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Specification for insulating materials based on mica - Part 2: Methods of test (IEC 60371-2:1987 + IEC 60371-2 :1987/A1:1994)

Specification for insulating materials based on mica -- Part 2: Methods of test

Bestimmung für Isolierstoffe auf der Basis von Glimmer -- Teil 2: Prüfverfahren

Spécification pour les matériaux isolants à base de mica -- Partie 2: Méthodes d'essais

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Specification for insulating materials based on mica
Part 2: Methods of test

(IEC 371-2:1987 + A1:1994)

Spécification pour les matériaux isolants à base de mica
Partie 2: Méthodes d'essais
(CEI 371-2:1987 + A1:1994)

Bestimmung für Isolierstoffe auf der Basis von Glimmer
Teil 2: Prüfverfahren
(IEC 371-2:1987 + A1:1994)

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CENELEC

European Committee for Electrotechnical Standardization
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Foreword

The text of the International Standard IEC 371-2:1987 and its amendment 1:1994, prepared by SC 15C, Specifications, of IEC TC 15, Insulating materials, was submitted to the formal vote and was approved by CENELEC as EN 60371-2 on 1996-10-01 without any modification.

This European Standard supersedes HD 352.2 S1:1978.

The following dates were fixed:

— latest date by which the EN has to be implemented at national level by publication of an identical national standard or by endorsement (dop) 1997-06-01

— latest date by which the national standards conflicting with the EN have to be withdrawn (dow) 1997-06-01

Annexes designated "normative" are part of the body of the standard.

In this standard, Annex ZA is normative.

Annex ZA has been added by CENELEC.

Endorsement notice

The text of the International Standard IEC 371-2:1987 and its amendment 1:1994, was approved by CENELEC as a European Standard without any modification.

Contents

Introduction

This standard is one of a series which deals with insulating materials for use in electrical equipment built up from mica splittings or mica paper, with or without reinforcement, and with mica paper in its pure state.

The series consists of three parts:

1. Definitions and General Requirements.
2. Methods of Test.
3. Specifications for Individual Materials.

1 Scope

This Part 2 defines the methods of test which are applicable to built-up mica materials, products based on them and mica paper.

General notes on tests

Tests are carried out at ambient temperature (15 °C to 35 °C), unless a test temperature is specified either in the method or in the specification for individual materials.

2 Preparation of test specimens for curable materials

Test specimens are prepared in accordance with the following methods which are applicable only to curable materials:

Method 1

Clean off all loose particles and projecting fibres from sufficient material to provide the test pieces required for the particular test.

Cut and stack the pieces required to form the test laminate. For tape material, build up the laminate to the required thickness using half-lapped layers with successive layers at right angles, where necessary cutting the sides to obtain the required dimensions.

Adjust the press temperature to 160 ± 5 °C, unless otherwise specified.

Place the test laminate in the centre of two caul plates not exceeding 1.5 mm thick and at 15 °C to 35 °C.

Insert stops of a size to provide the required test laminate thickness.

Insert the assembly of plates and test piece in the centre of the preheated press.

Close the press immediately and apply sufficient pressure to reach stops. Cure the test piece for a minimum of 30 min.

Remove the test piece and post-cure for the length of time at the temperature given in Part 3 or according to the recommendation of the supplier.

Unless otherwise specified, condition the test piece for 24 h in a controlled atmosphere of 50 ± 5 % r.h. and 23 ± 2 °C before the test.

Method 2

Clean off all loose particles and projecting fibres from sufficient material to provide the test sheets required for the particular test.

For full-width and sheet material, cut and stack the sheets required to form the test laminate.

There are two suggested ways of producing a laminate from tape material:

a) Cut the tape in pieces to the length of the laminate. Stack the pieces parallel and half-overlapped. The second and the following layers shall be moved sideways, so that the overlapping edges do not lie one upon another. In order to fix the pieces, the use of a hot iron is recommended.

b) Take a metal sheet of the size of the required laminate and of a thickness of 2 mm to 3 mm. Wind the tape half-overlapped and always in the same direction around this sheet until the required thickness is reached. It is recommended to start each layer separately and to move the second and the following layers sideways, so that the overlapping edges do not lie one upon another. It is necessary to put an interleaving release material between the metal sheet and the tape. Two laminates with the same thickness are formed.

The prepared stacks, having a composition as shown in Figure 1, page 42, will be put in the press.

The following press procedure, illustrated by Figure 2, page 42, is an example. Other press procedures shall be subject to contract.

— Close the cold press and bring it to a pressure of 0.15 N/mm^2.

— Heat the press to 70 °C under pressure of 0.15 N/mm^2.

— Reduce the pressure down to zero, open the press for a short time (ventilate).

— Heat the press to 90 °C under pressure of 0.15 N/mm^2.

— Reduce the pressure down to zero, open the press for a short time (ventilate).

— Heat the press to 110 °C under pressure of 0.15 N/mm^2.

— Reduce the pressure down to zero, open the press for a short time (ventilate).

— Heat the press to 160 ± 5 °C under pressure of 0.15 N/mm^2 until the resin starts to gel. This point of time is visually controlled by a test rod. As soon as the resin starts to gel, bring the pressure up to 3 N/mm^2.

— Cure under 3 N/mm^2 and 160 °C for 60 min or at a temperature otherwise specified

— Specimen to be cooled under pressure.

After this press procedure, post-cure the laminate for the time and the temperature specified in Part 3 or according to the recommendation of the supplier.

3 Thickness

3.1 Test apparatus

Depending on the materials to be tested, the apparatus for measuring thickness is as follows:

3.1.1 A constant pressure measurement device having flat measuring faces of 6 mm to 8 mm diameter, the graduations being in divisions of 0.01 mm and permitting reading to within 0.005 mm. The pressure exerted on the specimen shall be 0.1 MPa ± 10 %. The accuracy of measurement, when checked by a setting gauge, shall be within 0.005 mm.

3.1.2 A device as described in Sub-clause **3.1.1**, but with a pressure of 0.7 MPa ± 10 % exerted on the specimen.

3.1.3 A device as described in Sub-clause **3.1.1**, but with a pressure of 7.0 MPa ± 10 % exerted on the specimen.

3.1.4 Test apparatus capable of producing a constant pressure of 30 MPa ± 10 % uniformly distributed over the faces of the test specimen. It consists of a press with parallel plates and a system permitting measurement within ± 0.02 mm.

3.2 Test specimen

3.2.1 Where the material is delivered in plates or in sheets, the test specimen consists of an entire plate or sheet.

3.2.2 Where the material is delivered in rolls, the test specimen consists of a strip taken across the full width of the roll to give an area of 0.2 m^2.

3.2.3 Where the material is delivered in the form of tapes, the test specimen consists of a strip 2 m long.

3.2.4 For commutator separators with a surface area of 10 cm^2 or less, the test specimen consists of five separators to be measured individually.

3.2.5 For commutator separators with a surface area greater than 10 cm^2, the test specimen depends on the method given in the specification for the individual material
(see IEC Publication 371-3-1: *Specification for Insulating Materials Based on Mica, Part 3: Specifications for Individual Materials, Sheet 1: Commutator Separators and Materials*):

a) the test specimen consists of one separator;

b) the test specimen consists of one entire press-gauged stack of separators (separated if necessary by intermediate layers), the number of separators in the press-gauged stack to be specified by the purchaser.

3.2.6 For flat pieces cut to shape other than separators, the test specimen consists of one piece.

3.3 Procedure

The thickness shall be measured by one of the following procedures:

3.3.1 Where materials other than for commutator separators are delivered as sheets (including strips), rolls and tapes, the thickness on each test specimen is measured at ten points uniformly distributed along the diagonals for sheets and along a line which is approximately central for rolls and tapes (not at the edges); the measuring device is that described in Sub-clause **3.1.1** with a pressure of 0.1 MPa.

3.3.2 For commutator separators and for sheets and strips to be used in making commutator separators, one of the following procedures shall be adopted:

3.3.2.1 *Sheets:* the thickness is measured on each specimen as stated in Sub-clause **3.3.1** using the apparatus defined in Sub-clause **3.1.3** with a pressure of 7.0 MPa.

3.3.2.2 *Separators having a surface area of 10 cm^2 or less:* the thickness is measured at one single point chosen at random on each of the five separators using the apparatus defined in Sub-clause **3.1.3** with a pressure of 7.0 MPa.

3.3.2.3 *Separators having a surface area greater than 10 cm^2:* the thickness is measured as in Item a) or Item b) below; the method used is indicated in the specification for the individual material:

a) in the case of separators delivered individually, the thickness is measured at three points uniformly distributed over the test specimen using the apparatus defined in Sub-clause **3.1.3** with a pressure of 7.0 MPa.

b) in the case of separators delivered in press-gauged packeted stacks, each test specimen, consisting of one stack, is measured under the conditions defined in Sub-clause **3.1.4** with a pressure of 30 MPa, ensuring that all the separators in the stack to be tested are properly aligned when the measurement is made.

Before each test the deformation of the press should be measured by carrying out a measurement with a steel block of known dimensions approximately equal to those of the test specimen.

In obtaining the thickness of the single test specimen (d_1), including that of any intermediate layers (d_2), the correction for the deformation of the test apparatus is added to or subtracted from the measured values.

The total thickness of a stack (d), of n separators with ($n - 1$) intermediate layers is given by:

$$d = nd_1 + (n-1)d_2$$

where:

d = thickness of the whole stack composed of n separators and ($n - 1$) intermediate layers

d_1 = thickness of one separator

n = number of separators

d_2 = thickness of intermediate layers

$n - 1$ = number of intermediate layers

3.4 Statement of results

For packeted stacks, report as the thickness of the stack the value of nd_1 and the number of separators per stack. For all other cases, report as the thickness of each test specimen the central value of the results and also report the maximum and minimum values.

4 Density

Determine the density by displacement of liquid. Use a liquid which will not affect the test specimen or be absorbed by it.

In the case of curable materials, use a laminate with trimmed edges of any convenient dimension, but prepared in accordance with Clause **2**.

5 Apparent density

The apparent density (d) is calculated from the central values of the mass per unit area and thickness by means of the following equation:

$$d = \frac{m_a}{d_e \times 10^{-3}} \text{ (g/m}^3\text{)}$$

where:

m_a = mass per unit area (g/m^2)

d_e = thickness (mm)

6 Composition

6.1 Test specimen

The test specimen shall have a mass of approximately 5 g (for thin materials, two pieces of approximately 250 cm^2 are suitable). The entire thickness of material shall be included in the test specimen.

6.2 Mass per unit area in the "as received" condition

The test specimen shall be weighed with an accuracy of 1 mg within 4 h of removal from the original package and at a temperature of 23 ± 2 °C (mass m_1). The area (A) in square metres of the test specimen shall be determined with an accuracy of ± 1 %.

The mass per unit area in the "as received" condition (m_t) is:

$$m_t = \frac{m_1}{A} \text{ (g/m}^2\text{)}$$

6.3 Content of volatiles and mass per unit area of the dried material

The test specimen (mass m_1) shall be heated for 1 h at 150 ± 3 °C, unless otherwise agreed upon between purchaser and supplier. After cooling in a desiccator, the test specimen is weighed (mass m_2).

The volatile content (T_v) is:

$$T_v = \frac{m_1 - m_2}{m_1} 100 \text{ (\%)}$$

The mass per unit area of the dried product (m'_t) is:

$$m'_t = \frac{m_2}{A} \text{ (g/m}^2\text{)}$$

6.4 Binder content

6.4.1 *Material without reinforcement or with inorganic reinforcement*

The test specimen, dried according to Sub-clause **6.3** (mass m_2), is heated in a muffle oven at a temperature of 500 ± 25 °C. Unless otherwise specified, the period of heating shall be 2 h. After cooling in a desiccator, the mass (m_3) is determined.

The binder content (C_b) is:

$$C_b = \frac{m_2 - m_3}{m_2} 100 \text{ (\%)}$$

NOTE In the case of dispute, the heating should be continued to constant mass, the mass being considered constant when consecutive weighings differ by not more than 0.1 %.

The mass per unit area of binder (m'_b) is:

$$m'_b = \frac{m_2 - m_3}{A} \text{ (g/m}^2\text{)}$$

6.4.2 ***Material with organic reinforcement and soluble binder***

The test specimen, dried according to Sub-clause **6.3** (mass m_2), is placed in the extraction thimble of a Soxhlet extraction apparatus with a capacity of 500 cm^3.

The type of solvent as recommended by the supplier shall be capable of dissolving the binder completely, but shall not dissolve the reinforcement. The boiling under reflux is continued for 2 h or longer if necessary for the complete dissolution of the binder. The treated test specimen is taken out of the extraction thimble and shall be dried for half an hour at 135 °C. After cooling in a desiccator, the mass (m_4) is determined.

The binder content (C_b) is:

$$C_b = \frac{m_2 - m_4}{m_2} 100 \ (\%)$$

The mass per unit area of binder (m'_b) is:

$$m'_b = \frac{m_2 - m_4}{A} \ (g/m^2)$$

NOTE The normal extraction time is 2 h. For thicker materials, it may be of help to split the material carefully in order to facilitate penetration of the solvent.

6.4.3 ***Material with organic reinforcement and insoluble binder***

Using values of m_2 (see Sub-clause **6.3**) and m_3 (see Sub-clause **6.4.1**) and the mass of organic reinforcement (m_5) stated by the supplier, the binder content (C_b) is:

$$C_b = \frac{m_2 - (m_3 + m_5)}{m_2} 100 \ (\%)$$

The mass per unit area of binder (m'_b) is:

$$m'_b = \frac{m_2 - (m_3 + m_5)}{A} \ (g/m^2)$$

6.4.4 ***Silicone binder content***

The determination of the silicone content shall be subject to contract. An example of a possible method is given below.

6.4.4.1 *Method of test*

Weigh the test specimen in a previously dried and weighed extraction thimble to the nearest milligramme. The difference in mass is the mass of the specimen.

Put sufficient diethylamine (reagent grade) into a Soxhlet extraction flask to fill the siphon one and a half times. Extract the test specimen completely at a siphon rate of 6 to 10 times per hour (the minimum time of extraction is 4 h for thin materials, but may be much longer for thick materials).

Allow the apparatus to cool, then replace the diethylamine with acetone and extract as before for 1 h 30 min.

Remove the thimble, allow it to dry in air on a watchglass for 10 min, then heat for 30 min in an oven at 105 ± 2 °C.

Cool the thimble in a desiccator, then weigh it to the nearest milligramme. Subtract the weight of the thimble.

6.4.4.2 *Results*

Report the silicone binder content as a percentage to the first decimal place.

$$\text{Silicone binder content} = \frac{\text{loss in mass}}{\text{specimen mass}} 100 \ (\%)$$

6.5 Mass per unit area of reinforcement material (m'_r)

The supplier shall state the mass per unit area of the reinforcement material used. The method for determining this property shall be subject to contract.

Alternatively, one of the following procedures may be used and stated in the contract.

a) For a material with inorganic reinforcement:

On completion of the heating period according to Sub-clause **6.4.1**, carefully separate the reinforcement and weigh (mass m_6).

The mass per unit area of reinforcement material (m'_r) is:

$$m'_r = \frac{m_6}{A} \ (g/m^2)$$

b) For a material with organic reinforcement and soluble binder:

On completion of the extraction according to Sub-clause **6.4.2**, carefully separate the reinforcement and weigh (mass m_7).

The mass per unit area of reinforcement material (m'_r) is:

$$m'_r = \frac{m_7}{A} \ (g/m^2)$$

6.6 Mica content

From the results of the previous tests, the mica content (C_m) and the mass per unit area of mica (m'_m) can be calculated.

For material without reinforcement or with organic reinforcement:

$$C_m = \frac{m_3}{m_2} 100 \ (\%)$$

$$m'_m = \frac{m_3}{A} \ (g/m^2)$$

For material with inorganic reinforcement:

$$C_m = \frac{\frac{m_3}{A} - m'_r}{m'_t} \ 100 \ (\%)$$

$$m'_m = m'_t - m'_b - m'_r \ (g/m^2)$$

6.7 Size of splittings

6.7.1 *Test specimen*

The size of the test specimen of sheet to be tested shall be 300 mm × 300 mm. The test specimen and special test conditions for tape materials are specified in Part 3.

6.7.2 *Method of test*

To remove the shellac bonding material, the test specimen is placed in a tray or shallow bath and boiled with a 15 % aqueous solution of caustic potash (KOH) until disintegration takes place. If the bond cannot be loosened by the above means, any other suitable solvent may be used or the test specimen may be heated in a muffle oven until the binder is sufficiently degraded to permit examination of the splittings. Alternatively, the splittings may be removed mechanically provided no splitting is torn in the process.

After disintegration, the splittings are washed several times with hot water, or with fresh solvent, and then allowed to dry. The size of the splittings is determined with the template given in ISO Standard 67: *Muscovite Mica Blocks, Thins and Films — Grading by Size.*

7 Tensile strength and elongation at break

7.1 Test apparatus

Either a constant rate-of-load machine or a constant rate-of-traverse machine may be used; the machine shall preferably be power-driven and graduated so that a reading of 1 % of the value required by the specification sheet is possible.

7.2 Test specimen

Five test specimens are used. The length of the test specimens shall be such that it allows a length of 200 mm between the jaws of the testing machine.

When testing full-width material or sheets, the width shall be 25 mm; five test specimens shall be cut in the machine direction and five test specimens shall be cut perpendicular to that direction. Test specimens shall be cut so that no two test specimens cut in the same direction contain the same longitudinal threads if a reinforcement is used.

Tape material is tested in the machine direction and in the width as delivered up to a maximum of 25 mm.

7.3 Procedure

Fix a test specimen in the testing machine and apply the load in such a way that the time from the commencement of the application of the load to the moment at which the load corresponding to the specified minimum tensile strength is reached is 60 ± 10 s; continue until the test specimen breaks. Record the breaking force and the elongation at break or failure of one component in reinforced materials.

If the test specimen breaks in or at a jaw of the testing machine, discard the result and make a further test using another test specimen.

When the tensile strength of a join is to be determined, position the join approximately midway between the jaws.

NOTE With some materials, extra precautions may be required to prevent slippage in the jaws of the machine.

7.4 Results

The tensile strength shall be reported in the two directions separately (where applicable). For each direction, take the central value of the five loads at break and calculate the tensile strength of the material in the relevant direction expressed in newtons per 10 mm of width.

The elongation result is the central value of the five measurements expressed as percentages of the original length; the maximum and minimum values are also reported.

8 Flexural strength and elastic modulus in bend

8.1 Test specimen

For the determination of the flexural strength take five test pieces in the direction parallel to one edge and another five in a direction at right angles to this. Each test piece shall be of a length not less than 20 times the measured thickness, of a width 10 mm to 25 mm and of a thickness 4 ± 0.2 mm.

For the determination of the elastic modulus, two sets of two similar test pieces are taken.

In the case of curable materials, cut the test pieces from a laminate prepared in accordance with Clause 2.

8.2 Procedure

Use the methods described in ISO Standard 178: Plastics — Determination of Flexural Properties of Rigid Plastics. This determination shall be made at temperatures of 23 °C and 155 °C.