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**Methods of test for electrical
insulating materials based on mica**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Electrical Insulating and Advanced Performance Materials Industrial Association (JEIA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS C 2116:2004** is replaced with this Standard.

This **JIS** document is protected by the Copyright Law.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public or utility model right. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public or the utility model right.

Methods of test for electrical insulating materials based on mica

Introduction

This Japanese Industrial Standard has been prepared based on the third edition of **IEC 60371-2** published in 2004 with some modifications of the technical contents to accommodate the current situation in the Japanese market.

For information, Annex JA describes the test methods which are specified in individual product standards, but not specified in the corresponding International Standard, and Annex JB describes the test methods which are widely used in Japan for testing the mica based products, but not specified in the corresponding International Standard.

The portions with continuous sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with the explanations is given in Annex JC.

1 Scope

This Standard applies to methods of test for electrical insulating materials based on mica.

In this Standard, where test requirements are given for both method A and method B in parallel, either of the methods shall be selected to be applied.

NOTE : The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

IEC 60371-2:2004 *Specification for insulating materials based on mica—Part 2: Methods of test* (MOD)

The symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. For standards with the year indication, only the editions of the indicated year shall be applied and the revisions (including amendments) made thereafter shall not be applied. For those without the indication of the year, the most recent edition (including amendments) shall be applied.

JIS B 7502 *Micrometer callipers*

JIS C 2110-1:2010 *Solid electrical insulating materials—Test methods for electric strength—Part 1: Tests at power frequencies*

NOTE : Corresponding International Standard: IEC 60243-1:1998 *Electrical strength of insulating materials—Test methods—Part 1: Tests at power frequencies* (MOD)

JIS C 2250 *General rules for electrical insulating mica products*

JIS K 7171:2008 *Plastics—Determination of flexural properties*

NOTE : Corresponding International Standard: ISO 178:2001 *Plastics—Determination of flexural properties* and Amendment 1 (2004) (IDT)

IEC 60216 (all parts) *Electrical insulating materials—Thermal endurance properties*

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 60371-3 (all parts) *Specification for insulating materials based on mica—Part 3: Specifications for individual materials*

3 Terms and definitions

For the purposes of this Standard, the terms and definitions given in **JIS C 2250** apply.

4 Preparation of test specimens for curable materials

4.1 General

Test specimens are prepared in accordance with the methods given in **4.2** or **4.3** in the following. The methods are applicable only to curable materials.

4.2 Method 1

Clean off all loose particles and projecting fibres from sufficient material.

Cut and stack the pieces in dimensions required to provide the test specimens for the particular test. 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 $160\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$, unless otherwise specified.

Place the test laminate in the centre of two caul plates not exceeding 1.5 mm in thickness and at a temperature of $15\text{ }^{\circ}\text{C}$ to $35\text{ }^{\circ}\text{C}$.

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

NOTE : When forming a test piece, blocks of metal, etc. of a specified thickness may be placed as stops in the outside of the product in order to obtain the specified thickness.

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

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

Remove the test specimen and post-cure for the length of time and at the temperature given in **IEC 60371-3** standard series (hereafter referred to as “individual product standards”) or according to the recommendation of the supplier.

Unless otherwise specified, condition the test specimen for 24 h in a controlled atmosphere $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ in temperature and $(50 \pm 5)\%$ in relative humidity.

4.3 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 in dimensions required to provide the test specimens for the particular test.

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

- a) Cut the tape in pieces to the specified length of the laminate. Stack the pieces parallel and half-overlapped. The second and 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, shall be put in the press.

The following press procedure, illustrated in figure 2, provides an example. Other press procedures shall be subject to agreement between the purchaser and the supplier.

- Close the cold press and bring it to a pressure of 0.15 MPa.
- Heat the press to $70\text{ }^{\circ}\text{C}$ under pressure of 0.15 MPa.
- Reduce the pressure down to zero, open the press for a short time (ventilate).
- Heat the press to $90\text{ }^{\circ}\text{C}$ under pressure of 0.15 MPa.
- Reduce the pressure down to zero, open the press for a short time (ventilate).
- Heat the press to $110\text{ }^{\circ}\text{C}$ under pressure of 0.15 MPa.
- Reduce the pressure down to zero, open the press for a short time (ventilate).
- Heat the press to $160\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ under pressure of 0.15 MPa 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 MPa.
- Cure under 3 MPa and $160\text{ }^{\circ}\text{C}$ 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 temperature specified in the individual product standards or according to the recommendation of the supplier.

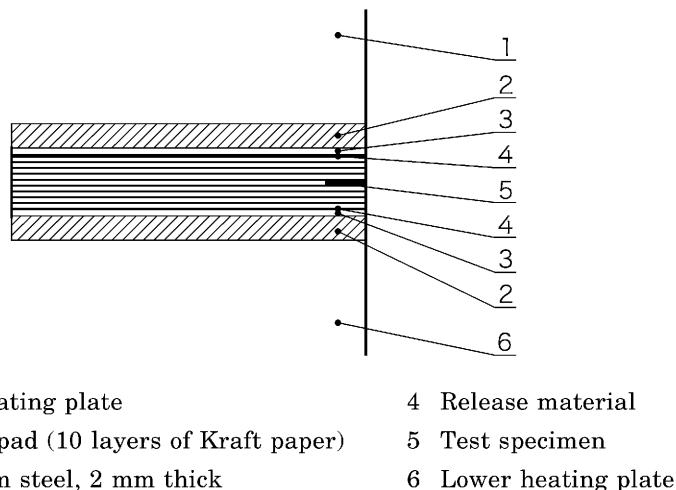


Figure 1 Assembly of stacks for preparing test laminates

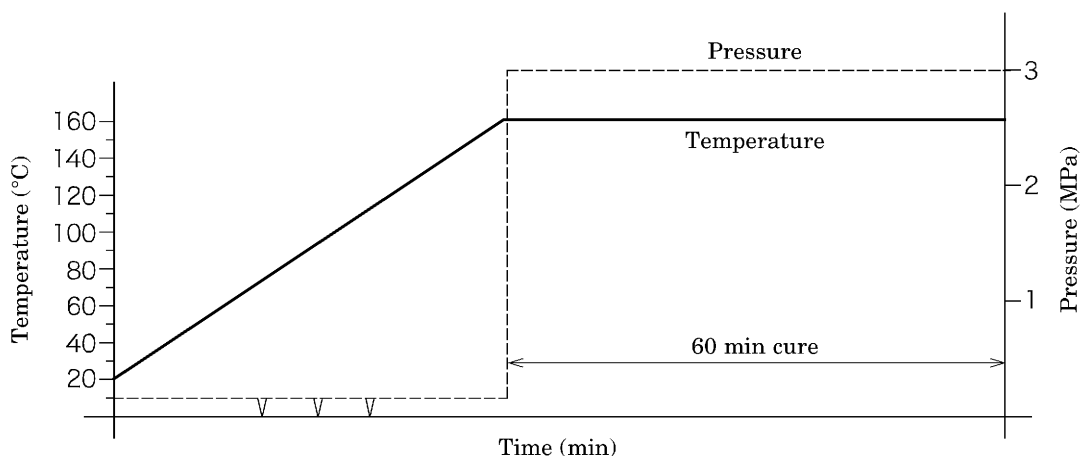


Figure 2 Conditions for press procedure

5 Thickness

5.1 Test apparatus

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

5.1.1 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 $0.1 \text{ MPa} \pm 10 \%$. The accuracy of measurement, when checked by a setting gauge, shall be within 0.005 mm. Measurement shall be made at room temperature and at least 5 s after the application of pressure.

5.1.2 A device as described in **5.1.1**, but with a pressure of $0.7 \text{ MPa} \pm 10 \%$ exerted on the specimen.

5.1.3 A device as described in **5.1.1**, but with a pressure of $7.0 \text{ MPa} \pm 10 \%$ exerted on test specimen.

5.1.4 Test apparatus capable of producing a constant pressure of $30 \text{ MPa} \pm 10 \%$ uniformly distributed over the faces of the test specimen. It shall consist of a press with parallel plates and a system permitting measurement within 0.02 mm.

5.1.5 Micrometer callipers specified in **JIS B 7502**.

5.2 Test specimen

5.2.1 Where the material is delivered in plates or in sheets, the test specimen shall consist of an entire plate or sheet.

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

5.2.3 Where the material is delivered in the form of tapes, the test specimen shall consist of a strip 2 m long.

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

5.2.5 For commutator separators with a surface area greater than 10 cm^2 , the test specimen depends on the method given in the specification in the individual product standards.

- a) The test specimen shall consist of one separator.
- b) The test specimen shall consist 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.

5.2.6 For flat pieces cut to shape other than separators, the test specimen shall consist of one piece.

5.3 Procedure

The procedure shall be as follows.

5.3.1 Where material other than for commutator separators are delivered as sheets (including strips), rolls and tapes, the thickness on each test specimen shall be measured at ten points uniformly distributed along the diagonals for sheets and along a line which shall be approximately central for rolls and tapes (not at the edges); measuring device shall be that described in **5.1.1** or **5.1.5**.

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

5.3.2.1 Sheets: the thickness shall be measured on each specimen as stated in **5.3.1** using the device defined in **5.1.3** or **5.1.5**.

5.3.2.2 Commutator separators having a surface area of 10 cm^2 or less: the thickness shall be measured at one single point chosen at random on each of the five separators using the device defined in **5.1.3** or **5.1.5**.

5.3.2.3 Commutator separators having a surface area greater than 10 cm²: the thickness shall be measured according to either of the followings below; the method used is indicated in the specification of the individual product standards.

- a) In the case of separators delivered individually, the thickness shall be measured at three points uniformly distributed over the test specimen using the device defined in **5.1.3** or **5.1.5**.
- b) In the case of separators delivered in press-gauged packeted stacks, each test specimen, consisting of one stack, shall be measured using the apparatus specified in **5.1.4** 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 shall 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 one separator, d_1 , the whole thickness including the thickness of intermediate layers, d_2 , the correction for the deformation of the test apparatus shall be added to or subtracted from the measured values. The total thickness of a stack, d , of n separators with $(n - 1)$ intermediate layers shall be given by:

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

where, d : the thickness of the whole stack (mm)
 d_1 : the thickness of one separator (mm)
 n : the number of separators
 d_2 : the thickness of intermediate layer (mm)
 $n - 1$: the number of intermediate layers

5.4 Statement of results

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

6 Width and length

6.1 General

The measurement of width and length shall be performed as specified in **6.2** to **6.4**.

It may be performed in accordance with the agreement between the purchaser and the supplier.

6.2 Measuring apparatus

For the measuring apparatus of width and length, any of the following shall be used according to the material under test.

6.2.1 Measuring apparatus of which the graduations are in divisions of 0.05 mm or smaller.

6.2.2 Measuring apparatus of which the graduations are in divisions of 1.0 mm or smaller.

6.2.3 Measuring apparatus of which the graduations are in divisions of 1.0 m or smaller.

6.3 Procedure

The measurement procedure of width and length shall be as follows.

6.3.1 When the material under test is delivered in small cut plates, one or more points shall be measured using the measuring apparatus specified in **6.2.1**.

6.3.2 When the material under test is delivered in sheets or in plates, three or more points shall be measured using the measuring apparatus specified in **6.2.2**.

6.3.3 When the material under test is delivered in the form of tapes or rolls, for the width, one or more points shall be measured using the measuring apparatus specified in **6.2.2** and for the length, one point shall be measured using the measuring apparatus specified in **6.2.3**.

6.4 Statement of results

When multiple measurements are performed, the average shall be reported.

7 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 4.

8 Calculated density

The density can be calculated from the average values of the mass per unit area and the thickness by means of the following equation:

$$d = \frac{m_a}{d_e} \times 10^{-3}$$

where, d : calculated density (g/cm³)

m_a : mass per unit area of test specimen (g/m²)

d_e : average value of thickness (mm)

9 Composition

9.1 Test specimen

The test specimen shall have a mass of approximately 5 g (for thin materials, two pieces of approximately 250 cm² are suitable). The entire thickness of material shall be included in the test specimen.

9.2 Mass per unit area in the “as received” condition

The mass, m_1 , of 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 $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. The area A in square metres of the test specimen shall be determined with an accuracy of $\pm 1\%$.

The mass per unit area in the “as received” condition, m_a is:

$$m_a = \frac{m_1}{A}$$

where, m_a : mass of test specimen per unit area (in “as received” condition) (g/m^2)

m_1 : mass of test specimen (g)

A : area of test specimen (m^2)

9.3 Content of volatiles and mass per unit area of dried test specimen

The test specimen, m_1 in mass, shall be heated for 1 h at $150\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$. After cooling in a desiccator, the mass, m_2 , of test specimen shall be weighed.

The volatile content, T_v , is:

$$T_v = \frac{m_1 - m_2}{m_1} \times 100$$

The mass per unit area, m'_a , of the dried test specimen is:

$$m'_a = \frac{m_2}{A}$$

where, T_v : volatile content (%)

m_1 : mass of test specimen (g)

m_2 : mass of dried test specimen (g)

m'_a : mass per unit area of dried test specimen (g/m^2)

A : area of test specimen (m^2)

9.4 Binder content

9.4.1 Method A

9.4.1.1 Material without reinforcement or with inorganic reinforcement

The test specimen, dried according to 9.3 and m_2 in mass shall be heated in a muffle oven at a temperature of $500\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. Unless otherwise specified, the period of heating shall be 2 h. After cooling in a desiccator, the mass, m_3 , shall be determined.

The binder content, C_b , is:

$$C_b = \frac{m_2 - m_3}{m_2} \times 100$$

where, C_b : binder content (%)

m_2 : mass of dried test specimen (g)

m_3 : mass of test specimen after heating in a muffle oven (g)

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:

$$m'_b = \frac{m_2 - m_3}{A}$$

where, m'_b : mass per unit area of binder (g/m²)
 m_2 : mass of dried test specimen (g)
 m_3 : mass of test specimen after heating in a muffle oven (g)
 A : area of test specimen (m²)

9.4.1.2 Material with organic reinforcement and soluble binder

The test specimen, dried according to 9.3 and m_2 in mass, shall be placed in the extraction thimble of a Soxhlet extraction apparatus with a capacity of 500 cm³. The solvent shall be capable of dissolving the binder completely, but shall not dissolve the reinforcement. The boiling under reflux shall be continued for 2 h or longer if necessary for the complete dissolution of the binder. The treated test specimen shall be taken out of the extraction thimble and dried for 30 min at 135 °C. After cooling in a desiccator, the mass, m_4 , shall be determined.

The binder content, C_b is:

$$C_b = \frac{m_2 - m_4}{m_2} \times 100$$

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

$$m'_b = \frac{m_2 - m_4}{A}$$

where, C_b : binder content (%)
 m_2 : mass of dried test specimen (g)
 m_4 : mass of test specimen after extraction (g)
 m'_b : mass per unit area of binder (g/m²)
 A : area of test specimen (m²)

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.

9.4.1.3 Material with organic reinforcement and insoluble binder

Using values of m_2 (see 9.3) and m_3 (see 9.4.1.1) and the mass of organic reinforcement, m_5 , stated by the supplier, the binder content, C_b is:

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

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

$$m'_b = \frac{m_2 - (m_3 + A \times m_5)}{A}$$

- where, C_b : binder content (%)
 m_2 : mass of dried test specimen (g)
 m_3 : mass of test specimen after heating in a muffle oven (g)
 m_5 : mass per unit area of organic reinforcement (g/m²)
 m'_b : mass per unit area of binder (g/m²)
 A : area of test specimen (m²)

9.4.1.4 Silicone binder content

The determination of the silicone content shall be subject to agreement between the purchaser and the supplier. An example of a possible method is given below.

- a) **Method of test** Weigh the test specimen in a previously dried and weighed extraction thimble to the nearest milligram. The difference in mass shall be taken as the mass of the specimen m_6 . 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 1.5 h. 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 °C ± 2 °C. Cool the thimble in a desiccator, then weigh it to the nearest milligram. Subtract the weight of the thimble, and obtain the mass after extraction, m_7 .
- b) **Statement of results** Report the silicone binder content, C_b , percentage to the first decimal place.

$$C_b = \frac{m_6 - m_7}{m_6} \times 100$$

- where, C_b : binder content (%)
 m_6 : mass of test specimen (g)
 m_7 : mass of test specimen after extraction (g)

9.4.2 Method B

The procedure of treatment shall be as specified in 9.4.1.1.

For materials with organic reinforcement, the mass per unit area of the reinforcement material may be determined in accordance with the agreement between the purchaser and the supplier.

9.4.2.1 In the case of binders other than silicone binders

Obtain the binder content according to either of the following.