

Ceramic and glass insulating materials —

Part 2: Methods of test

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Foreword

The text of document 15C/1049/FDIS, future edition 2 of IEC 60672-2, prepared by SC 15C, Specifications, of IEC TC 15, Insulating materials, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 60672-2 on 2000-01-01.

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The following dates were fixed:

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- latest date by which the national standards conflicting
with the EN have to be withdrawn (dow) 2003-01-01

Annexes designated "normative" are part of the body of the standard.
In this standard, annexes A and ZA are normative.
Annex ZA has been added by CENELEC.

Endorsement notice

The text of the International Standard IEC 60672-2:1999 was approved by CENELEC as a European Standard without any modification.

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CERAMIC AND GLASS INSULATING MATERIALS –

Part 2: Methods of test

1 Scope

This part of IEC 60672 is applicable to ceramic, glass and glass-ceramic materials to be used for electrical insulation purposes. This standard specifies methods of test. It is intended to provide test results typical of the material from which the test pieces are processed. Since, in the majority of cases, ceramic components for insulating purposes are of rather different size and shape to test pieces, the results of such tests provide only a guide to the actual properties of components. The limitations imposed by the method of forming and processing are discussed where relevant.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of IEC 60672. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of IEC 60672 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

IEC 60093:1980, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials*

IEC 60212:1971, *Standard conditions for use prior to and during the testing of solid electrical insulating materials*

IEC 60243-1:1998, *Electric strength of insulating materials – Test methods – Part 1: Tests at power frequencies*

IEC 60250:1969, *Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies, including metre wavelengths*

IEC 60345:1971, *Method of test for electrical resistance and resistivity of insulating materials at elevated temperatures*

IEC 60672-1:1995, *Ceramic and glass insulating materials – Part 1: Definitions and classification*

IEC 60672-3:1997, *Ceramic and glass insulating materials – Part 3: Specifications for individual materials*

IEC 61006:1991, *Methods of test for the determination of the glass transition temperature of electrical insulating materials*

ISO/DIS 463, *Geometrical product specifications (GPS) – Dimensional measuring instruments – Dial gauges – Design and metrological requirements* (Revision of ISO/R 463:1965) ¹⁾

ISO 758:1976, *Liquid chemical products for industrial use – Determination of density at 20 °C*

ISO 3534-1:1993, *Statistics – Vocabulary and symbols – Part 1: Probability and general statistical terms*

ISO 3611:1978, *Micrometer callipers for external measurement*

ISO 6906:1984, *Vernier callipers reading to 0,02 mm*

ISO 7884-8:1987, *Glass – Viscosity and viscometric fixed points – Part 8: Determination of (dilatometric) transformation temperature*

3 General notes on tests

3.1 Test pieces

Test pieces shall be processed under conditions closely similar to those normally employed for the manufacture of components, and in sufficient numbers to be representative of those conditions. It is emphasized that results from the test pieces are affected by the method of forming and, in many cases, by the method of surface finishing; methods used should, as far as possible, be those used in the production of items. For each test result reported, the method of manufacture of the test piece shall be specified. All numerical values determined according to these test methods apply only to the test pieces prescribed. They cannot be extended to test pieces and ceramic products of other shapes and dimensions nor of other types of manufacture. The minimum number of test pieces for each test is given in table 1.

NOTE For thermally toughened glass test pieces, the thermally pre-stressed state of glass depends on the following factors:

- thermal expansion below and above the transition range (see IEC 61006);
- viscosity/temperature relation;
- thermal diffusivity, i.e. thermal conductivity (specific heat capacity × bulk density);
- elastic properties;
- starting temperature of cooling;
- heat transfer coefficient;
- thickness and form of glass product.

As a result of the last factor, test pieces from the same glass but of different shape and thickness have different tempering levels, although they are tempered under the same conditions. Consequently, it is impossible to have a special test piece which represents the properties of toughened glass items of other shapes and thickness. Therefore, physical properties of thermally tempered glass items which show corresponding dependence on the tempering state can be determined only on the item itself, and it is recommended that this procedure is adopted whenever possible. This applies to properties such as flexural strength, resistance to thermal shock, volume resistivity and dissipation factor.

¹⁾ To be published.

Table 1 – Characteristics and minimum number of test pieces for each test

Clause	Test	Number of test pieces	Shape and dimensions
4	Dye penetration test	3	Fragments or small components; see 4.2
5	Bulk density/ open porosity	3	Fragments or small components; see 5.2, note
6	Flexural strength	10	See table 3
7	Modulus of elasticity	3	Bar-shaped, preferably with a support span-to-thickness ratio > 50; see 7.3
8	Mean coefficient of linear thermal expansion	2	Appropriate to the apparatus used
9	Specific heat capacity	2	Appropriate to the apparatus used
10	Thermal conductivity	2	Appropriate to the apparatus used; see 10.2
11	Thermal shock	30	Rods 10 mm diameter × 120 mm long
12	Glass transition temperature (T_g , for glass only)	2	Appropriate to the apparatus used
13	Electric strength	10	Discs as described in 13.3
14	Withstand voltage	10	Discs as described in 13.3
15	Relative permittivity/ dissipation factor	3	Discs in accordance with IEC 60250, as described in clause 15
16	Volume resistivity	2	Discs in accordance with IEC 60345, as described in clause 16

3.2 Presentation of results

The test report shall include the following:

- name of testing establishment;
- a reference to this standard;
- date of test;
- identification of the item, test piece or test material (type, manufacturer, shaping process, batch number, date of manufacture, etc., as appropriate);
- the test performed;
- the preparation, shape and dimensions of the test pieces and the number tested (see table 1 for the minimum number for each test);
- details relevant to the test or tests undertaken (see requirements listed under each test method);
- individual results from each test piece;
- the arithmetic mean value of the individual results, and the standard deviation.

3.3 Evaluation against a minimum specification

For the purpose of assessing whether a material has satisfactory properties compared with the minimum specification laid down in IEC 60672-3, the mean value of the specified number of determinations shall be compared with the maximum or minimum value required in IEC 60672-3, or with the range of values permitted.

4 Dye penetration test (liquid absorption)

NOTE 1 This test is intended to detect the presence of continuous interconnected porosity or microcracks which would render the material unsatisfactory from the high-voltage dielectric breakdown point of view. The test is not applicable to glass materials with the exception of sintered glass products. The test is also not intended as a routine inspection test for minor cracks or pores in small components, for which alternative less stringent tests, for example a liquid dye vacuum test, may be appropriate.

NOTE 2 For dark-coloured materials, the dye chosen should be such as to give contrast with the natural colour of the material. Fluorescent dyes may be appropriate, but only if used as described in 4.4.

4.1 Test apparatus

The test apparatus shall include a pressure vessel capable of withstanding a pressure of at least 30 MPa, a high-pressure pump and a pressure gauge. Test pieces are immersed in dye solution which either directly fills the pressure vessel, or is contained in a metal container inside the pressure vessel to which pressure can be transmitted by the pressurizing hydraulic oil through a rubber bung or piston (see figure 1). An oven capable of maintaining $120\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ is required for test piece conditioning.

4.2 Test pieces

Fragments of ceramic shall be used. No more than 25 % of the total surface area may be glazed or have an "as-fired" skin. The test shall be made on fragments from at least three separate components or test pieces.

4.3 Dye

The dye solution shall be prepared containing typically 1 g to 3 g of dye in 1 l of ethyl alcohol or methylated spirits, or other suitable solvent.

NOTE 1 Suitable non-toxic dyes include xanthan/triaryl methane mixtures.

NOTE 2 Due regard should be paid to health hazards and environmental implications of using and disposing of organic dyes and solvents.

4.4 Method of test

The test pieces shall be free from oil or dirt of any type, and shall be washed if necessary. The test pieces shall be dried at $120\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for a period of not less than 3 h prior to the test, and are then broken into fragments of appropriate size.

The test pieces shall be immersed in the dye solution, which is either directly in the pressure vessel, or in the metal container sealed with the rubber seal or piston which is then placed in the pressurising vessel. The system shall be pressurized to not less than 15 MPa, and for a time period such that the product of pressure in megapascals (MPa) and time in hours (h) is not less than 180. After the appropriate time, the fragments shall be taken from the system, washed in water, dried and broken. The freshly broken surfaces shall be examined, using normal vision for any sign of penetration of the dyestuff. These surfaces shall show no penetration. Penetration of dye into small cracks, produced when initially preparing fragments, shall not be taken into consideration.

4.5 Test report

In addition to the information required under items a) to i) in 3.2, the test report shall contain the following:

- a) the pressure, in MPa, and the time under pressure, in h;
- b) the size, shape and number of test pieces and the fragments produced therefrom;
- c) whether or not penetration of dye was observed on any freshly fractured surface of fragment.

5 Bulk density and open (apparent) porosity

NOTE 1 These methods are not appropriate for the determination of open porosity levels of less than 0,5 % by volume, since reliable numerical results cannot be obtained. The existence of such porosity levels can be more reliably determined using the dye test described in clause 4.

NOTE 2 These tests are appropriate for the determination of the bulk characteristics of materials. It should be noted that as-fired skins on ceramic components may be impervious, even if bulk material may have some open porosity.

NOTE 3 The use of liquid immersion media other than distilled water is permissible provided that the density of the liquid is measured to $\pm 0,001 \text{ Mg.m}^{-3}$ at the temperature of the weighings of the immersed test piece. Suitable liquids include paraffin, butyl alcohol and other organic liquids of low volatility.

NOTE 4 By agreement, other methods, for example gas pycnometry, may be used, but the results may not be comparable to those produced by the methods described below.

5.1 Test apparatus

The following apparatus is required for this test:

- a) a hydrostatic balance (a balance suitable for determining the apparent mass of a test piece suspended in a liquid) capable of weighing to an accuracy of $\pm 0,01 \text{ g}$;
 - b) a thin, de-greased metallic suspension wire of diameter not exceeding 0,20 mm;
 - c) either:
 - method A: a gas-tight vessel (bell jar or desiccator) connected to a suitable vacuum pump, and which is provided with suitable means for measuring the pressure and for admitting liquid, or
 - method B: a vessel for containing boiling water and equipped with non-corrodable wide-mesh netting to support the test piece(s) positioned at least 10 mm above the base of the vessel;
- NOTE Method B is not recommended if inflammable organic liquids are to be used owing to potential safety hazards.
- d) an oven for drying test pieces;
 - e) a lint-free cloth for removing excess liquid from test pieces;
 - f) a supply of demineralized or freshly distilled water, or other suitable liquid (see note 3 above).

5.2 Test pieces

At least three determinations shall be made. If an open porosity determination is required, each test shall employ at least three fragments of ceramic material with a total mass of between 50 g and 80 g. No more than 25 % of the surface may be either glazed or have an "as-fired" skin. The fragments shall be clean and degreased. Any chips liable to become detached during the test and any loose dust shall be removed by brushing before the test.

NOTE If density determinations are required on individual, small components weighing less than 50 g each, the method cited here may be used provided that the accuracy of weighing is $\pm 0,001$ g for test pieces of greater than 3 g mass, or $\pm 0,0001$ g for test pieces of 1 g to 3 g mass. For components of mass less than 1 g the methods described below are not adequate, and several components should be tested together to make a larger mass. Individual components should not have a mass of less than 0,1 g.

5.3 Method of test

Dry the test piece fragments in the oven at $120\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for at least 2 h and until constant mass m_0 is achieved.

Method A: Place the test piece fragments in a suitable container inside the vacuum vessel. Close the vessel and evacuate it to a pressure of less than 3 kPa and maintain this for at least 5 min. Isolate the vessel from the vacuum pump and observe any steady rise in pressure. If a rise to greater than 4 kPa occurs in less than 5 min, the test piece is continuing to out-gas. Reconnect the vacuum pump and continue evacuating until there is no significant change in a 5 min period on isolation of the vacuum vessel. Admit freshly boiled distilled water at $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ to the test piece in the container until the test piece is covered, and continue evacuation for a further period of at least 5 min. After this period, isolate the vacuum pump and admit air to the vessel, and remove the test piece in its container of water. For low-porosity materials, allow to stand in distilled water for at least 6 h.

Method B: Place the test piece fragments in the vessel and cover with distilled water. Heat to boiling point and boil for at least 30 min; then allow to cool to room temperature while still immersed, and stand in distilled water for at least 6 h.

NOTE Method A and method B may not give exactly equivalent results for open-porosity determination, method B being considered to be less effective in removing entrained air from the test pieces.

Whichever method is used, weigh the test piece fragments in distilled water, using a thin, clean metal suspension wire (m_w). Remove the test pieces and replace in the distilled water. Weigh the suspension wire submerged to the same depth as when used for weighing the immersed test pieces (m_s). Record the temperature of the water (T) to the nearest 0,1 $^{\circ}\text{C}$. Remove the test piece fragments from the water, wipe each with the damp lint-free cloth to remove surface water only, and reweigh in air (m_h).

5.4 Calculation of results

Calculate the bulk density (ρ_a) and open (apparent) porosity (p_a) as follows:

$$\rho_a = \frac{m_o \times \rho_w}{m_h - m_w + m_s} \quad p_a = \frac{m_h - m_o}{m_h - m_w + m_s} \times 100 \quad (1)$$

where

- ρ_a is the bulk density, in $\text{Mg} \cdot \text{m}^{-3}$;
- p_a is the open (apparent) porosity, in vol %;
- m_o is the mass of the dry test piece, in g;
- m_w is the apparent mass of the test piece and suspension wire immersed in water, in g;
- m_s is the apparent mass of the suspension wire immersed to same level in water, in g;
- m_h is the mass of the soaked test piece in air, in g;
- ρ_w is the density of water or other immersion liquid at the test temperature T , in $\text{Mg} \cdot \text{m}^{-3}$.

The density of distilled water at temperature T is given in table 2. If a liquid other than distilled water is used, its density shall be determined following the procedure laid down in ISO 758 at the same temperature as used for the test piece density determination.

Table 2 – Density of distilled water

Temperature °C	Density ρ_w , $\text{Mg} \cdot \text{m}^{-3}$
18	0,99860
19	0,99841
20	0,99820
21	0,99799
22	0,99777
23	0,99754
24	0,99730
25	0,99704
26	0,99678

5.5 Test report

In addition to the information required under items a) to i) in 3.2, the test report shall include the following:

- a) the method used (method A or method B);
- b) the immersion liquid used, if not distilled water;
- c) the temperature at which the density determination was made, in °C;
- d) the density of the immersion liquid at the test temperature, in $\text{Mg} \cdot \text{m}^{-3}$;
- e) the time taken to complete the soaking of the test piece, in h;
- f) the individual values of bulk density, in $\text{Mg} \cdot \text{m}^{-3}$ (rounded to the nearest 0,01 $\text{Mg} \cdot \text{m}^{-3}$), and open porosity, in % (rounded to the nearest 0,1 %), determined on each test piece.

6 Flexural strength

NOTE The fracture strength determined from a flexural test depends upon many factors including test piece size and cross-sectional geometry, surface finish, stressing rate, humidity of the environment, test jig geometry and mode of operation. The result of such a test gives a guide to the general strength properties of the material, but cannot be used directly as a figure for design purposes. It can be used as a quality-control check, and for a broad intercomparison of different materials, provided that relevant test parameters, such as those given above, are kept constant.

6.1 Test apparatus

The apparatus required for this test includes the following:

- a) mechanical testing machine capable of applying a force to a test piece at a constant loading rate or cross-head movement rate, and of recording peak force applied with an accuracy of $\pm 1\%$;

NOTE ISO 7500-1 [2]* provides information on the requirements for linearity and resolution of force recording in mechanical testing machines when used under tensile force, procedures which may be applied also to the compression mode, such as typically encountered in flexural strength testing.

- b) a specimen loading jig of geometry and dimensions appropriate to the test being performed (see 6.2 and table 3);
- c) a micrometer in accordance with ISO 3611 capable of measuring dimensions of test pieces to an accuracy of $\pm 0,01$ mm;
- d) a travelling microscope, vernier callipers in accordance with ISO 6906, or other suitable device for measuring spacing of centres of test piece support and loading rods to an accuracy of $\pm 0,05$ mm.

6.2 Loading jigs

Method A – three-point bend

The test jig or fixture consists of two parallel test piece support rods upon which the test piece is laid, and one loading rod to apply a force to the test piece, midway between the support rods. The support rods shall be free to rotate about an axis parallel to their lengths in order to minimize friction effects, and one of the support rods shall be free to rotate about an orthogonal axis parallel to the length of the test piece in order to permit alignment. The loading rod shall have a similar axis of rotation in order to achieve uniform loading across the test piece. The diameter of the support and loading rods and the distance between the centres of the support rods (the span) shall be as defined in table 3, and the surface shall be maintained in a polished, burr-free condition. The force is applied orthogonally to the plane of the test piece and/or support rods. A typical jig arrangement is shown in figure 2.

Method B – four-point bend

The test jig comprises two supports as in method A, with identical specification. Two loading rods spaced with their centres half the support span distance apart (see 6.4.1 c)) are placed symmetrically to within $\pm 0,2$ mm between the support rods, and force is applied orthogonally to the test piece. The two loading rods shall be free to rotate about an axis parallel to their lengths, and shall also be capable of separately rotating about an axis parallel to the length of the test piece. In this way, full alignment of loading and support rods may be achieved.

* Figures in square brackets refer to the bibliography.

NOTE The rotation of support or loading rods about an axis parallel to the test piece length is not required for testing round or flattened, circular test pieces. For test piece surfaces machined flat and parallel-faced, this rotation is not essential for the support rollers, nor for the loading rollers of method B, but there should still be relative rotation between the loading roller(s) and the support rollers.

6.3 Test pieces

Test pieces shall be prepared in the sizes given in table 3 and figure 3 according to their classification (see IEC 60672-1). The method of preparation of ceramic material should be appropriate to the expected manufacturing method used for components, for example isostatically pressed or extruded. Any surface preparation technique such as grinding, abrasion, milling, etc., should be noted.

NOTE 1 The test piece and jig dimensions given in table 3 may, by agreement between customer and supplier, be modified to suit particular circumstances. The support span to test piece-depth ratio should not be less than 10:1 for use of the equations in 6.5. Full details of the test procedure should be given in any test report. This standard is permitted to be cited only if all other aspects of procedure conform to the requirements for this test.

For glass and glass-ceramic materials, an abraded surface shall be used, prepared using falling sand, the quantity, flow rate and fall height of which are to be specified in the test report.

NOTE 2 The fracture strength of many ceramics, especially fine-grained ones, and of glasses and glass-ceramics is strongly influenced by the surface condition since the surface is subjected to the highest stress in a flexure test. The surface treatment used for test pieces should be comparable with that used for manufactured components.

The minimum number of test pieces for each test shall be 10.

NOTE 3 If required, a larger number of test pieces may be tested in order to obtain statistical information, for example a Weibull modulus. If such is the case, it is recommended that a minimum of 30 test pieces is tested.

6.4 Method of test

The support rod span, and, in the case of method B, the loading roller span as well, shall be measured to the nearest 0,1 mm. The test piece cross-sectional dimensions shall be measured with a micrometer to the nearest 0,02 mm. In the case of nominally round cross-section test pieces, measure the maximum and minimum diameters at three positions and calculate the arithmetic mean. For flattened circular and rectangular section bars, measure the widths and thicknesses at a minimum of three positions near the centre of the test piece, and calculate the arithmetic mean. Place the test piece centrally across the support rollers and the loading roller (method A) centrally or the pair of loading rollers (method B) symmetrically to within $\pm 0,2$ mm. If not already done, place the test jig in the test machine. Apply an increasing force to the loading roller (method A) or equally to the two rollers (method B) at a rate between $20 \text{ N}\cdot\text{s}^{-1}$ and $50 \text{ N}\cdot\text{s}^{-1}$, either directly selected, or indirectly by choice of an appropriate machine cross-head displacement rate, and continue until the test piece fractures. Record the peak force to an accuracy of 1 %. Repeat the test on each test piece of the batch.