
**Adhesives — Test methods for
isotropic electrically conductive
adhesives —**

**Part 2:
Determination of electrical
characteristics for use in electronic
assemblies**

*Adhésifs — Méthodes d'essai pour adhésifs à conductivité électrique
isotrope —*

*Partie 2: Détermination des propriétés électriques pour utilisation
dans des assemblages électroniques*



Reference number
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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.

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The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

ISO 16525 consists of the following parts, under the general title *Adhesives — Test methods for isotropic electrically conductive adhesives*:

- *Part 1: General test methods*
- *Part 2: Determination of electric characteristics for use in electronic assemblies*
- *Part 3: Determination of heat-transfer properties*
- *Part 4: Determination of shear strength and electrical resistance using rigid-to-rigid bonded assemblies*
- *Part 5: Determination of shear fatigue*
- *Part 6: Determination of pendulum-type shear impact*
- *Part 7: Environmental test methods*
- *Part 8: Electrochemical migration test methods*
- *Part 9: Determination of high-speed signal-transmission characteristics*

Adhesives — Test methods for isotropic electrically conductive adhesives —

Part 2: Determination of electrical characteristics for use in electronic assemblies

SAFETY STATEMENT — Persons using this part of ISO 16525 should be familiar with normal laboratory practice. This part of ISO 16525 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory conditions.

IMPORTANT — Certain procedures specified in this part of ISO 16525 might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This part of ISO 16525 specifies test methods for isotropic electrically conductive adhesives used in wiring, die attach, and surface assembly of printed circuit boards of electronic devices. The test methods focus on volume and interfacial contact resistivity.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

ISO 554, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 80000-1, *Quantities and units — Part 1: General*

IEC 60468, *Method of measurement of resistivity of metallic materials*

IEC 61249-2-7, *Materials for printed boards and other interconnecting structures - Part 2-7: Reinforced base materials clad and unclad - Epoxide woven E-glass laminated sheet of defined flammability (vertical burning test), copper-clad*

EN 923, *Adhesives – Terms and definitions*

ASTM B539-02, *Standard Test Methods for Measuring Resistance of Electrical Connections (Static Contacts)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472, ASTM B539-02 and EN 923 and the following apply.

3.1 isotropic electrically conductive adhesive

<electronic assembly> resin that contains fillers, which provide electrical conduction, and functions as an adhesive

3.2 volume resistivity

ρ
electrical resistance of the isotropic electrically conductive adhesive for a given cross-sectional area or given length

Note 1 to entry: Electrical volume resistivity is converted to resistance per given cross-sectional area or given length of the isotropic electrically conductive adhesive. This part of ISO 16525 specifies measurement methods for an isotropic electrically conductive adhesive, which is applied to a circuit board that is similar to the one used in practice.

Note 2 to entry: It is expressed as ohm metre ($\Omega \cdot m$).

3.3 interfacial contact resistivity

ρ_i
electrical resistance that is generated on the contact surface between the isotropic electrically conductive adhesive and the adherend

Note 1 to entry: It is expressed as electrical resistance per given cross-sectional area ($\Omega \cdot m^2$).

Note 2 to entry: Interfacial contact resistance is generated on the contact surface between the isotropic electrically conductive adhesive and the electrode, and it is thought to be associated with the dispersion of the metal component in the vicinity of the electrode interface, oxide film, and the arrangement of conductive particles contained in the isotropic electrically conductive adhesive. In addition, interfacial contact resistivity fluctuates during the endurance test. Apart from the resistance component brought by electrically volume resistivity of the isotropic electrically conductive adhesive, if a resistance component is placed on the contact surface, interfacial resistance is expressed by resistivity per given area and is converted to resistance per given cross-sectional area or length of the isotropic electrically conductive adhesive.

3.4 four-probe method

method for measuring resistance that consists of two terminals for current application and two terminals for voltage measurement

3.5 dry circuit

method for measuring resistance that prevents open-circuit voltage at the time of measurement from exceeding a specific level so that the thin insulating layer that develops at the contact surface cannot be broken

Note 1 to entry: The maximum current limit is 100 mA and voltage is 20 mV in order to prevent breakdown of the microstructure of adhesives.

4 Principle

The test circuit board consists of two or more terminals that are connected using an isotropic electrically conductive adhesive. The measurement of a potential difference, when a current is applied to one of the terminals, can be used for the calculation of electrically volume resistivity and interfacial contact resistivity.

NOTE This test uses the electrical characteristics of isotropic electrically conductive adhesives, which are used for connection of electronic circuits. This part of ISO 16525 describes electrically volume resistivity, which shows the electric characteristic of the isotropic electrically conductive adhesive, and interfacial resistance, which is specific to the isotropic electrically conductive adhesive.

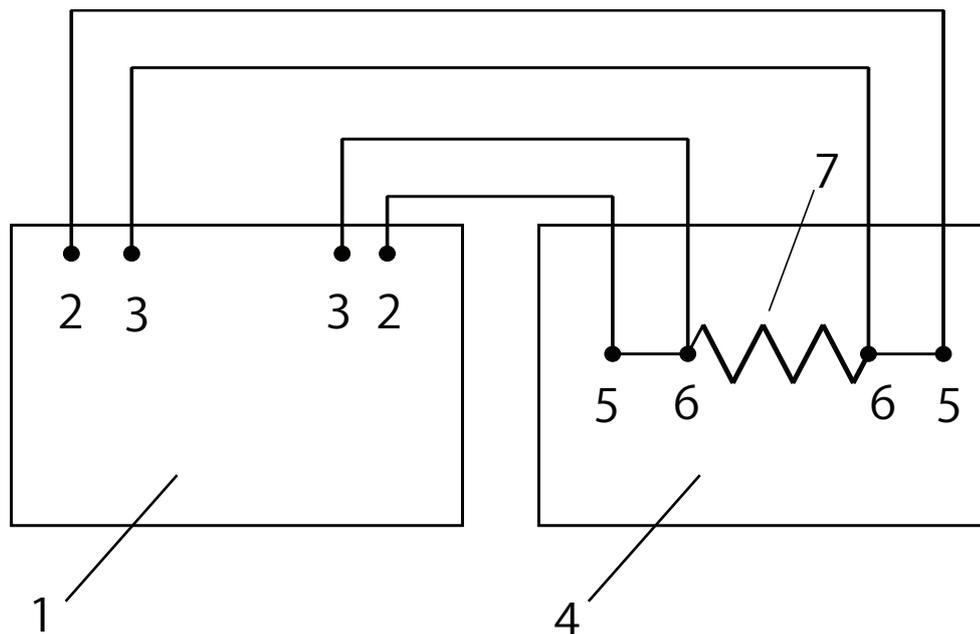
On the contact surface of the isotropic electrically conductive adhesives, a resistance component is present, which can be regarded as interfacial resistance. This might be so high that it cannot be neglected when compared with electrically volume resistivity of the isotropic electrically conductive adhesive. This test provides a measurement, which measures this component only. This test is characterized by calculation of resistivity from a current passing through the test circuit and voltage.

5 Apparatus

5.1 Test apparatus, consisting of a current source and a voltage meter, both as specified in IEC 60468. The potential difference is measured at the voltage terminals by applying a current to the printed circuit board using the current source from the current terminals in a specific direction. [Figure 1](#) and [Figure 2](#) show examples of configuration of the apparatus. A digital ohmmeter, which combines all these devices, may be used as an alternative.

It is preferable to use a dry circuit-type apparatus because it does not affect the irreversibility of conductivity of the interface of the isotropic electrically conductive adhesive. For measurement of interfacial resistance, it is preferable to use a multichannel ohmmeter because it is necessary to switch from one pair of terminals to the other and the lengths of the wires differs.

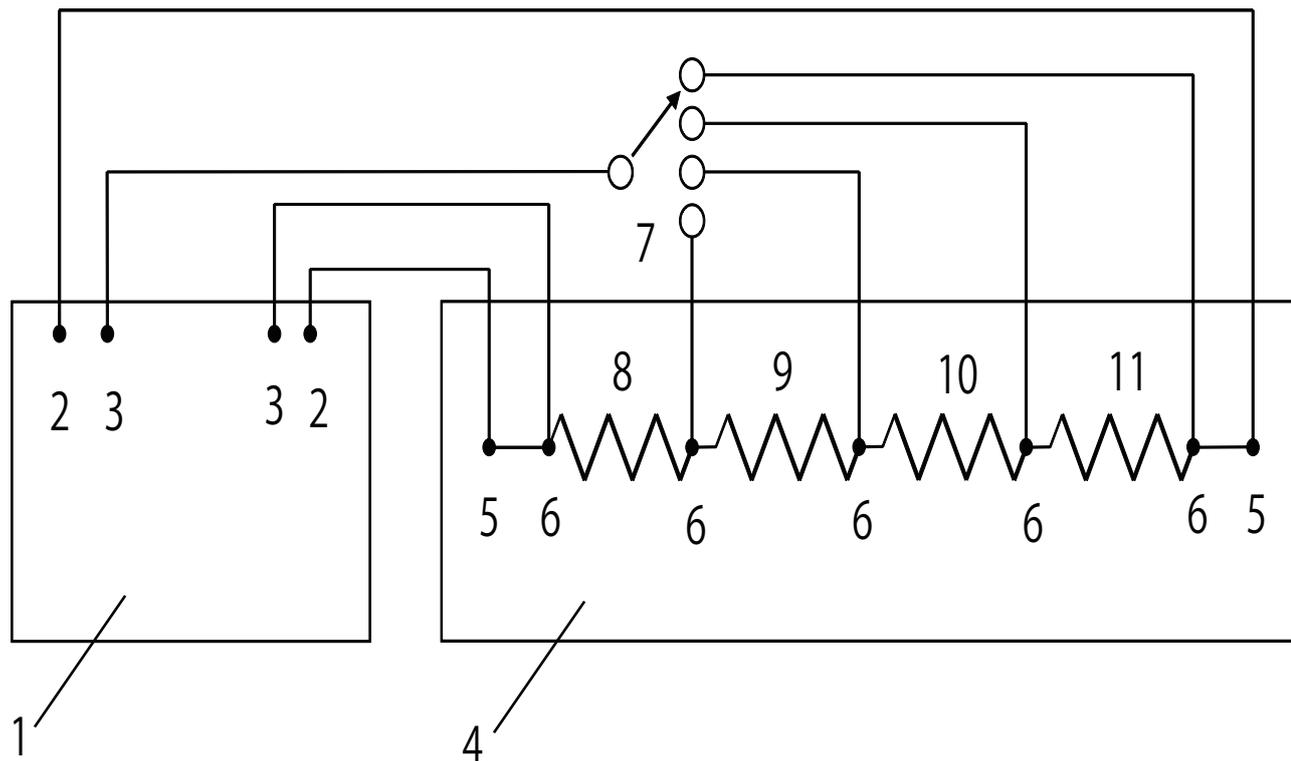
Resistance is obtained from the voltage drop when a constant current is applied. Therefore, set a current value so that voltage resolution can be sufficiently small compared to the measured voltage. In the unlikely event that the specimen generates heat because an excessively high current occurs, reduce the current value and shorten the period for each measurement session. Usually, a maximum current is recommended to be approximately 10 mA.



Key

- | | |
|--------------------------------|-------------------|
| 1 ohmmeter | 5 current probe |
| 2 current source terminal | 6 voltage probe |
| 3 voltage measurement terminal | 7 resistance, R |
| 4 test circuit board | |

Figure 1 — Test apparatus — For measurement of electrically volume resistivity



Key

- | | | | |
|---|------------------------------|----|---------------------|
| 1 | multichannel ohmmeter | 7 | channel switch |
| 2 | current source terminal | 8 | resistance 1, R_1 |
| 3 | voltage measurement terminal | 9 | resistance 2, R_2 |
| 4 | test circuit board | 10 | resistance 3, R_3 |
| 5 | current probe | 11 | resistance 4, R_4 |
| 6 | voltage probe | | |

Figure 2 — Test apparatus — For measurement of interfacial contact resistivity

5.2 Test circuit board, fulfilling the following requirements.

- Material of substrate: use a glass fabric-based epoxy resin copper-clad laminate specified as a general-purpose, single-sided substrate, as described in IEC 61249-2-7.
- Thickness of substrate: the thickness of substrates is $(1,6 \pm 0,2)$ mm or that specified in IEC 61249-2-7.
- Pattern and dimensions of circuit board: a basic pattern for circuit boards to measure electrically volume resistivity and interfacial contact resistivity is shown in [Figure 3](#) (for measurement of electrically volume resistivity) and [Figure 4](#) (for measurement of interfacial contact resistivity), respectively.

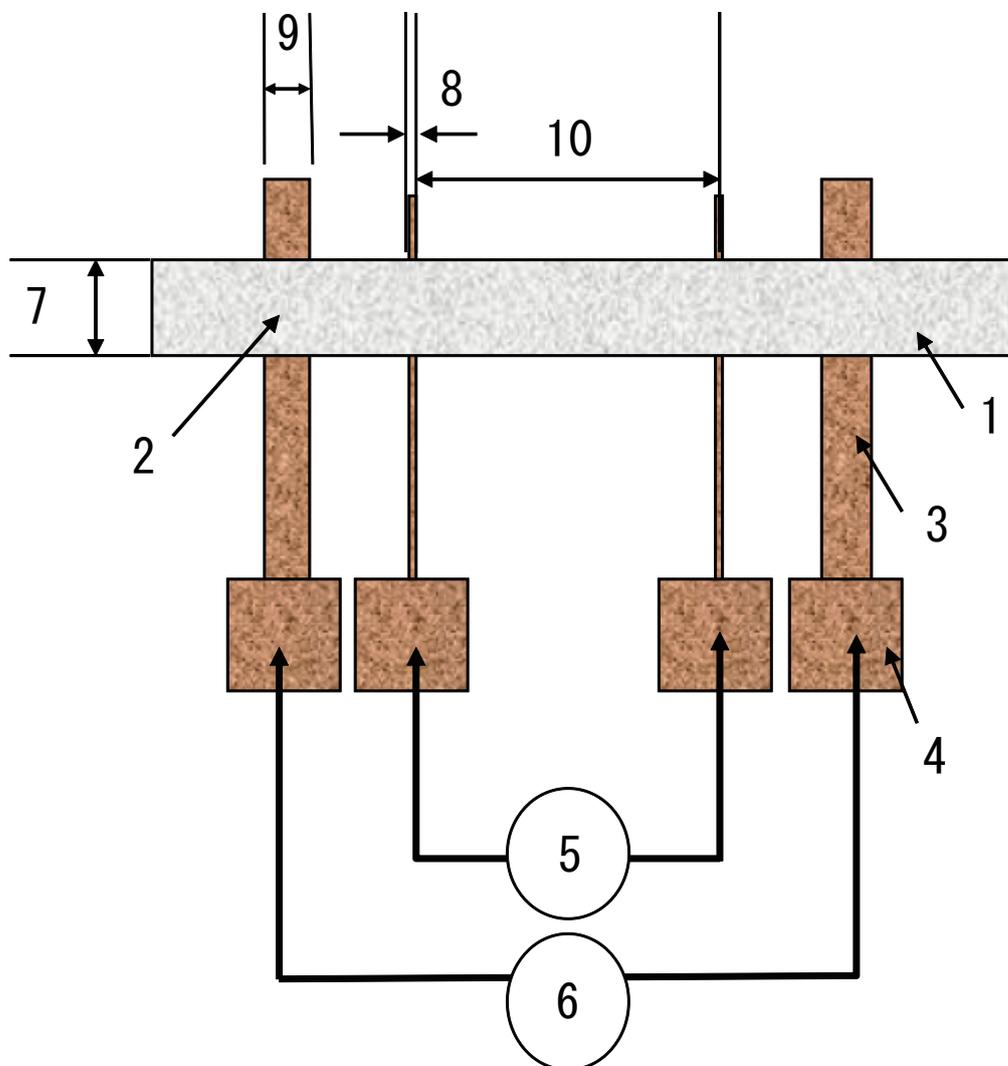
The allowed values for dimension a in [Figure 3](#) are from 2 mm to 10 mm and for l are from 20 mm to 100 mm. For workability, the recommended values for dimensions a and l are 10 mm and 50 mm, respectively. Diameter, b , of the wire for measuring a voltage drop should be so thin that it does not affect the measurement and is recommended to be 0,5 mm.

The recommended dimension for a in [Figure 4](#) is 0,8 mm. When dimension a is lower than this value, fluctuation of the measurement becomes large. When it is larger than this value, the measurement sensitivity for interfacial resistance is reduced. The wires connected to the joint, at which measurement is performed, are taken out from both ends so that the current distribution at the location of measurement

will be uniform. Dimension b should be as small as possible so that it does not affect measurement of a voltage drop and is recommended to be 0,1 mm.

These dimensions should be observed because they are designed to minimize their impacts on measurement.

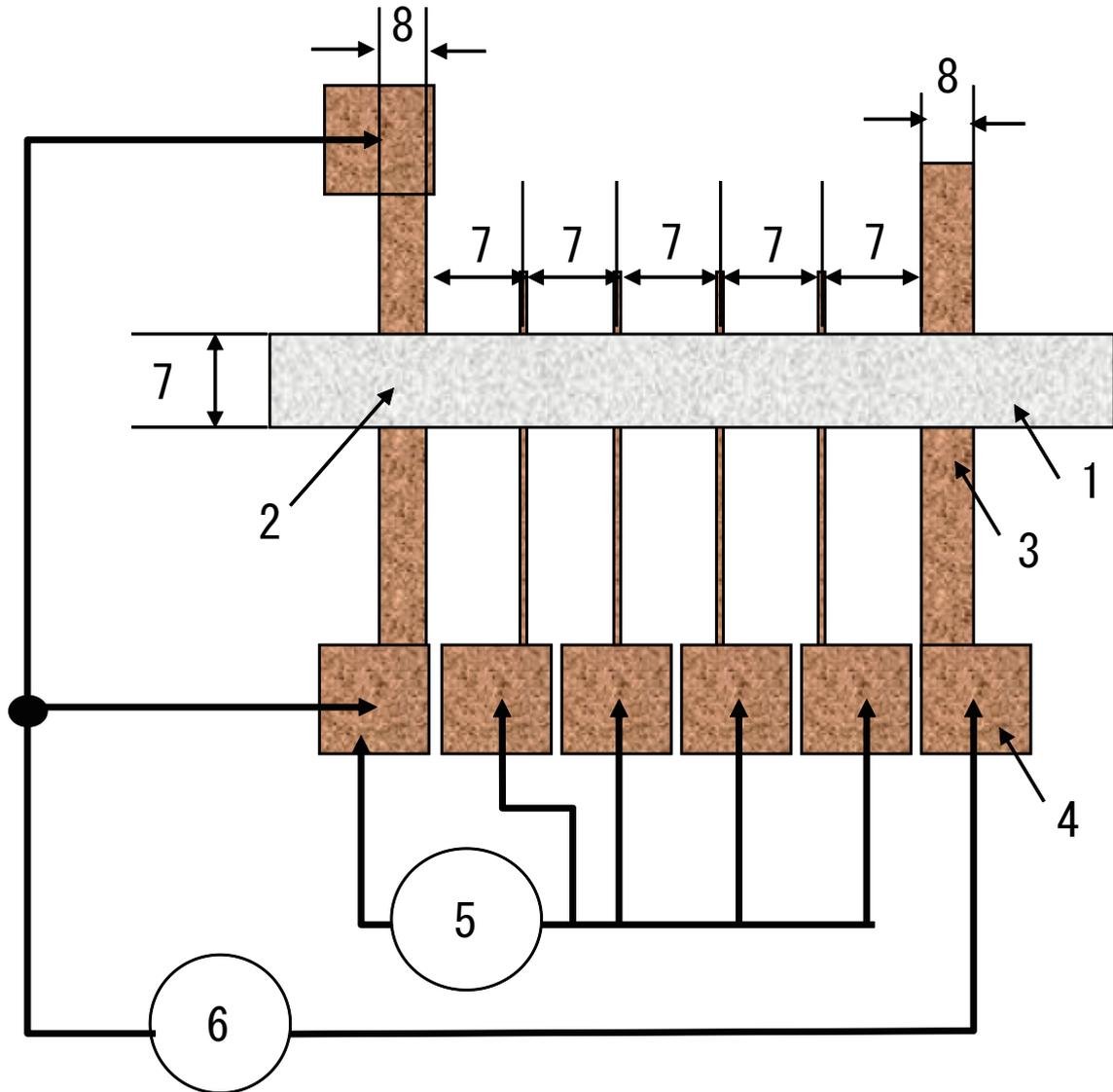
- d) Plating of the electrode surface of the circuit board: in [Figure 3](#), an antioxidant should be applied to the substrate to prevent oxidation of copper and other metals. In [Figure 4](#), plating of the electrode surface is subject to requirements for uses and/or requirements of the delivering and receiving parties.



Key

- | | |
|--|-------------------------------------|
| 1 isotropic conductive adhesives (ICA) | 6 current sources |
| 2 adhesion area (back/rear) | 7 width of ICA line, a |
| 3 metal strip line | 8 width of voltage line, b |
| 4 wire attachment pad | 9 width of current source line, c |
| 5 voltage meter (digital multimeters) | 10 length of ICA, l |

Figure 3 — An example of configuration of test circuit board for measurement of electrically volume resistivity



Key

- | | | | |
|---|--------------------------------------|---|--------------------------------------|
| 1 | isotropic conductive adhesives (ICA) | 5 | voltage meter (digital multimeters) |
| 2 | adhesion area (back/rear) | 6 | current sources |
| 3 | metal strip line | 7 | width of ICA line, a |
| 4 | wire attachment pad | 8 | width of current source line, $1/2a$ |

Figure 4 — Example of configuration of test circuit board for measurement of interfacial contact resistivity

e) Preparation of test circuit board: specified as follows.

1) Isotropic electrically conductive adhesive

In terms of general behaviour and processes, use a paste-type isotropic electrically conductive adhesive containing an organic binder, generally heat-curing resin, in which metal particles or flakes disperse.

2) Preparation

Form a pattern of wires on the test substrate by screen-printing the isotropic electrically conductive adhesive at intervals of a . When the adhesive applied has a low viscosity, it affects precision of

dimensions. In such a case, it is preferable to make the dams with width a intervals on the substrate in [Figure 3](#). The dams are made using heat-resistant adhesive tape, for example. In [Figure 4](#), if it is difficult to form a pattern by screen-printing, it is possible to lay solder resist with width a on the substrate and apply the isotropic electrically conductive adhesive to the area over the solder resist windows. It can be measured because the measured current is limited by solder resistance. Curing conditions depend on the specifications of the isotropic electrically conductive adhesive.

3) Thickness and precision of wire

Measure the thickness, t , in millimetres, of the isotropic electrically conductive adhesive to a precision of 0,01 mm.

First, measure the thickness of the circuit board, t_b , with the micrometer.

Next, measure the total thickness of the circuit board and the isotropic electrically conductive adhesive, t_a .

t_b and t_a should be measured at the same point. t is calculated by:

$$t_a - t_b \tag{1}$$

It is preferable to measure multiple points and calculate the average using Formula (2):

$$\frac{1}{t} = \frac{n}{(t_1 + t_2 + t_3 \dots)} \tag{2}$$

where

t is the average thickness;

n is the number of measurement points.

If the isotropic electrically conductive adhesive has low elasticity, or the thickness is not enough, it is preferable to measure the thickness by an optical displacement meter.

The thickness of the isotropic electrically conductive adhesive should preferably be 0,10 mm to 0,15 mm.

- f) Wiring, fulfilling the following conditions: the resistance of conductors connecting the test circuit board to the resistance measurement system shall be 0,01 Ω or less. To connect the conductor to the electrode on the test circuit board, use the solder.

6 Procedure

6.1 Atmospheric conditions

The test atmospheric conditions should, in principle, be standard temperature class 5 [i.e. (25 \pm 5) $^{\circ}$ C] and standard humidity class 10 [i.e. (50 \pm 10) %] as specified in ISO 554. Other atmospheric conditions can be used by mutual agreement between the parties. In such a case, record the temperature and humidity used in a test report.

6.2 Measurement and calculation

- a) Volume resistivity

To measure electrically volume resistivity, apply constant current i (mA) to the test circuit board, and record voltage drop, V_d (mV), measured by the voltage meter. Calculate electrically volume resistivity, ρ , using Formula (3). Round off the result in accordance with ISO 80000-1.

$$\rho = \frac{V_d}{i} \times \frac{(a \times t)}{l} \quad (3)$$

where

ρ is the resistivity of the isotropic electrically conductive adhesive (Ωmm);

V_d is the voltage drop measured (mV);

i is the current applied (mA);

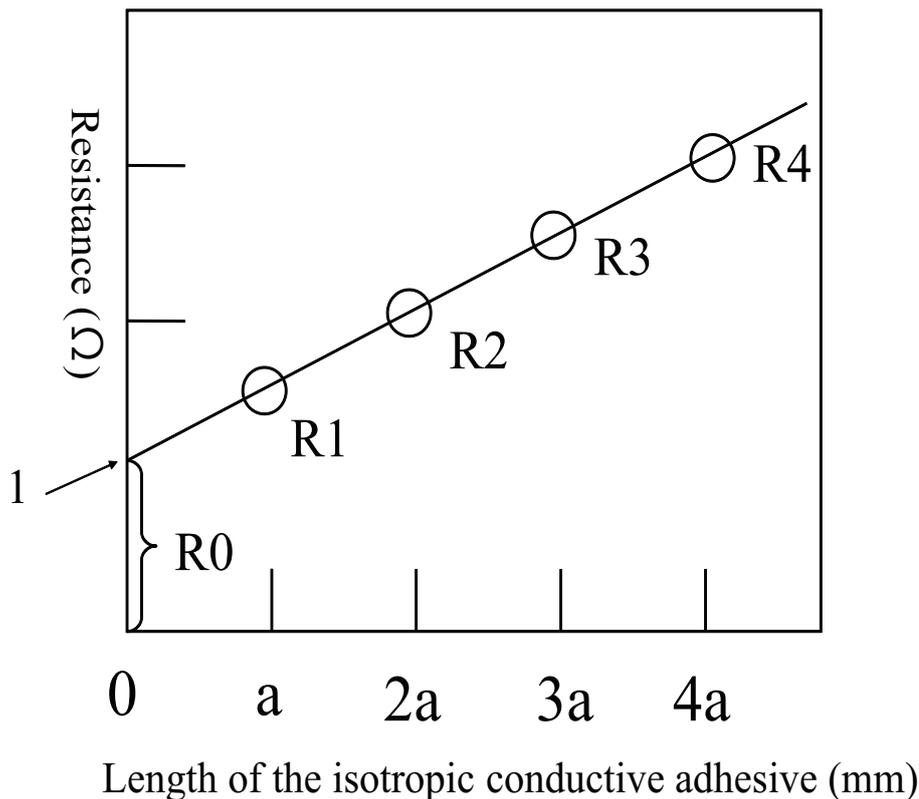
a is the width of the isotropic electrically conductive adhesive (mm);

t is the thickness of the isotropic electrically conductive adhesive (mm);

l is the length of the isotropic electrically conductive adhesive (mm).

b) Interfacial contact resistivity

To obtain interfacial contact resistivity, apply constant current i (mA) to the test circuit board, and measure the voltage at each voltage terminal one after another. Data measured are referred to as V_{d1} , V_{d2} , V_{d3} , and V_{d4} . Resistance values, which are obtained through dividing each voltage by its corresponding current applied, are referred to as R_1 , R_2 , R_3 , and R_4 , respectively. As shown in [Figure 5](#), plot integral times of dimension a along the X-axis and R_1 , R_2 , R_3 , and R_4 along the Y-axis. Then, apply the method of least squares to these four points to obtain the Y-intercept R_0 of the line.



Key
 1 Y intercept

Figure 5 — Calculation of interfacial contact resistivity

Calculate interfacial contact resistivity, ρ_i , using Formula (4). Present the result in accordance with ISO 80000-1.

$$\rho_i = R_0 \times \frac{a^2}{2} \tag{4}$$

where

ρ_i is the interfacial contact resistivity of the isotropic electrically conductive adhesive ($\Omega \cdot \text{mm}^2$);

R_0 is the Y-intercept obtained from the plots (Ω);

a is the wire width of the isotropic electrically conductive adhesive (mm).

6.3 Judgement

The following are examples of the criteria for judging calculations.

- a) Volume resistivity: the lower the volume resistivity, the higher the conduction. However, the electrically volume resistivity measured represents that along the printing direction of the isotropic electrically conductive adhesive. In general, isotropic electrically conductive adhesives contain fillers and, therefore, they might become anisotropic in some cases.
- b) Interfacial contact resistivity: interfacial contact resistivity is also affected by the state of treated surface of the test circuit board. This measurement method might be said to serve to measure the affinity of these properties. Furthermore, interfacial contact resistivity is influenced by the

arrangement of the conductive fillers contained in the isotropic electrically conductive adhesive. Thus, it can vary, depending on the curing of the adhesive. Take these factors into consideration in judging calculation results.

7 Test report

The test report shall contain the following items. Some items may be selected from items b) to g) upon agreement between the delivering and receiving parties:

- a) a reference to this part of ISO 16525, i.e. ISO 16525-2;
- b) the name of the isotropic electrically conductive adhesive and its data, including the kinds of resin, material of fillers, manufacturer code and lot number;
- c) the method of preparation of the test circuit board, including the method of application, curing temperature, setting time, temperature and the procedures of adhesion;
- d) the dimensions of the test circuit board, including the material and dimensions of the substrate, the pattern and dimensions of the circuit, and the number of substrates;
- e) the type of surface treatment for the electrode of the test circuit board;
- f) the date, institution and atmospheric conditions of the measurement;
- g) the calculation conditions, such as voltage and current, of volume resistivity and interfacial contact resistivity.

