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Ceramic and glass insulating materials –

Part 2:
Methods of test

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

CERAMIC AND GLASS INSULATING MATERIALS –

Part 2: Methods of test

FOREWORD

1) The IEC (International Electrotechnical Commission) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of the IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, the IEC publishes International Standards. Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. The IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
2) The formal decisions or agreements of the IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested National Committees.
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International Standard IEC 60672-2 has been prepared by subcommittee 15C: Specifications, of IEC technical committee 15: Insulating materials.

This second edition cancels and replaces the first edition published in 1980 and constitutes a technical revision. In redrafting this standard, the intention has been to improve the instructions in the test methods so that the document becomes more usable in the testing laboratory. Some of the ambiguities of test conditions have been removed, particularly for mechanical testing for which the recent development of improved understanding of significant factors in testing has allowed a better definition of requirements and a restriction of the range of previously optional test piece sizes.

The text of this standard is based on the following documents:

FDIS	Report on voting
15C/1049/FDIS	15C/1069/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 3.

IEC 60672 consists of the following parts under the general title Ceramic and glass insulating materials:

Part 1:1995, Definitions and classification;

Part 2:1999, Methods of test;

Part 3:1997, Specifications for individual materials.

Annex A forms an integral part of this standard.

The committee has decided that this publication remains valid until 2008. At this date, in accordance with the committee's decision, the publication will be

- reconfirmed;
- withdrawn;
- replaced by a revised edition, or
- amended.

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:

a) name of testing establishment;
b) a reference to this standard;
c) date of test;
d) identification of the item, test piece or test material (type, manufacturer, shaping process, batch number, date of manufacture, etc., as appropriate);
e) the test performed;
f) the preparation, shape and dimensions of the test pieces and the number tested (see table 1 for the minimum number for each test);
g) details relevant to the test or tests undertaken (see requirements listed under each test method);
h) individual results from each test piece;
i) 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 °C ± 5 °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 °C ± 5 °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 ±0,001 $Mg.m^{-3}$ 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 pyknometry, 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 ±0,01 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).