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Cylindrical stoppers of natural cork — Physical tests — Reference methods

Bouchons cylindriques en liège naturel — Essais physiques — Méthodes de référence

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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.

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.

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Cylindrical stoppers of natural cork — Physical tests — Reference methods

1 Scope

This International Standard specifies the reference methods for determining the following characteristics of cylindrical stoppers of natural corkwood: dimensions; deviations from parallelism of bases and from perpendicularity of sides and bases; apparent density; moisture content; behaviour under compression; extraction and penetration strengths; absorption; and capillarity[1].

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2859-1:1989, *Sampling procedures for inspection by attributes — Part 1: Sampling plans indexed by acceptable quality level (AQL) for lot-by-lot inspection.*

ISO 3951:1989, *Sampling procedures and charts for inspection by variables for percent nonconforming.*

ISO 4707:1981, *Cork — Stoppers — Sampling for inspection of dimensional characteristics.*

1) Sealing behaviour will be covered later.

2) Orange II.

3 Sampling

Unless otherwise agreed between purchaser and vendor the size of the sample and the acceptable quality level shall be in accordance with ISO 2859-1, ISO 3951, and ISO 4707.

4 Reagents

4.1 Ethanolic solution, 10 % (V/V).

4.2 Coloured[2] ethanolic solution, 10 % (V/V).

5 Apparatus

5.1 Apparatus, (see figure 1), to measure dimensions and angles of inclination of bases, and check base parallelism of stoppers, consisting of

5.1.1 Rectangular base, in transparent acrylic plastic, dimensions 220 mm × 220 mm, fitted with four levelling-screws.

5.1.2 Plate, in stainless steel, dimensions 170 mm × 150 mm × 9 mm, fitted with two upright cylindrical stainless steel rods, 200 mm high and 10 mm diameter. These rods are perpendicular to the plate and 120 mm apart (measured from centre to centre). This plate fits completely into the base (5.1.1).

5.1.3 Two dial-gauges, with a measuring range of 50 mm and a resolution of 0,01 mm, with forked plunger ends and rotary dials for zero setting.

5.1.4 Two supports, with a circular opening in one end through which passes one of the cylindrical rods referred to in 5.1.2. The supports are fixed to the rods by screws. The other end of the supports is fixed to the inside of roller bearings set on the

covers of the dial-gauges (5.1.3); the supports and the dial-gauges can be screwed together.

5.1.5 Ruler, stainless steel, length 130 mm, with a reference point in its centre, fixed by the forked plunger ends referred to in 5.1.3. The contact of the ruler to the plate (5.1.2) corresponds to the zero of the dial-gauges.

5.1.6 Accessory, consisting of a stainless steel cylinder in which a cut is made defined by two half-surfaces vertical in relation to the bases, with the origin in the cylinder axis and with an angle of approximately 60°.

5.2 Balance, accurate to ± 0,1 mg.

5.3 Cutting-dies.

5.4 Oven, with thermostat control and air circulation controlled at 103 °C ± 2 °C.

5.5 Desiccator.

5.6 Dynamometric press, with fixed head and movable base, moving at speeds of 1 cm/min and 30 cm/min (see figure 2), equipped with

5.6.1 Sensor, jointed to the fixed head.

5.6.2 Measure and control unit.

5.6.3 Potentiometric recorder.

5.7 Bottles, with "plate unique" finish (diameters 18,5 mm ± 0,5 mm)[3].

5.8 Bottling machine, with four jaws of polished metal capable of pressing the stopper into a cylindrical form to a diameter 2 mm smaller than that of the bottle finish.

5.9 Apparatus for compression test, (see figure 3), consisting of

5.9.1 Device, in stainless steel, to support the stopper on the movable base of the press (5.6), for the compression test (see figure 3).

5.9.2 Rectangular indentor, 50 mm × 20 mm, connected to the sensor (5.6.1) by a stainless steel rod.

5.9.3 Dial-gauge, graduated in 0,1 mm, accurate to ± 0,01 mm, capable of moving parallel to the movable base of the press (5.6), which shall support the indentor.

5.10 Device, for the determination of extraction and penetration strength (see figure 4 and figure 5), consisting of

5.10.1 Fixing device, for fixing the bottle to the movable base of the press (see figure 4).

5.10.2 Corkscrew, stainless steel, with a penetrating helicoidal stick, thread between 12 mm and 14 mm, length greater than 60 mm, and with 5 or 6 helices with at least a 3 mm internal diameter and 10 mm to 15 mm external diameter.

The corkscrew shall have a triangular handle capable of being turned manually.

5.10.3 Connecting device, to the sensor and to the corkscrew handle, which ensures that the perpendicularity between the extraction device and the movable base is maintained.

5.11 Punch, capable of being adjusted to the sensor in order to insert the cork stopper into the bottle by sliding it through its neck (see figure 5), with 1 cm^2 of surface contact with the cork stopper.

5.12 Open container, to hold the coloured ethanolic solution (4.2).

5.13 Filter paper, Whatman No. 4[4].

5.14 Ruler, with an accuracy of 0,5 mm.

6 Normal test conditions

Unless otherwise specified tests shall be carried out on corks conditioned for 24 h at a temperature of 20 °C ± 2 °C and a humidity of 65 °C ± 5 %.

3) See CETIE Standards.

4) Whatman No. 4 is the trade-name of a product supplied commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7 Tests

7.1 Dimensions, deviations from parallelism of bases, and from perpendicularity of sides and bases

7.1.1 Setting up the apparatus (5.1)

Place the ruler (5.1.5) on the plate (5.1.2). Adjust the position of the two supports (5.1.4) for an approximate setting of zero in the comparators.

Screw the supports to the rods and adjust the zeroes in the dial-gauges by means of the turning dials.

To ensure that the rods are perpendicular adjust the ruler one way and then the other; the vertical position corresponds to the minimum value given by the comparators.

Fix only one of the dial-gauges to its support with the corresponding screw.

7.1.2 Deviation from perpendicularity of sides and bases

7.1.2.1 Procedure for testing

Tests to be carried out on stoppers tested in 7.1.3.

Place the apparatus (5.1) on a table with a horizontal top. Place each stopper inside the accessory (5.1.6) and fix it with adhesive tape so that the base of the cork stopper is higher than the base of the accessory. Place the ruler (5.1.5) on the cork along its diameter. Read out the result on both dial-gauges. Rotate the cork 90° and repeat the procedure.

7.1.2.2 Results

The deviation from perpendicularity in the first position is

$$\beta_1 = \arcsin\left(\frac{Z_1 - Z_2}{120}\right)$$

where Z_1 and Z_2 are the readings on both dial-gauges, resulting from measurement in the first position.

The deviation from perpendicularity in the second position (position at a right angle to the first) is

$$\beta_2 = \arcsin\left(\frac{Z_3 - Z_4}{120}\right)$$

where Z_3 and Z_4 are the readings on both dial-gauges, resulting from the measurement in the second position.

The deviation from perpendicularity in relation to the base, is

$$\gamma_1 = \frac{\beta_1 + \beta_2}{2}$$

The deviation from perpendicularity of sides and the other base is

$$\gamma_2 = \frac{\beta_3 + \beta_4}{2}$$

where β_3 and β_4 are the new deviations measured when the cork stopper is released from the apparatus and turned upside down.

The average deviation from perpendicularity between generating lines and bases is

$$\gamma = \frac{\gamma_1 + \gamma_2}{2}$$

The result of the test for each cork stopper in the sample shall be expressed in degrees, rounded off to the nearest 0,1°.

7.1.3 Length and deviation from parallelism of bases

7.1.3.1 Procedure

Place the apparatus (5.1) on a table with a horizontal top. Place each cork from the sample on the plate (5.1.2) under the ruler (5.1.5) in its central area, on one of its bases. Read out the result on both comparators. Rotate the cork stopper 90° and repeat the procedure.

7.1.3.2 Results

The average length of the cork stopper in the first position is

$$l_1 = \frac{\kappa_1 + \kappa_2}{2}$$

and the deviation from parallelism of the bases for this first position is

$$\alpha_1 = \arcsin\left(\frac{\kappa_1 - \kappa_2}{120}\right)$$

where κ_1 and κ_2 are the readings on both dial-gauges, resulting from the measurement of the first position.

The average length of the cork stopper in the second position is

$$l_2 = \frac{\kappa_3 + \kappa_4}{2}$$

and the deviation from parallelism of the bases for this second position is

$$\alpha_2 = \arcsin\left(\frac{\kappa_3 - \kappa_4}{120}\right)$$

The average length of each cork stopper is

$$l = \frac{l_1 + l_2}{2}$$

and the angular deviation from parallelism of the bases of the cork stopper is

$$\alpha = \frac{\alpha_1 + \alpha_2}{2}$$

The result of the test for each cork stopper in the sample shall be expressed in millimetres, rounded off to the nearest 0,1 mm for the length, and in degrees, rounded off to the nearest 0,1° for the deviation from parallelism of bases.

7.1.4 Diameter[5]

7.1.4.1 Procedure

Test the cork stoppers tested in 7.1.2.

Place the apparatus (5.1) on a table with a horizontal top. Put each cork stopper under the ruler (5.1.5), in its central area, on one of its sides. Read both dial-gauges. Rotate the cork stopper 90° and repeat the procedure.

7.1.4.2 Results

The average diameter of the cork stopper in the first position is

$$d_1 = \frac{Y_1 + Y_2}{2}$$

where Y_1 and Y_2 are the readings on both dial-gauges, resulting from the measurement of the diameter of the cork stopper in the first position.

The average diameter of the cork stopper in the second position

$$d_2 = \frac{Y_3 + Y_4}{2}$$

where Y_3 and Y_4 are the readings on both dial-gauges, resulting from the measurement of the diameter of the cork stopper in the second position.

The average diameter of each cork stopper is

$$d = \frac{d_1 + d_2}{2}$$

The result of the test for each cork stopper of the sample shall be expressed in millimetres, and rounded off to the nearest 0,1 mm.

7.2 Apparent density

7.2.1 Procedure

Determine, using the balance (5.2), the mass of each cork in the sample; their dimensions being determined in accordance with 7.1.3 and 7.1.4.

7.2.2 Results

The apparent density of each cork stopper, expressed in kilograms per cubic metre is

$$\frac{4}{\pi} \times \frac{m}{d^2 \times l} \times 10^6$$

where

m is the mass of each cork stopper, in grams, rounded off to the nearest 1 mg;

d is the diameter of each cork stopper, in millimetres, rounded off to the nearest 0,1 mm;

l is the length of each cork stopper, in millimetres, rounded off to the nearest 0,1 mm.

The apparent density for each cork stopper in the sample shall be expressed in kilograms per cubic metre, rounded off to the nearest integer.

7.3 Moisture content[6]

7.3.1 Procedure

Use the dies (5.3) to cut each cork stopper from the sample weighed in 7.2, into four pieces, through their diameters. Place the pieces in the oven (5.4) set at 103 °C ± 2°C for 3 h.

Remove the cork stopper pieces from the oven and place them in the desiccator (5.5) for 30 min. Weigh them once more.

7.3.2 Results

The moisture content of each cork stopper, expressed as a percentage is

$$\frac{m_0 - m_1}{m_0}$$

where

m_0 is the initial mass of each cork stopper, determined in 7.2, in grams, rounded off to the nearest 1 mg;

5) This method is considered as a reference method. It may either be used as a gauge, or the apparatus (5.1) can be used as the calibrator.

6) This test does not require prior conditioning.

m_1 is the mass of each dried cork stopper, in grams, rounded off to the nearest 1 mg.

The moisture content of each cork stopper in the sample shall be expressed as a percentage, rounded off to the nearest 1 %.

7.4 Behaviour under compression over a period of time

7.4.1 Procedure

Place each cork stopper on the device (5.9.1) which is placed on the movable base of the press (5.6) so that an angle of 45° is made between the veins and the indentor base (5.9.2).

Adjust the indentor to the sensor (5.6.1) and move the dial-gauge (5.9.3) in such a way that the indentor will be applied on the cork stopper and set the dial-gauge to zero.

Start the movable base at a speed of 1 cm/min. When the dial-gauge reads 33 % of the diameter value of the cork stopper, read on the measure and control unit (5.6.2) the corresponding compression strength (F_1). After 1 h[7] again read the compression strength (F_2).

The recorder (5.6.3) registers the curve corresponding to the function F(t).

7.4.2 Results

The result of the test is given by the forces F_1, F_2, and the difference

$$F_1 - F_2$$

where

F_1 is the compression force corresponding to a reduction of 33 % of the cork stopper diameter, in decanewtons, and rounded off to the nearest 0,1 daN;

F_2 is the compression force, 1 h after reading F_1, in decanewtons, and rounded off to the nearest 0,1 daN.

7.5 Behaviour under compression after bottling

7.5.1 Procedure

Place a cork stopper on the device (5.9.1) which is placed on the movable base of the press (5.6) so that an angle of 45° is made between the veins and the indentor base (5.9.2).

Adjust the indentor in the sensor (5.6.1) and move the dial-gauge (5.9.3) so that the indentor will be applied on the movable base of the press, setting the dial-gauge to zero.

Start the movable base at a speed of 1 cm/min. When the dial-gauge reads a value equal to the diameter (d_m) of the bottling machine jaws, record the corresponding compression strength (F_e). Quickly relieve the press in order to obtain cork expansion, and read on the dial-gauge a value equal to the average internal diameter of the bottle neck (d_g)[8].

After 2 min, read the reaction strength F_r, and record during this time the curve corresponding to the cork stopper relaxation during this period.

7.5.2 Results

The result for each cork stopper is given by the values of F_c and F_r, obtained on the measure unit (5.6.2), in decanewtons, rounded off to the nearest 0,1 daN.

F_c is the compression strength needed to obtain the deformation of the cork stopper of

$$\frac{d_r \times d_m}{0{,}75}$$

F_r is the reaction strength shown by the cork stopper when submitted to the deformation of

$$\frac{d_g - d_m}{0{,}75}$$

where

d_m is the diameter of the bottling machine jaws, in millimetres, rounded off to the nearest 0,1 mm;

d_r is the average diameter of the cork stoppers, in millimetres, rounded off to the nearest 0,1 mm;

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7) If further information is required it will be necessary to wait 24 h, 2 d or 7 d, these periods of time should be noted in the test report.

8) This diameter may be determined with the aid of a suitable mechanical device or by the use of a model in paraffin obtained from the bottle neck. It may also be determined by using the value equal to the internal diameter of the bottle neck at a level corresponding to the length of the stopper.

d_g is the average diameter of the bottle neck, in millimetres, rounded off to the nearest 0,1 mm;

0,75 is a testing coefficient.

7.6 Extraction strength

7.6.1 Procedure

Fill a bottle (5.7)[9] with the ethanolic solution (4.1) and close it with a cork stopper from the sample, using the bottling machine (5.8).

After 5 min place the bottle in a horizontal position for 8 d[10]. Then, introduce the corkscrew (5.10.2) into the cork stopper until its helicoidal stick can be seen 3 mm outside the cork stopper.

Fix the bottle with the corkscrew to the movable base of the press (5.6), using the fixing device (5.10.1).

With the connecting device (5.10.3), connect the triangular handle of the corkscrew to the sensor of the press.

Start the press, with its movable base, at a speed of 30 cm/min.

Read the extraction strength on the measure and control unit (5.6.2). The function, $F(t)$, is shown on the recording device (5.6.3).

7.6.2 Results

The result for each cork stopper in the sample shall be expressed in decanewtons, rounded off to the nearest 0,1 daN.

7.7 Penetration strength

7.7.1 Procedure

Fill a bottle (5.7)[9] with ethanolic solution (4.1) and close it with a cork stopper from the sample, using the bottling machine (5.8).

After 5 min place the bottle in a horizontal position for 8 d[10]. Fix the corked bottle to the movable base of the press, using the fixing device (5.10.1).

Connect the punch (5.11) to the sensor (5.6.1).

Start the press (5.6), with its movable base, at a speed of 30 cm/min.

Read, on the measure and control unit (5.6.2), the force necessary for slipping the cork stopper through the bottle's neck into the bottle. Record the result on the recorder (5.6.3).

7.7.2 Results

The result of the test is the value of the strength needed to introduce each cork stopper into the bottle, by slipping it through the bottle's neck.

The penetration strength for each cork stopper in the sample shall be expressed in decanewtons, rounded off to the nearest 0,1 daN.

7.8 Absorption[11]

7.8.1 Procedure

Number and weigh each cork stopper from the sample. Fill the bottles (5.7) with the coloured ethanolic solution (4.2)[12] and close them with the cork stoppers from the sample, using the bottling machine (5.8).

Place the bottles in a horizontal position for 8 d.

Extract the cork[13] stopper from the bottles, and put them on Whatman No. 4 filter paper for 1 min.

Weigh the cork stoppers again, one by one.

7.8.2 Results

The absorption for each cork stopper, expressed in milligrams is

$$m_0 - m_1$$

where

m_0 is the initial mass of the cork stopper, in milligrams, rounded off to the nearest 0,1 mg;

m_1 is the mass of the cork stopper after the test, in milligrams, rounded off to the nearest 0,1 mg.

The absorption for each cork stopper in the sample shall be expressed in milligrams, and rounded off to the nearest 0,1 mg.

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9) An empty space of 15 mm shall be left between the level of the solution and the bottom of the cork stopper.

10) This test can also be carried out after 24 h and a reference shall be included in the test report.

11) Stoppers used in test (7.6) can also be used in the absorption test as long as precautions are taken when unscrewing the cork to be weighed.

12) Wine can also be used.

13) See extraction test (7.6).

7.9 Capillarity

7.9.1 Procedure

Number the cork stoppers from the sample and place them on their bases in the container (5.12) with the coloured ethanolic solution (4.2) which shall reach a height of 3 mm on the cork stopper. Wait 24 h. Remove the cork stoppers and put them on filter paper (5.13) for 1 min.

With the ruler (5.14), measure the maximum height reached by the coloured ethanolic solution on the lateral surface of each cork stopper.

7.9.2 Results

The capillarity of each cork stopper in the sample shall be expressed in millimetres, rounded off to the nearest 0,5 mm.

8 Test report

The test report shall include the following:

a) all the information necessary for the complete identification of the sample;

b) reference to the method used;

c) the results obtained, including average values and the deviations;

d) all operating details either not specified in this International Standard or regarded as optional;

e) any occurrences that may have affected the results.

f) number of test pieces used for each test;

g) reference to this International Standard.