparative standard reference compound

##### B.2.1 Composition

Table B.1 — Composition of standard reference compound No. 1

Ingredient	Parts by mass
Natural rubber (TSR L)	100,0
Zinc oxide, class B4c (see ISO 9298:1995, Annex D <sup>a</sup> )	50,0
<i>N</i> -Isopropyl- <i>N'</i> -phenyl- <i>p</i> -phenylenediamine (IPPD) <sup>b</sup>	1,0
Benzothiazyl disulfide (MBTS) <sup>c</sup>	1,8
Carbon black N330 <sup>d</sup>	36,0
Sulfur	2,5
Total	191,3
NOTE Trade names of products and their suppliers are given in footnotes a to d. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the products named.	
<sup>a</sup> Zinkweiss Pharma 8, from Grillo Zinkoxid GmbH. <sup>b</sup> Vulkanox 4010NA <sup>®</sup> , from Lanxess AG. <sup>c</sup> Vulkacit DM/C <sup>®</sup> , from Lanxess AG. <sup>d</sup> Corax N330 <sup>®</sup> , from Evonik Degussa GmbH.	

Equivalent materials may be used provided that the resulting standard sheets meet the requirements of B.2.4.

### B.2.2 Recommended mixing procedure

The following mixing procedure has been found to be suitable to meet the requirements. Other procedures may be used provided that a good dispersion is obtained and that the properties meet the requirements of B.2.4.

Use an internal mixer to mix all ingredients, following the procedure specified in Table B.2. Subsequently, homogenize the batch using a roll mill, as specified in Table B.3.

**Table B.2 — Mixing procedure using an internal mixer**

Mixing chamber volume: 4.6 (determined by means of wheat grains or another suitable methods)	
Chamber filled to: (65 ± 5)%	
Speed: 30 r/min	
Cooling fully operative	
Mass of rubber: 2 000 g	
Mixing step	Elapsed time, min
Add rubber	0
Add zinc oxide, antioxidant, and accelerator, well premixed	7,5
Add carbon black	11
Add sulfur	14
Dump	18
Final temperature of batch: 100 °C to 110 °C	

**Table B.3 — Homogenization of batch using a roll mill**

Roll diameter: 250 mm		
Working width: 400 mm		
Surface temperature of rolls: 50 °C ± 5 °C		
Speeds of rolls: approximately 12,4 r/min and 18,1 r/min		
Milling step	Elapsed time, min	Nip opening, mm
Band hot masterbatch	0	
Make 3 to 4 cuts	1	0,5
Turn the rolled sheet	5	
Sheet off	10	5,0
Final temperature of sheet: approximately 70 °C		

### B.2.3 Vulcanization

Ply up enough milled sheets to give a minimum thickness of 6 mm. Insert the blank into a mould preheated to 150 °C ± 2 °C. Place the mould in a press and subject to several bumping cycles. Slowly apply a pressure of at least 3,5 MPa and vulcanize for 25 min ± 1 min.

The recommended dimensions of the vulcanized sheet are 8 mm × 181 mm × 181 mm which yields approximately 90 test pieces.

## B.2.4 Quality control

### B.2.4.1 General

The procedure for quality control specified in B.2.4.2, B.2.4.3, and B.2.4.4 has been found to be suitable to achieve a consistent level of abrasion loss.

Tests shall be carried out not earlier than 16 h and not later than 7 days after vulcanization.

### B.2.4.2 Reference test pieces

From a sufficient number of sheets, cut, as specified in 7.1, one test piece from each sheet to provide reference test pieces for quality control of further test piece production. These test pieces can be stored in accordance with B.2.5 for up to 3 years.

### B.2.4.3 Mass loss

All measurements of mass loss for quality control shall be made with a separate abrasive sheet that is only used for this purpose. This abrasive sheet is checked using 15 reference test pieces. With each test piece, the mass loss is measured with three runs with a non-rotating test piece and the median reported. The mean,  $\Delta m_{\text{Ref}}$ , of the 15 median values shall be between 180 mg and 220 mg.

This procedure is carried out for every fifth “production run”, which may include several batches produced under the same conditions by the same operator over a period of 1 day or 2 days.

The mass loss,  $\Delta m_{\text{Prod}}$ , of a production run is determined using a representative sheet from each production run. From this sheet, cut out 15 test pieces as specified in 7.1 and, for each test piece, measure the mass loss,  $\Delta m$ , with three runs using a non-rotating test piece and report the median value. From these 15 median values, calculate the mean,  $\Delta m_{\text{Prod}}$ , and the standard deviation. The difference between  $\Delta m_{\text{Prod}}$  and the last  $\Delta m_{\text{Ref}}$  shall be not more than 15 mg.

In order to ensure uniform quality, it is strongly recommended that reference test pieces obtainable commercially be used for the first check, and occasionally later. The difference between  $\Delta m_{\text{Ref}}$  for test pieces produced in-house and the  $\Delta m_{\text{Ref}}$  for test pieces obtained commercially shall be not more than 10 mg.

### B.2.4.4 Hardness

Measure the Shore hardness in accordance with ISO 7619-1 at a minimum of four places on each sheet and report the median value for each sheet.

The mean for all sheets (i.e. all the median values) in a production run shall be  $(60 \pm 3)$  IRHD.

## B.2.5 Storage

Test sheets shall be stored in accordance with ISO 2230. Wrap the sheets with a material (e.g. polyethylene) which protects the sheets from atmospheric attack, and store them in a cool, dark place.

### B.3 Standard reference compound No. 2 (representative of a simple tyre tread rubber) for use as a comparative standard reference compound

#### B.3.1 Composition

Table B.4 — Composition of standard reference compound No. 2

Ingredient	Parts by mass
Natural rubber (TSR L)	100,0
Stearic acid	2,0
Zinc oxide	5,0
Carbon black N330	50,0
<i>N</i> -Isopropyl- <i>N'</i> -phenyl- <i>p</i> -phenylenediamine (IPPD)	1,0
<i>N</i> -Cyclohexylbenzothiazole-2-sulfenamide (CBS)	0,5
Sulfur	2,5
Total	161,0

#### B.3.2 Mixing and vulcanization

The equipment and procedure used for preparation, mixing and vulcanization shall be in accordance with the relevant requirements of ISO 2393. An internal mixer or a mixing mill may be used. Sheets shall be vulcanized at 140 °C for 60 min.

#### B.3.3 Storage

Wrap the sheets with a material that protects the sheets from attack by ozone (e.g. polyethylene), and store them in a cool, dark place.

#### B.3.4 Quality

The mass losses for two different batches of standard reference compound, determined in accordance with Clause 9, shall agree to within  $\pm 10$  %.

## Annex C (normative)

### Calibration schedule

#### C.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded on any calibration report or certificate. It shall be reported whether calibration is 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.

#### C.2 Schedule

Verification or 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 C.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, part of that apparatus or 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 more specific or detailed calibration procedure than that specified 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
- U In use

Table C.1 — Calibration frequency schedule

Parameter	Requirement	Relevant subclause in ISO 18899:2004	Verification frequency guide	Notes
Test apparatus	Figure 1			
Cylinder diameter length	150 mm ± 0,2 mm About 500 mm	15.2 C	N N	
Rotation frequency	40 r/min ± 1 r/min	14.3	S	
Direction of rotation	As Figure 1	C	N	
Test piece holder diameter	Adjustable from 15,5 mm to 16,3 mm	C	N	
Length protruding	Adjustable to 2 mm ± 0,2 mm	15.1	S	
Lateral displacement	4,2 mm/r ± 0,04 mm/r	15.2	S	
Rotation of test piece	Preferably 1 r per 50 drum r	C	N	Not mandatory
Centre axis of holder	3° to perpendicular in direction of rotation	15.9	S	
Position	Within ±1 mm of long axis	15.2	S	
Swivel arm and test piece holder	Free from vibration	C	U	
Vertical force	20 N ± 0,4 N 10 N ± 0,2 N 5 N ± 0,1 N	21.3	S	By adding masses for special purposes
Attachment of abrasive cloth	Three evenly spaced strips of double-sided tape along complete length	C	U	
Double sided tape Thickness	Width about 50 mm ≤ 0,2 mm	C	U	
Join in abrasive Gap	Double sided tape beneath 0 mm to 2 mm; no overlap	C	U	
Test piece holder	No contact with abrasive Automatic protection recommended	C	U	
Placement of test piece on cloth	Automatic	C	N	
Removal of test piece from cloth	Automatic	C	N	
Test run	40 m (84 r) or 20 m (42 r)	23.3	S	To be stopped automatically
New sheet of abrasive	Direction of rotation to be indicated	C	U	
Hollow drill for test piece preparation	Figure 2 and 5.3. Punching not permitted	15.2 and 15.9	S	
Balance	Accurate to ±1 mg	22.1	S	
Brush diameter length	About 55 mm 70 mm	C	U	
Density	Determine using ISO 2781			
Rubber B1	180 mg to 220 mg	19.3	U	
Mass loss of rotating standard rubber B2	About 150 mg (no limits specified)	19.3	U	
Abrasive sheet	Abrasive cloth, aluminium oxide grain size 60, at least 400 mm wide, 473 mm long, and 1 mm average thickness to cause a mass loss of 180 mg to 220 mg on standard reference compound No. 1 (see Annex A for further details). Standard reference compounds are specified in Annex B.			

## Annex D (informative)

### Precision and bias

**D.1** This annex deals with test results obtained in an international interlaboratory programme organized in accordance with ISO 5725-2.

**D.2** The precision results give an estimate of the precision of these test methods with the materials used in the particular interlaboratory test programme as described in the following. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation stating that the parameters are applicable to the group of materials and the specific testing protocols of the test methods.

**D.3** A type 1 international interlaboratory test programme was conducted in 1986 with 16 participating laboratories, using five different vulcanized rubbers at different abrasion levels. The vulcanized rubbers were distributed in sheet form by one laboratory and test pieces were cut from these sheets by each of the participating laboratories. Abrasion tests were conducted in triplicate on each rubber on each of two days, separated by 3 days to 4 days. The test result was reported as the mean of three individual test runs.

**D.4** The precision results are given in Tables D.1 to D.3. The symbols used in the tables are defined as follows:

- $r$  is the repeatability limit, in measurement units;
- $(r)$  is the repeatability, as a percentage (relative);
- $R$  is the reproducibility limit, in measurement units;
- $(R)$  is the reproducibility, as a percentage (relative).

**D.5** The precision of each of these test methods may be expressed in the form of the following statements which use an “appropriate value” of  $r$ ,  $R$ ,  $(r)$  or  $(R)$ , that is, that value to be used in decisions about test results obtained with the test method. The “appropriate value” is that value of  $r$ ,  $R$ ,  $(r)$  or  $(R)$ , associated with the mean level in Tables D.1, D.2 or D.3 closest to the mean level under consideration (at any given time, for any given material) in routine testing operations.

**Table D.1— Method A, standard reference compound No. 1 (see B.2 )**

Test rubber	Mean relative volume loss mm <sup>3</sup>	Within laboratory		Between laboratories	
		$r$	$(r)$	$R$	$(R)$
A (NR/BR)	68	6,5	9,6	18,8	27,6
B (NR/SBR)	106	10,8	10,2	21,4	20,2
C (NR)	160	23,2	14,5	30,4	19,0
D (IIR)	257	30,2	11,8	57,5	22,4
E (EPDM)	345	39,8	11,5	83,0	24,1

**Table D.2 — Method A, standard reference compound No. 2 (see B.3)**

Test rubber	Mean relative abrasion index %	Within laboratory		Between laboratories	
		$r$	( $r$ )	$R$	( $R$ )
A (NR/BR)	157	30,8	19,6	59,6	38,0
B (NR/SBR)	102	15,0	14,7	19,1	18,7
C (NR)	67	9,1	13,6	13,6	20,3
D (IIR)	43	7,9	18,4	10,2	23,7
E (EPDM)	32	3,8	11,9	7,2	22,5

**Table D.3 — Method B, standard reference compound No. 2 (see B.3)**

Test rubber	Mean relative abrasion index %	Within laboratory		Between laboratories	
		$r$	( $r$ )	$R$	( $R$ )
A (NR/BR)	144	20,2	14,0	45,8	31,8
B (NR/SBR)	101	9,6	9,5	15,9	15,7
C (NR)	71	7,0	9,9	11,0	15,5
D (IIR)	43	3,4	7,9	5,3	12,3
E (EPDM)	34	3,3	9,7	7,2	21,2

**D.6 Repeatability** — The repeatability limit,  $r$ , of these test methods has been established as the appropriate value tabulated in Table D.1, D.2 or D.3. Two single test results, obtained under normal test method procedures that differ by more than this tabulated  $r$  (for any given level) should be considered to have come from different or non-identical sample populations.

**D.7 Reproducibility** — The reproducibility limit,  $R$ , of these test methods has been established as the appropriate value tabulated in Table D.1, D.2 or D.3. Two single test results obtained in two different laboratories, under normal test method procedures that differ by more than the tabulated  $R$  (for any given value) should be considered to have come from different or non-identical sample populations.

**D.8 Repeatability and reproducibility**, expressed as a percentage of the mean level, ( $r$ ) and ( $R$ ), have equivalent application statements as above for  $r$  and  $R$ . For ( $r$ ) and ( $R$ ) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

**D.9** In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for these test methods since the values of abrasion loss are exclusively defined by the test method. Bias, therefore, cannot be determined.



## Bibliography

- [1] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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