



Edition 1.0 2016-02

INTERNATIONAL STANDARD

Printed electronics -

Part 202: Materials - Conductive ink





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Printed electronics –
Part 202: Materials – Conductive ink

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 31.180; 87.080 ISBN 978-2-8322-3197-5

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

PRINTED ELECTRONICS -

Part 202: Materials - Conductive ink

FOREWORD

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International Standard IEC 62899-2-1 has been prepared by IEC technical committee 119: Printed electronics.

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

FDIS	Report on voting
119/88/FDIS	119/101A/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 2.

A list of all parts in the IEC 62899 series, published under the general title *Printed electronics*, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

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- withdrawn,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

A bilingual version of this publication may be issued at a later date.

INTRODUCTION

The IEC 62899-20x series relates mainly to evaluation methods for materials of printed electronics. The series also include storage methods, packaging and marking, and transportation conditions.

The IEC 62899-20x series is divided into parts for each material. Each part is prepared as a generic specification containing fundamental information for the area of printed electronics.

The IEC 62899-20x series consists of the following parts:

Part 201: Materials - Substrates

Part 202: Materials - Conductive ink

Part 203: Materials - Semiconductor ink1

(Subsequent parts will be prepared for other materials.)

Furthermore, sectional specifications, blank detail specifications, and detail specifications of each material will follow these parts.

This part of IEC 62899 is prepared for conductive materials used in printed electronics and contains the test conditions, the evaluation methods and the storage conditions.

Convright International Electrotechnical Commission

¹ Under consideration.

PRINTED ELECTRONICS -

Part 202: Materials - Conductive ink

1 Scope

This part of IEC 62899 defines the terms and specifies the standard methods for characterisation and evaluation.

This International Standard is applicable to conductive inks and conductive layer that are made from conductive inks.

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 5-2, Photography and graphic technology Density measurements Part 2: Geometric conditions for transmittance density
- ISO 5-3, Photography and graphic technology Density measurements Part 3: Spectral conditions
- ISO 124, Latex, rubber Determination of total solids content
- ISO 291, Plastics Standard atmospheres for conditioning and testing
- ISO 304, Surface active agents Determination of surface tension by drawing up liquid films
- ISO 489:1999, Plastics Determination of refractive index
- ISO 758, Liquid chemical products for industrial use Determination of density at 20 degrees C
- ISO 1183-1, Plastics Methods for determining the density of non-cellular plastics Part 1: Immersion method, liquid pyknometer method and titration method
- ISO 2555, Plastics Resins in the liquid state or as emulsions or dispersions Determination of apparent viscosity by the Brookfield Test method
- ISO 2592, Determination of flash and fire points Cleveland open cup method
- ISO 2719, Determination of flash point Pensky-Martens closed cup method
- ISO 2811-1, Paints and varnishes Determination of density Part 1: Pyknometer method
- ISO 2811-2, Paints and varnishes Determination of density Part 2: Immersed body (plummet) method

ISO 2884-1, Paints and varnishes – Determination of viscosity using rotary viscometers – Part 1: Cone-and-plate viscometer operated at a high rate of shear

ISO 3219, Plastics – Polymers/resins in the liquid state or as emulsions or dispersions – Determination of viscosity using a rotational viscometer with defined shear rate

ISO 3451-1, Plastics – Determination of ash – Part 1: General methods

ISO 3664, Graphic technology and photography – Viewing conditions

ISO 3679, Determination of flash no-flash and flash point – Rapid equilibrium closed cup method

ISO 4576, Plastics – Polymer dispersions – Determination of sieve residue (gross particle and coagulum content)

ISO 9276-6, Representation of results of particle size analysis – Part 6: Descriptive and quantitative representation of particle shape and morphology

ISO 13319, Determination of particle size distributions – Electrical sensing zone method

ISO 13320, Particle size analysis – Laser diffraction methods

ISO 13321, Particle size analysis – Photon correlation spectroscopy

ISO 13322-1, Particle size analysis – Image analysis methods – Part 1: Static image analysis methods

ISO 13468-1:1996, Plastics – Determination of the total luminous transmittance of transparent materials – Part 1: Single beam instrument

ISO 13468-2:1999, Plastics – Determination of the total luminous transmittance of transparent materials – Part 2: Double-beam instrument

ISO 13655, Graphic technology – Spectral measurement and colorimetric computation for graphic arts images

ISO 14488, Particulate materials – Sampling and sample splitting for the determination of particulate properties

ISO 14782, Plastics – Determination of haze for transparent materials

ISO 14887, Sample preparation – Dispersing procedures for powers in liquids

ISO 20998-1, Measurement and characterization of particles by acoustic methods – Part 1: Concepts and procedures in ultrasonic attenuation spectroscopy

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

NOTE The terms in italic font are those defined in Clause 3.

3.1

conductive material

ingredient of a printing or coating material, which itself is electrically conductive or becomes electrically conductive by post treatment such as heating

3.2

conductive ink

fluid in which one or more small molecules, polymers, or particles are dissolved or dispersed, and which becomes an electrically *conductive layer* (3.3) by post treatment such as heating

3.3

conductive layer

film-like electrically conductive body made of *conductive ink* (3.2), which is printed or coated on a substrate, followed as necessary by post treatment such as heating

3.4

conductive film

substrate (sheet or roll) with conductive layer (3.3)

3.5

solid content

mass fraction of an ingredient which effectively functions as a conductive substance, in conductive ink (3.2)

3.6

non-volatile content

mass fraction of residue obtained by evaporation of the volatile solvent under specific conditions, in *conductive ink* (3.2)

3.7

ash content

mass fraction of residue excluding ingredients which are combusted or carbonised by pyrolysis, in *conductive ink* (3.2)

3.8

foreign matter

substances and/or particles of aggregated grains, filmed ink and ingredients which do not function as a part of *conductive ink* (3.2)

3.9

spherical particle

particle with three approximately equal dimensions of length, width and height

3.10

rod

substance with thin elongated cylindrical shape

3.11

wire

substance with thin long fibre-like shape having electrically conductive or semi-conductive property

3.12

tube

substance with fiber-like hollow cylindrical shape

3.13

dispersion

heterogeneous system in which fine separated materials are distributed uniformly in other materials

3.14

flash point

lowest liquid temperature at which, under certain standardized conditions, a liquid gives off vapours in quantity such as to be capable of forming an ignitable vapour/air mixture

[SOURCE: IEC 60050-212:2010, 212-18-05]

4 Atmospheric conditions for evaluation and conditioning

The standard atmosphere for evaluation (test and measurement) and storage of the specimen shall be a temperature of 23 °C \pm 2 °C and relative humidity of (50 \pm 10) %, conforming to standard atmosphere class 2 specified in ISO 291. If a polymer substrate is used for a test piece coated with a conductive layer, the standard atmosphere for evaluation shall be a temperature of 23 °C \pm 1 °C and relative humidity of (50 \pm 5) %, conforming to standard atmosphere class 1 specified in ISO 291.

If conditioning is necessary, the same standard atmosphere specified above shall apply.

5 Evaluation of properties of conductive ink

5.1 Specimen

The specimen for evaluation shall be prepared according to ISO 14488 or an equivalent method. If necessary, dilution by a compatible solvent may be allowed.

5.2 Contents

5.2.1 Solid content

5.2.1.1 Determination of solid content

Solid content of conductive materials and non-conductive materials shall be determined by the theoretical mass fraction (expressed as a percentage) of functional ingredients to the total ink mass. Functional ingredients include conductive materials, their precursors or binders, or any additives.

5.2.1.2 Report of the results

The report shall include the following items:

- a) unique specimen identification;
- b) atmospheric conditions of test;
- c) solid content;
- d) tester, test place, and test time (ex. company name, city, year).

5.2.2 Non-volatile content

5.2.2.1 Principle

Non-volatile content is determined by measuring the mass of residue after evaporation of the volatile ingredients and calculating the mass fraction (expressed as a percentage) to the total ink mass.

5.2.2.2 Test method

The test method shall be as specified in ISO 1625 with the following exceptions:

- a) Air pressure: 86 kPa to 106 kPa.
- b) If specified by the manufacturer, the test may be performed under reduced pressure. The conditions and procedures for reducing the pressure shall be as specified in ISO 124 or by the manufacturer.
- c) The materials which do not react with the ink during an examination shall be used.
- d) Repeat the test until the weight becomes constant within 5 %.

5.2.2.3 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) test conditions (air pressure if reduced, drying temperature and time);
- c) specimen mass;
- d) results;
- e) tester, test place, and test time (ex. company name, city, year).

5.2.3 Ash content

5.2.3.1 Test method

The test method shall be as specified in ISO 3451-1, method A, with the following exceptions:

- a) If appropriate for the properties of the ink, a calcination temperature other than that specified in ISO 3451-1 may be used.
- b) If the ash content is very low, considering weighing accuracy, a specimen mass other than that specified in ISO 3451-1 may be used.

The detailed product specifications shall specify the applicable calcination temperature and specimen mass.

5.2.3.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) calcination temperature;
- c) specimen mass;
- d) results and variation;
- e) tester, test place, and test time (ex. company name, city, year).

5.2.4 Foreign matter

5.2.4.1 Principle

Foreign matter is determined by measuring the mass of filtration residue and calculating the mass fraction (expressed as a percentage) to the total ink mass.

5.2.4.2 Test method

The test method shall be as specified in ISO 4576, with the following exceptions:

a) The specimen mass shall be 1 g to 10 g.

- b) For filtration of the specimen, a membrane filter with appropriate pore size shall be used instead of a metal sieve. The pore size of the filter shall be as specified by the manufacturer.
- c) For diluting the specimen or cleaning the filtration residue, a solvent appropriate for the properties of the ink shall be used.
- d) Prior to filtering, the specimen may be diluted by an appropriate solvent if necessary. No other treatment shall be allowed.
- e) The drying temperature for the filter and filtration residue may be other than as specified in ISO 4576.
- f) The test shall be conducted until the difference between the results of two consecutive tests is not more than 20 %; the higher result shall be used for the report.

5.2.4.3 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) test conditions (filter pore size and drying temperature);
- c) results;
- d) tester, test place, and test time (ex. company name, city, year).

5.3 Physical properties

5.3.1 Density

5.3.1.1 Measuring method

The measuring method shall either be the pyknometer method as specified in ISO 758, ISO 1183-1 and ISO 2811-1, the method using oscillation-type density meters as specified in ISO 15212-1, or the immersed body (plummet) method as specified in ISO 2811-2. The detailed product specifications shall specify the measuring method to be used.

5.3.1.2 Equipment

Equipment shall be as specified in the measurement method (see 5.3.1.1) or shall be equipment considered equivalent or superior.

5.3.1.3 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) measurement method;
- c) measurement atmosphere (temperature and relative humidity);
- d) results;
- e) tester, test place, and test time (ex. company name, city, year).

5.3.2 Rheology

5.3.2.1 Measuring method

Viscosity shall be measured using a Brookfield type rotational viscometer as specified in ISO 2555, cone-and-plate viscometer as specified in ISO 2884-1, or rotational viscometer as specified in ISO 3219.

The detailed product specifications shall specify the measuring method and measuring temperature to be used.

5.3.2.2 Report of the results

The report shall include the following items:

- a) standard number of the measurement method;
- b) specimen identification;
- c) measuring temperature;
- d) viscometer model;
- e) viscosity expressed in millipascal second (mPa·s);
- f) tester, test place, and test time (ex. company name, city, year).

5.3.3 Surface tension

5.3.3.1 Measuring method

Surface tension shall be measured using the drawing up liquid film (Wilhelmy) method as specified in ISO 304 with the following exceptions:

- a) equipment considered equivalent to that in ISO 304 may be used;
- b) the test jig shall be made of platinum;
- c) the equipment shall be calibrated using pure water and a hanging weight.

5.3.3.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) measuring temperature;
- c) surface tension expressed in millinewton per metre (mN/m);
- d) tester, test place, and test time (ex. company name, city, year).

5.3.4 Size of conductive materials

5.3.4.1 Sampling

The specimen used for measuring the size of a conductive material shall be sampled according to the method specified in ISO 14488 or a similar method. If re-dispersion is required, perform re-dispersion according to the method specified in ISO 14887 or a similar method.

5.3.4.2 Sphere

5.3.4.2.1 Measuring method

The mean particle size of spherical particles or near spherical particles (such as equants) shall be measured using the electric sensing zone method specified in ISO 13319, the laser diffraction method specified in ISO 13320, the photon correlation method specified in ISO 13321, or the ultrasonic attenuation spectroscopy method specified in ISO 20998-1.

Measuring equipment shall be as specified in one of the above standards. Measuring equipment which uses a method specified in any of these standards may be used.

Mean particle size is obtained as the sphere-equivalent diameter or circle-equivalent diameter, depending on the measurement method. The type of diameter shall be recorded and included in the report of the results.

NOTE Equants express the particles of similar length, width, and thickness. Both cubical and spherical particles are included.

5.3.4.2.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) mean particle size;
- c) type of diameter;
- d) tester, test place, and test time (ex. company name, city, year).

5.3.4.3 Rod, wire and tube

5.3.4.3.1 Measuring method

Rods, wires and tubes shall be measured using the method specified in ISO 13322-1, or an equivalent method. As this method measures the widths and lengths of particles from an electron microscope image, it requires a standard length. The scale shown on the image may be used as the standard length. In order to decide the standard length, particles having known accurate size may be mixed in with the specimen as a reference and photographed with the specimen to calibrate the scale in the photograph. The smallest number of particles required for obtaining the mean width and length is determined according to ISO 13322-1.

ISO 9276-6 treats rods, wires and tubes as acicular or columnar particles and specifies a method for measuring their lengths. The width and length may be obtained using this method and software based on it.

NOTE 1 The acicular particle is a slender, needle-like particle of similar width and thickness.

NOTE 2 The columnar particle is a long, thin particle with the width and thickness that are greater than those of an acicular particle.

5.3.4.3.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) width and length of particles;
- c) definition of the standard of length;
- d) number of particles used for calculating width and length;
- e) tester, test place, and test time (ex. company name, city, year).

5.3.4.4 Other shapes

5.3.4.4.1 Measuring method

For irregularly shaped particles which are not classified in 5.3.4.2 and 5.3.4.3 above, the mean particle size shall be calculated according to the method specified in ISO 9276-6 or software based on it.

5.3.4.4.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) mean particle size;
- c) items whose defaults have been changed during data processing;
- d) tester, test place, and test time (ex. company name, city, year).

5.3.5 Flashpoint

5.3.5.1 Measuring method

Flashpoint shall be measured according to ISO 2592 (open system). The method of "open system" is preferable for safety, however, "closed systems" are also widely used. The measurement method based on ISO 2719 (closed system) and ISO 3679 (closed system) may be applied if a closed system is required.

5.3.5.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) test conditions (temperature, humidity and atmospheric pressure);
- c) sampling conditions (type of ink used, dispersive media and concentration);
- d) results;
- e) tester, test place, and test time (ex. company name, city, year).
- f) others (special items).

5.3.6 Evaporation rate

5.3.6.1 **General**

The evaporation rate is a property which is necessary for the PE ink, but the details of the evaluation condition and the measuring method are significantly different for the ink. In this standard, a common framework of the method is specified as a guideline. The detailed conditions and measurements may be determined between trading partners depending on the properties of the ink.

5.3.6.2 Measuring method

The evaporation rate of solvent from an ink formulation can be determined by measuring the time taken to evaporate up to 90 % of the mass of the solvent content of the ink formulation.

A flat absorbent material (such as a filter paper) is positioned on a sensitive mass balance located inside a dry air (< 5 % relative humidity) or nitrogen cabinet at atmospheric pressure. A known volume of ink (such as 1 mL) is dispensed in a straight line on the absorbent material to produce a repeatable area of wetted film on the filter paper. Dried air or nitrogen is passed through the cabinet at a controlled temperature and flow rate. The temperature and flow rate may be determined between trading partners depending on the properties of the ink, but these conditions shall be included in the report. The location of the entry and exit ports for the dry air or nitrogen should be chosen so as not to disturb the mass balance readings throughout the test.

The evaporation rate can be calculated by measuring the difference in mass over a period of time. It is recommended that sufficient measurements are made to allow 5 or more points to be plotted on a graph of mass loss vs. time for values of between 10 % and 90 % loss of solvent from the ink. The test should be repeated a total of 3 times and the evaporation rates averaged for that ink. In order to make a comparison, the procedure should be conducted with a known solvent such as n-butyl acetate and the evaporation rate normalised to this solvent.

In the case of an ink comprising a solvent mixture, an increased number of measurements shall be made in order to clearly show how the evaporation rate changes over time. It is recommended that a balance with automated data logging be used in order to facilitate the capture of sufficient data to describe the detailed behaviour. The results may be presented in graphical form for the case where the solvent evaporation rate varies with time in a complex manner. These results can also be compared with those for n-butyl acetate by plotting both data on the same graph.

5.3.6.3 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) test conditions (mass of ink, flow rate of air/nitrogen, solvent used for comparison);
- c) results (normalised to the solvent used for comparison);
- d) tester, test place, and test time (ex. company name, city, year).

6 Evaluation of the properties of a conductive layer

6.1 Test piece

6.1.1 General

Test pieces are used for evaluating the conductive layer.

6.1.2 Substrate

The substrate for the test piece shall be a clean and smooth-surface non-alkali glass which will not affect the ink. Other substrate materials may be used if agreed between the trading partners (supplier and purchaser).

6.1.3 Conductive ink

According to 5.1, except no dilution is allowed.

6.1.4 Dimensions of test piece

The dimensions of the test piece shall be as specified in each test method. If evaluation is possible, a test piece with smaller and/or thinner dimensions than specified may be used.

6.1.5 Preparation of test piece

The test piece shall be prepared according to the following procedure:

- a) Prior to ink printing or coating, the substrate surface shall be cleaned by an appropriate means using an organic solvent such as acetone.
- b) Print or coat the ink onto the substrate surface using an appropriate method to form a uniform layer of ink.
- c) Solidify the ink by an appropriate means to produce an electrically conductive layer.

6.2 Electrical properties

6.2.1 Volume resistivity

6.2.1.1 **General**

The volume resistivity of a conductive layer made of conductive ink is measured using the four-point probe method. This method, as shown in Figure 1, arranges four electrodes linearly on the test piece, passes current I between two electrodes such as probe A and probe D, measures the potential difference V between the other two electrode, and calculates resistance V/I.

The four-point probe head which does not damage the conductive layer should be used.

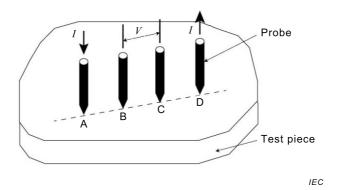


Figure 1 – Example of four-point probe measurement

6.2.1.2 Equipment and tools

6.2.1.2.1 Test equipment

6.2.1.2.1.1 Test equipment constitution

The test equipment consists of a constant-current source, a voltmeter and probes, as shown in Figure 2.

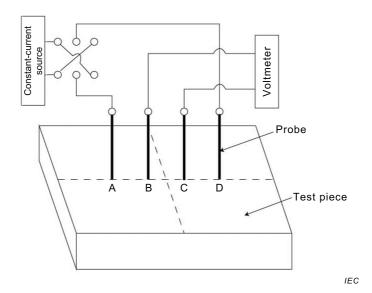


Figure 2 - Example of four-probe measurement equipment

6.2.1.2.1.2 Constant-current source

The constant-current source shall be able to pass an applied current I_0 as listed in Table 1 through the resistance range of the test piece. The applied current is a direct current passing from the constant-current source to the probe. Applied current I_0 is determined as follows. Assuming potential difference V between probe B and probe C, the resistance of the resistor is given by V/I_0 . Select the current from Table 1 corresponding to the resistor, which is applied current I_0 . The allowable variation ratio of applied current I_0 during measurement shall be ± 0.05 % or less.

Table 1 – Resistance range of the test piece and the applied current

Resistance range of the test piece	$<$ 20 m Ω	$<$ 200 m Ω	< 20 Ω	< 2 kΩ	< 20 kΩ	< 200 kΩ	< 2 MΩ
Applied current (I_0)	1 000 mA	100 mA	10 mA	1 mA	100 μΑ	10 μΑ	1 μΑ

6.2.1.2.1.3 Voltmeter

The voltmeter shall be a digital type with a display having at least three digits. As it is required to measure the entire range of resistivity shown in Table 1, the voltmeter shall preferably be able to measure voltages ranging from 0,1 mV to 2 V and an input resistance of $10^{10}~\Omega$ or higher.

6.2.1.2.1.4 Probe

Each probe shall be a metal pin with a diameter of 0,5 mm to 0,8 mm with a semi-spherical or flat tip. The four probes are arranged linearly at equal spacing with semi-spherical or flat tips touching the test piece. The spacing shall be 1,5 mm and the insulation resistance between adjacent probes shall be $10^{10}~\Omega$ or higher. The spacing may be 0,5 mm, 1,0 mm, 2,2 mm, or 5,0 mm. When the spacing of 0,5 mm is used, the probe shall be calibrated. The report shall include that the calibrated probe with the spacing of 0,5 mm has been used. During measurement, an appropriate force shall be applied to each probe. Examples of the probes which can apply for the appropriate force are shown in Annex A.

If a 0,5 mm spacing probe is used it is recommended that a calibrated specimen having known resistivity is measured and that its measured value is within ± 3 % of the known value.

6.2.1.2.2 Measuring equipment for specimen thickness

Measuring equipment for specimen thickness shall be a non-contact type or probe type.

6.2.1.3 Evaluating the test piece

6.2.1.3.1 Preparing the specimen

The specimen shall be prepared according to the procedure specified in 6.1, however, the substrate shall have the following properties:

- resistance $> 10^6 \Omega$.
- no dent and no distortion excluding the dents from the probe test itself.

If a substrate is used that is subject to distortion then the specimen shall be fixed on a flat plane during the measurement.

6.2.1.3.2 Dimensions of the specimen

The dimensions of the specimen shall be as follows:

a) the size of the specimen shall be selected from Table 2;

Table 2 – List of the size of the specimen

	Length	Width
	(mm)	(mm)
Type A	$30\pm0,2$	$10\pm0,1$
Type B	$30\pm0,2$	$30\pm0,2$
Type C	$80\pm0,2$	$50\pm0,2$

b) the thickness of the specimen shall be 200 μm or less and measured at the same positions where the resistance is measured. The thickness includes only the conductive layer, and the substrate is not included.

6.2.1.4 Test procedure

6.2.1.4.1 Resistance measurement

The measuring positions of resistance shall be A1 to A3 in Figure 3, B1 to B5 in Figure 4, or C1 to C9 in Figure 5.

Dimensions in millimetres

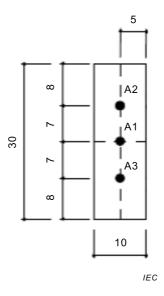


Figure 3 – Measuring positions of resistance (Type A)

Dimensions in millimetres

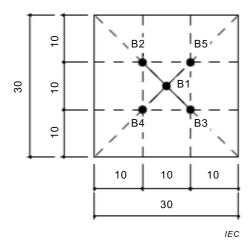


Figure 4 – Measuring positions of resistance (Type B)

Dimensions in millimetres

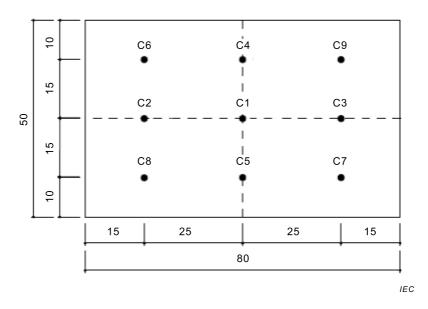


Figure 5 – Measuring positions of resistance (Type C)

Resistance shall be measured at three points: A1 to A3 for Type A, B1 to B3 for Type B, or C1 to C3 for Type C. However, in consideration of the uniformity of the film and the past test results, measuring only one point (A1, B1, or C1), five points (B1 to B5 for Type B, or C1 to C5 for Type C) or nine points (C1 to C9 for type C) is acceptable. The resistance shall be measured by placing the centre of the probe on each measuring position. The probe shall be placed parallel to the long side of the specimen. For the stable measurement, it is necessary that the measuring positions are more than 10 mm inside the edge of the specimen. Therefore, when the resistance of the Type A is measured, a template may be used in order to keep the same measuring position.

6.2.1.4.2 Calculating resistivity

Resistivity shall be calculated by using the following equation:

$$\rho = F \cdot t \cdot R \tag{1}$$

where:

 ρ is the resistivity ($\Omega \cdot \text{cm}$);

F is the correction factor;

t is the conductive layer thickness (cm);

R is the measured resistance value (Ω).

The correction factor F shall be used for the value which is calculated by Poisson's equation (2). The same value as the solution of Poisson's equation may be used in the correction factor. (The equation of the correction factor is described in Annex B. The influence of the measuring position and the size of the specimen are described in Annex C.)

$$F^{-1} = \frac{y_{\rm B} - y_{\rm c}}{a} + \sum_{m=1}^{\infty} \frac{2}{a\xi \sinh(b\xi)} \left[\left\{ \cos(\xi x_{\rm B}) \cosh\left[\xi\left(y_{\rm B} + \frac{b}{2}\right)\right] - \cos(\xi x_{\rm C}) \cosh\left[\xi\left(y_{\rm C} + \frac{b}{2}\right)\right] \right\} \times \cos(\xi x_{\rm A}) \cosh\left[\xi\left(y_{\rm A} - \frac{b}{2}\right)\right] - \left\{ \cos(\xi x_{\rm B}) \cosh\left[\xi\left(y_{\rm B} - \frac{b}{2}\right)\right] - \cos(\xi x_{\rm C}) \cosh\left[\xi\left(y_{\rm C} - \frac{b}{2}\right)\right] \right\} \times \cos(\xi x_{\rm D}) \cosh\left[\xi\left(y_{\rm D} + \frac{b}{2}\right)\right] \right]$$

$$+\sum_{n=1}^{\infty} \frac{2}{a\eta \sinh(b\eta)} \times \left[\left\{ \cosh \left[\eta \left(y_{\mathsf{B}} + \frac{b}{2} \right) \right] - \cosh \left[\eta \left(y_{\mathsf{C}} + \frac{b}{2} \right) \right] \right\} \times \cosh \left[\eta \left(y_{\mathsf{A}} - \frac{b}{2} \right) \right]$$

$$- \left\{ \cosh \left[\eta \left(y_{\mathsf{B}} - \frac{b}{2} \right) \right] - \cosh \left[\eta \left(y_{\mathsf{C}} - \frac{b}{2} \right) \right] \right\} \times \cosh \left[\eta \left(y_{\mathsf{D}} + \frac{b}{2} \right) \right] \right]$$

$$+ \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \frac{4}{a\zeta \sinh(b\zeta)} \times \left[\left\{ \cos(\xi x_{\mathsf{B}}) \cosh \left[\zeta \left(y_{\mathsf{B}} + \frac{b}{2} \right) \right] - \cos(\xi x_{\mathsf{C}}) \cosh \left[\zeta \left(y_{\mathsf{C}} + \frac{b}{2} \right) \right] \right\} \times \cos(\xi x_{\mathsf{A}}) \cosh \left[\zeta \left(y_{\mathsf{A}} - \frac{b}{2} \right) \right]$$

$$- \left\{ \cos(\xi x_{\mathsf{B}}) \cosh \left[\zeta \left(y_{\mathsf{B}} - \frac{b}{2} \right) \right] - \cos(\xi x_{\mathsf{C}}) \cosh \left[\zeta \left(y_{\mathsf{C}} - \frac{b}{2} \right) \right] \right\} \times \cos(\xi x_{\mathsf{D}}) \cosh \left[\zeta \left(y_{\mathsf{D}} + \frac{b}{2} \right) \right] \right]$$

$$(2)$$

where:

 (x_A, y_A) are the x, y coordinates of the probe A (cm);

 (x_{B}, y_{B}) are the x, y coordinates of the probe B (cm);

 (x_{C}, y_{C}) are the x, y coordinates of the probe C (cm);

 (x_D, y_D) are the x, y coordinates of the probe D (cm);

a, *b* are the length of the specimen;

 $\xi = m\pi/a$ (*m* represents an integral number), $\eta = n\pi/t$ (*n* represents an integral number, *t* represents the thickness of the conductive layer), $\zeta = (\xi^2 + \eta^2)^{1/2}$).

When obtaining changes in volume resistivity after humidification, the volume resistivity of the test piece shall be measured before and after humidification at a temperature of 60 °C \pm 2 °C and relative humidity of (90 \pm 3) % for 24 h, 48 h, or 72 h, the values compared, and the difference calculated. If agreed between the trading partners (supplier and purchaser), other humidification conditions may be used.

6.2.1.5 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) test conditions (air pressure and drying temperature);
- c) test piece size;
- d) volume resistivity and error;
- e) tester, test place, and test time (ex. company name, city, year).

6.2.2 Surface resistivity

6.2.2.1 Determination of surface resistivity

Surface resistivity shall be obtained by dividing the volume resistivity measured in 6.2.1 by the thickness of the conductive layer. If thickness is measured at several points, the average shall be used. When obtaining changes in surface resistivity after humidification, the volume resistivity before and after humidification, measured in 6.2.1.4, shall be divided by the thickness of the conductive layer and the difference shall be calculated.

6.2.2.2 Report of the results

The report shall include the following items:

a) specimen identification;

- b) test conditions (air pressure and drying temperature);
- c) test piece size;
- d) surface resistivity;
- e) tester, test place, and test time (ex. company name, city, year).

6.3 Optical properties

6.3.1 Overview

The tests specified in 6.3.2 through 6.3.5 shall be used for transparent or equivalent materials.

6.3.2 Luminous transmittance

6.3.2.1 General

Luminous transmittance is presented as total luminous transmittance.

6.3.2.2 Measuring method

Luminous transmittance shall be measured using the single-beam method as specified in ISO 13468-1, or the double-beam method as specified in ISO 13468-2, with the following details. If agreed between the trading partners (supplier and purchaser), another method which is considered equivalent may be used.

The detailed product specifications shall specify the applicable measuring method.

6.3.2.3 Measuring equipment

Measuring equipment shall be as specified in ISO 13468-1:1996, Clause 4, or ISO 13468-2:1999, Clause 4, as appropriate. Measuring equipment according to ISO 13655 and/or ISO 5-2 may be used.

6.3.2.4 Wavelength or wavelength range used in the test

Luminous transmittance shall be measured either at a particular wavelength or a wavelength range, as agreed between the trading partners (supplier and purchaser) considering factors such as material characteristics and/or application.

6.3.2.5 Report of the results

The report shall include the following items:

- a) measuring method and equipment;
- b) measuring wavelength or wavelength range;
- c) specimen thickness;
- d) luminous transmittance;
- e) tester, test place, and test time (ex. company name, city, year).

6.3.3 Chromaticity

6.3.3.1 General

According to ISO 11664-4, chromaticity is presented as the CIE (1976) $L^*a^*b^*$ colour space.

6.3.3.2 Measuring method

The measuring method shall be the reflected light method or the transmitted light method, depending on the application and the purpose.

If the reflected light method is used, a reflecting diffuser shall be placed on both the surface to be measured and the other surface, with the specimen in between.

The reflecting diffuser shall be a perfect reflecting diffuser or a reference diffuser used for calibrating measuring equipment.

6.3.3.3 Measuring equipment and auxiliaries

The measuring equipment and light source shall be in accordance with at least one of the following: ISO 5-2, ISO 5-3, ISO 3664 or ISO 13655, and shall be specified in the detailed product specifications.

6.3.3.4 Expression of the results

The results shall be presented as the numerical values of each of the $L^*a^*b^*$ coordinate axes, or shall be plotted in the $L^*a^*b^*$ colour space. If agreed between the trading partners (supplier and purchaser), the results may be presented instead by the numerical value of a specific coordinate axis or the numerical values of two specific coordinate axes of the $L^*a^*b^*$ colour space. In this case, the coordinate axis or axes concerned shall be clearly stated.

6.3.3.5 Report of the results

The report shall include the following items:

- a) measuring instrument and light source;
- b) measurement method (reflected light or transmitted light);
- c) chromaticity (numerical values of each of the $L^*a^*b^*$ coordinate axes, plotted in the $L^*a^*b^*$ colour space, or the numerical value of a specific coordinate axis or numerical values of two specific coordinate axes of the $L^*a^*b^*$ colour space);
- d) tester, test place, and test time (ex. company name, city, year).

6.3.4 Uniformity of colour

6.3.4.1 Principle

Colour differences are obtained on 10 points on the specimen, and their average is evaluated by the difference from the standard chromaticity and standard deviation.

6.3.4.2 Measuring equipment

According to 6.3.3.3.

6.3.4.3 Illuminant (light source)

According to 6.3.3.3.

6.3.4.4 Measuring method

According to 6.3.3, chromaticity shall be measured on 10 points on a single specimen using the same instrument and under the same conditions. Five or more pairs of points are selected so that all 10 points are chosen. From the colour difference between each pair of points, the mean colour difference, the difference between the reference chromaticity and the mean, and the standard deviation are calculated.

6.3.4.5 Calculation of colour difference

Colour difference is calculated by using the colour difference formula based on the numerical values of each of the $L^*a^*b^*$ coordinate axes, or using the colour difference formula based on lightness, chroma, and hue.

The colour difference based on the $L^*a^*b^*$ colour system is calculated by using the formula:

$$\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$
(3)

where:

 ΔE^*_{ab} is the colour difference based on the $L^*a^*b^*$ colour system;

 ΔL^* , Δa^* , Δb^* is the difference in L^* and difference in colour coordinates a^* and b^* of the chromaticity between the pair of points obtained in 6.3.3.

The colour difference based on the luminosity, chroma, and hue is calculated by using the formula:

$$\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta C^*_{ab})^2 + (\Delta H^*_{ab})^2]^{1/2}$$
(4)

where:

 ΔE^*_{ab} is the colour difference based on the $L^*a^*b^*$ colour system;

 ΔL^* is the difference in luminosity L^* of the chromaticity between the pair of points obtained in 6.3.3:

 ΔC^*_{ab} is the difference in ab chroma of the chromaticity between the pair of points obtained in 6.3.3;

 ΔH^*_{ab} is the difference in ab hue of the chromaticity between the pair of points obtained in 6.3.3.

The colour difference formula in CIE DE2000 (see CIE Publication No. 142) may also be used for calculating the colour difference.

6.3.4.6 Report of the results

The report shall include the following items:

- a) measuring instruments;
- b) measurement method (reflected light or transmitted light);
- c) standard chromaticity;
- d) difference between the standard chromaticity and the mean, and standard deviation;
- e) tester, test place, and test time (ex. company name, city, year).

6.3.5 Haze

6.3.5.1 Measuring method

Haze shall be measured using the method specified in ISO 14782. A similar method may be used as agreed between the trading partners (supplier and purchaser).

6.3.5.2 Report of the results

The report shall include the following items:

- a) thickness of the specimen;
- b) type of light source;
- c) haze;
- d) tester, test place, and test time (ex. company name, city, year).

6.3.6 Refractive index

6.3.6.1 Measuring method

The refractive index shall be measured using method A (for measuring the refractive index of films using a refractometer) specified in ISO 489 or a similar method.

6.3.6.2 Contacting liquid

A contacting liquid with a refractive index higher than that of the measured object and with a substrate which does not swell or dissolve as listed in ISO 489:1999, Table 1, shall be used. When using a plastic film for the substrate, choose the contact liquid in consideration of the refractive index, swelling and melting of the substrate.

6.3.6.3 Conditioning

Conditioning shall be performed at a temperature of 23 °C \pm 2 °C and a relative humidity of (50 \pm 10) % for 88 h or longer. Other conditioning shall be determined by the trading partners (supplier and purchaser), if necessary.

6.3.6.4 Report of the results

The report shall include the following items:

- a) refractometer used, and the type of the light source and wavelength;
- b) refractive index;
- c) dispersion (if applicable);
- d) tester, test place, and test time (ex. company name, city, year).

7 Storage

7.1 Storage conditions

Conductive ink shall be stored in a sealed container, at a temperature of 6 $^{\circ}\text{C} \pm 4$ $^{\circ}\text{C}$ unless otherwise specified.

7.2 Method for measuring aged deterioration

The degradation due to storage is expressed by the change in viscosity. The viscosity just before storage and the viscosity just after storage for 30 days under the conditions in 7.1 shall be measured according to the method specified in 5.3.2.

7.3 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) method for measuring viscosity;
- c) viscosity just before storage (mPa·s);
- d) viscosity after storage for one month (mPa·s);
- e) tester, test place, and test time (ex. company name, city, year).

Annex A (informative)

Examples of the four-point probe for applying an appropriate weight

A.1 Internal structure

Four-point probes have internal springs to keep appropriate contact. An example of the internal structure around the probe pin is shown in Figure A.1. Resistance is measured by pushing the probe in the direction of the arrow. A spring is installed in the root of the probe pin, and the force applied to the pin is controlled appropriately.

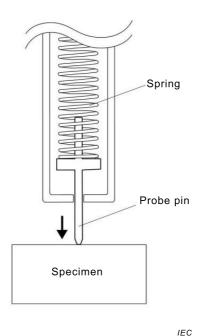
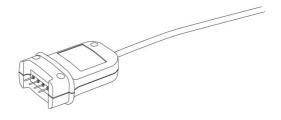


Figure A.1 – Example of the internal structure of probe

A.2 Example of the general view

An example of the general view of the probe is shown in Figure A.2. The four-probe pins like the one shown in Figure A.1 are installed at the tip of the probe. When the probe is used for measurement, the housing portion is held in the hand or fixed to the X-Y stage. Although the rectangular type probe is shown in the figure as the example, a columnar type is also available. These types can be used depending on the spacing of the probe pins and/or the constitution of the measurement system.



IEC

Figure A.2 – Example of the general view of the probe

Annex B (informative)

Equation of correction factor F

B.1 General

The resistivity ρ of the conductive layer is defined by the following equation (B.1):

$$\rho = Ft \frac{V}{I} \tag{B.1}$$

where:

t is the thickness of the conductive layer (cm);

F is the resistivity correction factor.

The configuration of probes A, B, C, and D is shown in Figure B.1. The four probes are placed on the surface of the rectangular parallelepiped of width a, length b, with thickness t.

With the potentials of the probe B and the probe C expressed as $\phi_{\rm B}$ and $\phi_{\rm C}$ respectively, the potential difference V is represented as the following equation:

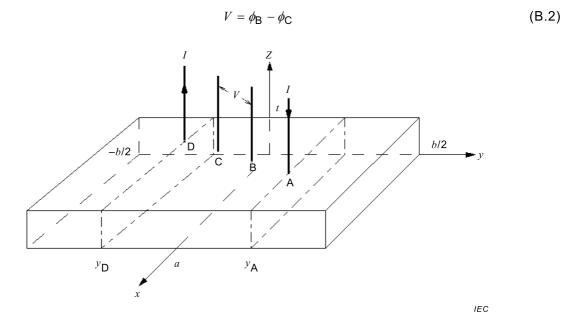


Figure B.1 – Schematic diagram of the geometry of the conductive layer and the configuration of probes A, B, C, and D

B.2 Conditions for correction factor F

With the potential ϕ of any point in the sample satisfying Poisson's equation, the following Equation B.3 is established:

$$\nabla^2 \phi(r) = 2\rho I \left[\delta(r - r_{\mathsf{D}}) - \delta(r - r_{\mathsf{A}}) \right] \tag{B.3}$$

where:

vector r is the coordinates (x, y, z) of any point in the conductive layer;

is the coordinates (x_A, y_A, t) of the contact point of the probe A and the conductive layer;

 $r_{\rm D}$ is the coordinates $(x_{\rm D}, y_{\rm D}, t)$ of the contact point of the probe D and the conductive layer:

 $\delta(r)$ is the delta function (the function is given by the product of $\delta(x)$, $\delta(y)$, and $\delta(z)$).

In the process of leading the equation, the function $q = \varepsilon^* \rho I/2\pi$ is used to replace current I by the equivalent charge q. (ε^* represents the dielectric constant of the conductive layer.)

When the conductive layer is divided into three areas by the planes parallel to the *x*-axis through the positions of the probe A and the probe D, Equation B.3 equals 0 due to the nature of the delta function, and Laplace's equation is obtained.

By applying the following three conditions in order to obtain the solution of Laplace's equation, it is possible to determine the potentials of each region:

- Current does not flow out to the outside of the conductive layer.
- The potential is always continuous at the boundary of each region.
- Gauss's law can be applied to the field of the differential volume in the centre of the contact of the probe and the conductive layer.

B.3 Equation of correction factor F

From these conditions, the correction factor F can be expressed as F^{-1} using equation (2) in 6.2.1.4.2.

Annex C (informative)

Influence of the measuring position and the size of specimen on resistance

C.1 Influence of the measuring position on resistance

The result of resistance is greatly affected by the measuring position. As shown in Figure C.1, when the correction factor is fixed at a constant value, it is impossible to obtain appropriate data because the resistance increases significantly at the end of the specimen scan in order to increase the density of the electric field energy. Therefore, it is necessary to perform a proper measurement of resistance by using the variable correction factor given by Poisson's equation in accordance with the accurate measuring position.

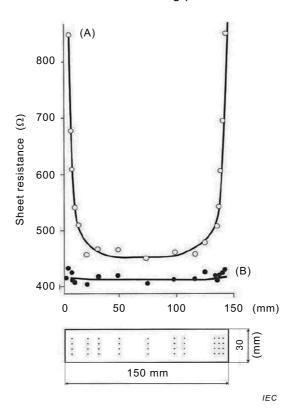


Figure C.1 – Measurement model for the influence of the measuring position on sheet resistance

Data (A) shows the measurement for the correction factor fixed at 4,532. Data (B) shows the data which is corrected properly by using the correction factor given by Poisson's equation. The rectangle under the figure expresses the image of the measured specimen. The black points express the position of the probe.

NOTE Although " Ω /square" may be familiar as the unit of sheet resistance, " Ω " is applied as the SI unit in Figure C.1.

C.2 Influence of the size of the specimen on resistance

The result of resistance is also greatly affected by the size of the specimen. For small specimens, the results become more sensitive to electric field energy in the same manner described in Clause C.1 because the distance from the edge of the specimen to the

measuring point is closer. At the 20 mm width point of the specimen (the distance to the end of the specimen from the measurement point is 10 mm), the same resistance can be obtained by the correction of Poisson's equation (Figure C.2). However, if the specimen width becomes less than 20 mm, the resistance begins to rise. When the specimen width is 10 mm measurements are difficult and proper correction cannot be made; a large error is predicted. The determination of the accurate measuring position and the accurate correction by Poisson's equation is required in order to obtain the stable and proper resistance.

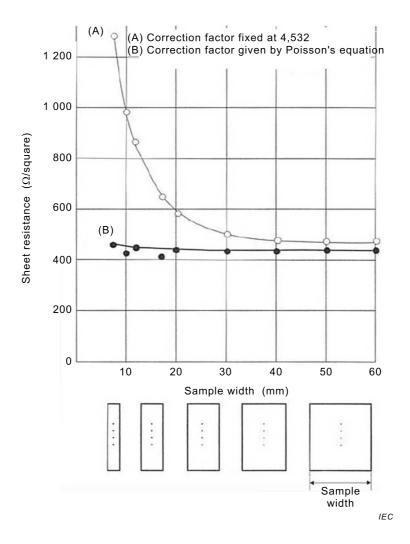


Figure C.2 - Model measurement of the influence of the specimen size

Data (A) shows the measurement for correction factor fixed at 4,532. Data (B) shows the data which is corrected properly by using the correction factor given by Poisson's equation. The rectangle under the figure expresses the image of the measured specimen. The black points express the position of the probe.

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