

INTERNATIONAL STANDARD



Printed electronics –
Part 202: Materials – Conductive ink

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Part 202: Materials – Conductive ink**

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

PRINTED ELECTRONICS –

Part 202: Materials – Conductive ink

FOREWORD

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This redline version of the official IEC Standard allows the user to identify the changes made to the previous edition IEC 62899-2-1:2016. A vertical bar appears in the margin wherever a change has been made. Additions are in green text, deletions are in strikethrough red text.

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

This second edition cancels and replaces the first edition published in 2016. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

- a) definitions of conductive material, conductive ink and conductive layer have been revised;
- b) a summary of test methods is added;
- c) mechanical tests for conductive layer are added.

The text of this International Standard is based on the following documents:

Draft	Report on voting
119/423/FDIS	119/428/RVD

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

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

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 document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

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- withdrawn,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

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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.~~

Printed electronics is a technology that spans the printing and electrical/electronic area, and it provides a variety of products. Since participants in this industry come from different areas, their backgrounds and customs can be barriers to smooth communication and transactions in the supply chain. The printed electronics industry continues to grow, but many barriers still remain. Particularly, the lack of standardised terms and evaluation methods is one of the major factors that inhibit smooth communication.

This document focuses on measurement and evaluation methods for conductive inks and provides tools to promote the smooth communication within the supply chain.

This document specifies the basic items to be communicated and their measurement or evaluation methods. This document includes the measurement methods for the basic properties of inks and electrical conductivity, which is obtained by the post treatment of inks. Additionally, storage methods, packaging and marking, and transportation conditions are also included.

This document is part of the IEC 62899-202 series and similar documents are available for other materials used in printed electronics.

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

IEC 62899-201: Materials – Substrates

IEC 62899-202: Materials – Conductive ink

IEC 62899-203: Materials – Semiconductor ink⁴

IEC 62899-204: Materials – Insulator ink

~~(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.~~

⁴ ~~Under consideration.~~

PRINTED ELECTRONICS –

Part 202: Materials – Conductive ink

1 Scope

This part of IEC 62899 defines the terms and specifies the standard test methods for characterization and evaluation of conductive inks.

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

This document also provides measurement methods for evaluating the properties of conductive layers made both from an additive process using conductive inks and from a subtractive process used in printed electronics.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 62899-202-3, *Printed electronics – Part 202-3: Materials – Conductive ink – Measurement of sheet resistance of conductive films – Contactless method*

IEC 62899-202-5, *Printed electronics – Part 202-5: Materials – Conductive ink – Mechanical bending test of a printed conductive layer on an insulating substrate*

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 489:1999/2022, *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 pycnometer method and titration method*

ISO 2409:2020, *Paints and varnishes – Cross-cut test*

ISO 2471, *Paper and board – Determination of opacity (paper backing) – Diffuse reflectance method*

ISO 2555, *Plastics – Resins in the liquid state or as emulsions or dispersions – Determination of apparent viscosity ~~by the Brookfield Test~~ using a single cylinder type rotational viscometer method*

ISO 2592, *Petroleum and related products – 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: Pycnometer 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 3251, *Paints, varnishes and plastics – Determination of non-volatile-matter content*

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 11664-4, *Colorimetry – Part 4: CIE 1976 L*a*b* colour space*

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 13468-1:19962019, *Plastics – Determination of the total luminous transmittance of transparent materials – Part 1: Single beam instrument*

ISO 13468-2:19992021, *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 powders in liquids*

ISO 15212-1, *Oscillation-type density meters – Part 1: Laboratory instruments*

ISO 18947-1:2021, *Imaging materials and prints – Abrasion resistance – Part 1: General rub testing methods*

ISO 20379, *Fine ceramics (advanced ceramics, advanced technical ceramics) – Measurement of thixotropic behaviour of ceramic slurry by use of a rotational viscometer*

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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

NOTE Words in italics are defined within 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~~ component with intrinsic property providing electrical conductivity

Note 1 to entry: The ingredient can be one or more small molecules, precursors, polymers, or particles.

Note 2 to entry: The ingredient can require post treatment to provide electrical conductivity

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~~

fluid in which one or more *conductive materials* (3.1) are dissolved or dispersed, and which is used to form an electrically conductive structure

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~~

film-like structure formed by printing or coating and *post treatment* (3.4) of *conductive ink* (3.2) on a substrate, which is electrically conductive

3.4

post treatment

process step following the deposition of ink to generate the intended functionality

Note 1 to entry: Process steps can be evaporation, annealing, curing or sintering

3.5

conductive film

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

3.6

solid content

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

3.7

non-volatile content

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

3.8

ash content

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

3.9

foreign matter

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

3.10

spherical particle

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

3.11

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

wire

flexible cylindrical conductor, with or without an insulating covering, the length of which is large with respect to its cross-sectional dimensions

Note 1 to entry: The cross-section of a wire may have any shape, but the term "wire" is not generally used for ribbons or tapes.

[SOURCE: IEC 60050-151:2001, 151-12-28]

3.13

tube

substance with fiber-like hollow cylindrical shape

3.14 dispersion

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

system consisting of two or more phases one of which is continuous and at least one other is finely dispersed

[SOURCE: IEC 62899-101:2019, 3.31]

3.15 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 pre-conditioning

The standard atmosphere for evaluation (test and measurement) and storage of the specimen shall be a temperature of $23\text{ °C} \pm 2\text{ °C}$ 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 $23\text{ °C} \pm 1\text{ °C}$ and relative humidity of $(50 \pm 5)\%$, conforming to standard atmosphere class 1 specified in ISO 291. Atmospheric pressure in test and measurement may be specified in a prior agreement of trade partners, but it shall be reported.

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

5 Summary characteristics and evaluation method of conductive ink

The conductive inks used in printed electronics shall be tested in accordance with the methods specified in Table 1. Unless there is a prior agreement between the user and supplier these test methods shall be applied without modification. In cases where the test has been modified, the changed condition shall be described in the report.

Table 1 – Test methods for conductive inks used in PE

	Items	Standards for each test method
Ink properties	Non-volatile content	ISO 3251
	Ash content	ISO 3451-1 method A
	Foreign matter	ISO 4576
	Density	Pyknometer (specified in ISO 758, ISO 1183-1 or ISO 2811-1) Oscillation-type density meters (specified in ISO 15212-1) Immersed body (plummet) method (specified in ISO 2811-2)
	Rheology (viscosity)	Brookfield type rotational viscometer (specified in ISO 2555) Cone-and-plate viscometer (specified in ISO 2884-1) Rotational viscometer (specified in ISO 3219)
	Rheology (thixotropic index)	ISO 20379 using rotational viscometers
	Surface tension	Liquid film (Wilhelmy) method (specified in ISO 304)

Items		Standards for each test method
	Size of conductive materials (spherical particles)	Electric sensing zone method (specified in ISO 13319) Laser diffraction method (specified in ISO 13320) Photon correlation method (specified in ISO 13321) Ultrasonic attenuation spectroscopy method (specified in ISO 20998-1)
	Size of conductive materials (rods, wires and tubes)	Static image analysis method (specified in ISO 13322-1)
	Size of conductive materials (other shapes)	ISO 9276-6
	Flashpoint	Open system; ISO 2592 Closed system; ISO 2719, ISO 3679
	Evaporation rate	Subclause 6.3.6 in this document
	Appearance of ink	Absorbance is measured by equipment specified in ISO 13468-1 or ISO 13468-2.
Properties of a conductive layer (Electrical properties)	Volume resistivity	Four-point probe method (7.2.1 in this document)
	Surface resistivity (four-point probe method)	Subclause 7.2.2 in this document
	Surface resistivity (contactless method)	IEC 62899-202-3
Properties of a conductive layer (Mechanical properties)	Bending test	IEC 62899-202-5
	Abrasion resistance	ISO 18947-1
	Adhesion strength	ISO 2409
Properties of a conductive layer (Optical properties)	Opacity	ISO 2471 (diffuse reflectance)
	Luminous transmittance	Single-beam method (specified in ISO 13468-1) Double-beam method (specified in ISO 13468-2)
	Chromaticity	Presented as the CIE (1976) L*a*b* (according to ISO 11664-4)
	Uniformity of colour	Measure of the chromaticity at 10 points and express with colour difference
	Haze	ISO 14782
	Refractive index	ISO 489 method A

6 Evaluation of properties of conductive ink

6.1 Specimen

The specimen for evaluation shall be prepared in accordance with ISO 14488 or an equivalent method. If necessary, dilution by a compatible solvent ~~may be~~ is allowed.

6.2 Contents

6.2.1 Solid content

6.2.1.1 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.

6.2.1.2 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 (for example company name, city, year).

6.2.2 Non-volatile content

6.2.2.1 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.

6.2.2.2 Test method

The test method shall be as specified in ~~ISO 1625~~ ISO 3251 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) 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 %.

6.2.2.3 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 (for example company name, city, year).

6.2.3 Ash content

6.2.3.1 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.

6.2.3.2 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 (for example company name, city, year).

6.2.4 Foreign matter

6.2.4.1 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.

6.2.4.2 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 different from that 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.

6.2.4.3 Report of the results

The report shall include the following items:

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

6.3 Physical properties

6.3.1 Density

6.3.1.1 Measurement method

The measurement method shall either be the pycnometer 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 measurement method to be used.

6.3.1.2 Equipment

Equipment shall be as specified in the measurement method (see 6.3.1.1) or shall be equipment considered equivalent or superior.

6.3.1.3 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 (for example company name, city, year).

6.3.2 Rheology

6.3.2.1 Measurement method for viscosity

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 measurement method and measuring temperature to be used.

6.3.2.2 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) type of liquidity (such as newtonian, non-newtonian, Ostwald, extended Ostwald, Bingham);
- f) viscosity expressed in millipascal second (mPa·s);
- g) tester, test place, and test time (for example company name, city, year).

6.3.2.3 Measurement for thixotropic index

The thixotropic index can be measured using the methods described in ISO 20379. The rotational viscometers in 6.3.2.1 can be used in this measurement, when the rotational viscometer used is reported.

6.3.2.4 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) measuring temperature;
- c) viscometer model;
- d) measurement conditions (maximum shear rate, standing time, time interval for ascending and descending operation, number of steps when ascending and descending stepwise, shear rate at each step, and sampling interval of data);
- e) viscosity expressed in millipascal second (mPa·s);

6.3.3 Surface tension

6.3.3.1 Measurement 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.

6.3.3.2 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 (for example company name, city, year).

6.3.4 Size of conductive materials

6.3.4.1 Sampling

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

6.3.4.2 Sphere

6.3.4.2.1 Measurement 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.

The DLS (dynamic light scattering) method according to ISO 22412 can be applied in a case where particle size is in submicrometre. However, since proper use of the instrument and interpretation of the result require certain precautions, good practices described in ISO/TR 22814 should be referred to when the DLS method is used.

6.3.4.2.2 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 (for example company name, city, year).

6.3.4.3 Rod, wire and tube

6.3.4.3.1 Measurement 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.

6.3.4.3.2 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 (for example company name, city, year).

6.3.4.4 Other shapes

6.3.4.4.1 Measurement method

For irregularly shaped particles which are not classified in 6.3.4.2 and 6.3.4.3, the mean particle size shall be calculated in accordance with the method specified in ISO 9276-6 or software based on it.

6.3.4.4.2 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 (for example company name, city, year).

6.3.5 Flashpoint

6.3.5.1 Measurement method

Flashpoint shall be measured in accordance with 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~~ or ISO 3679 (closed system) may be applied if a closed system is required.

6.3.5.2 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 (for example company name, city, year).
- f) others (special items).

6.3.6 Evaporation rate

6.3.6.1 General

The evaporation rate is a property which is necessary for the PE ink, but the details of the evaluation condition and the measurement method are significantly different for the ink. In this document, a common framework for 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.

6.3.6.2 Measurement method

The evaporation rate of solvent from an ink formulation can be determined by measuring the time taken to evaporate, for example, 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 be made to allow five or more points to be plotted on a graph of mass loss versus time for values of between 10 % and a higher value, such as 90 % loss of solvent from the ink. In the case where the test is stopped before 90 % loss of solvent from the ink, the test can be stopped when the rate of loss decreases almost to zero according to the graph plotted. The amount of solvent lost (%) should be added to the report in such case. The test should be repeated a total of three 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.

6.3.6.3 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 (for example company name, city, year).

6.3.7 Appearance of ink

6.3.7.1 Test method

For the inks used in PE, unlike graphic inks, the so-called "ink colour" does not pose a problem. However, the degree of suspension of the ink often becomes an issue. The degree of suspension of the ink is tested by using a spectrophotometer specified in ISO 13468-1 or ISO 13468-2. When the absorbance of the ink is 0,01 or less over the whole visible region, the ink can be said to be "transparent". The absorbance measurements using above ISO-specified equipment may also be used to monitor changes in ink condition. However, the relationship between the change of condition and the absorbance change varies depending on the ink, and there is no general principle yet. Details should be by agreement between user and supplier.

6.3.7.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) test atmosphere (temperature and humidity);
- c) results (transparent, or absorbance at a certain wavelength);
- d) tester, test place, and test time (for example company name, city, year).

7 Evaluation of the properties of a conductive layer

7.1 Test piece

7.1.1 General

Test pieces are used for evaluating the conductive layer.

7.1.2 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).

7.1.3 Conductive ink

According to 6.1, except no dilution is allowed.

7.1.4 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 either smaller ~~and/~~ or thinner dimensions than specified, or both, may be used.

7.1.5 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.

7.2 Electrical properties

7.2.1 Volume resistivity

7.2.1.1 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, and measures the potential difference V between the other two electrode, and calculates resistance V/I .

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

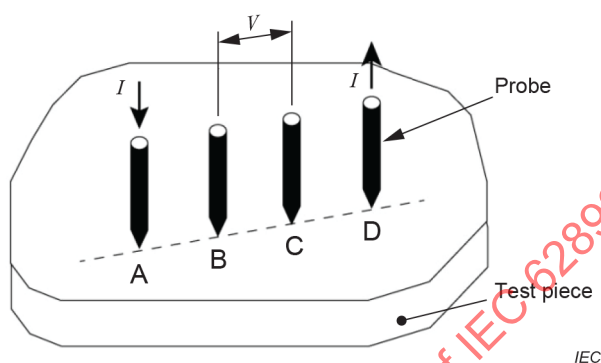


Figure 1 – Example of four-point probe measurement

6.2.1.2 Equipment and tools

7.2.1.2 Test equipment

7.2.1.2.1 Test equipment constitution

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

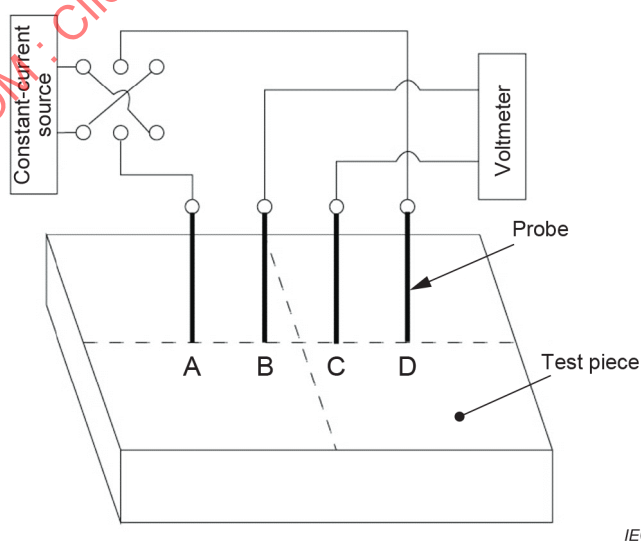


Figure 2 – Example of four-probe measurement equipment

7.2.1.2.2 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 $\pm 0,05$ % or less.

Table 2 – 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 k Ω	< 20 k Ω	< 200 k Ω	< 2 M Ω
Applied current (I_0)	1 000 mA	100 mA	10 mA	1 mA	100 μ A	10 μ A	1 μ A

7.2.1.2.3 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} Ω or higher.

7.2.1.2.4 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} Ω 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 be measured and that its measured value be within ± 3 % of the known value.

7.2.1.3 Measuring equipment for specimen thickness

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

7.2.1.4 Evaluating the test piece

7.2.1.4.1 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 Ω ,
- 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.

7.2.1.4.2 Dimensions of the specimen

The dimensions of the specimen shall be as follows:

- The size of the specimen shall be selected from Table 3.

Table 3 – List of the size of the specimen

	Length (mm)	Width (mm)
Type A	$30 \pm 0,2$	$10 \pm 0,1$
Type B	$30 \pm 0,2$	$30 \pm 0,2$
Type C	$80 \pm 0,2$	$50 \pm 0,2$

- 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.

7.2.1.5 Test procedure

7.2.1.5.1 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

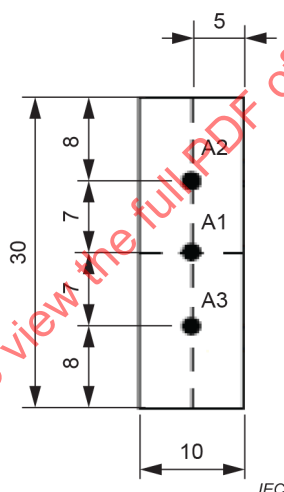


Figure 3 – Measuring positions of resistance (Type A)

Dimensions in millimetres

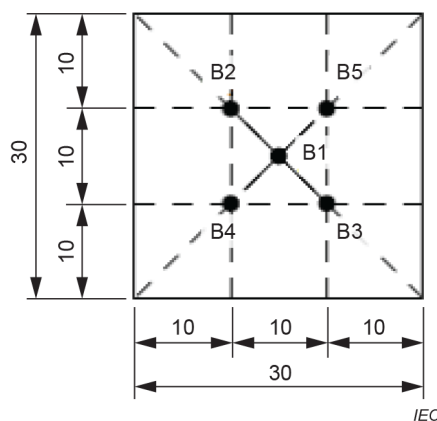


Figure 4 – Measuring positions of resistance (Type B)

Dimensions in millimetres

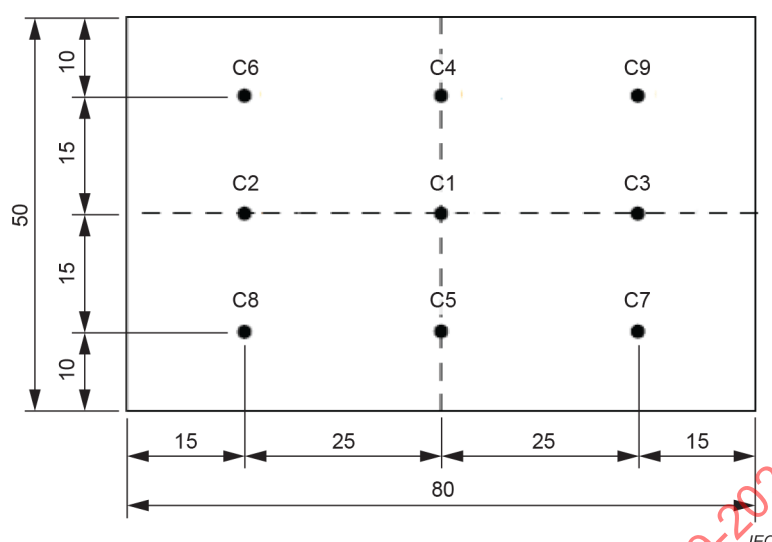


Figure 5 – Measuring positions of resistance (Type C)

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.~~ However, the number of measuring points may be increased or decreased by consideration of the uniformity of the film and the past test results. In that case, the number of measurement points can be selected from 1 point (for Type A, B, and C), 5 points (for Type B and C), and 9 points (for Type C). When measuring only one point, A1, B1, or C1 shall be measured. When measuring five points, B1 to B5 or C1 to C5 shall be measured, respectively. When measuring 9 points, all C1 to C9 shall be measured. 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 a 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.

7.2.1.5.2 Calculating resistivity

Resistivity shall be calculated by using the following formula:

$$\rho = F \cdot t \cdot R \quad (1)$$

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 Formula (2). The same value as the solution of Poisson's formula may be used in the correction factor. (The formula 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.)

$$\begin{aligned}
 F^{-1} = & \frac{y_B - y_C}{a} + \sum_{m=1}^{\infty} \frac{2}{a\zeta \sinh(b\zeta)} \left[\left\{ \cos(\zeta x_B) \cosh \left[\zeta \left(y_B + \frac{b}{2} \right) \right] - \cos(\zeta x_C) \cosh \left[\zeta \left(y_C + \frac{b}{2} \right) \right] \right\} \times \cos(\zeta x_A) \cosh \left[\zeta \left(y_A - \frac{b}{2} \right) \right] \right. \\
 & \left. - \left\{ \cos(\zeta x_B) \cosh \left[\zeta \left(y_B - \frac{b}{2} \right) \right] - \cos(\zeta x_C) \cosh \left[\zeta \left(y_C - \frac{b}{2} \right) \right] \right\} \times \cos(\zeta x_D) \cosh \left[\zeta \left(y_D + \frac{b}{2} \right) \right] \right] \\
 & + \sum_{n=1}^{\infty} \frac{2}{a\eta \sinh(b\eta)} \times \left[\left\{ \cosh \left[\eta \left(y_B + \frac{b}{2} \right) \right] - \cosh \left[\eta \left(y_C + \frac{b}{2} \right) \right] \right\} \times \cosh \left[\eta \left(y_A - \frac{b}{2} \right) \right] \right. \\
 & \left. - \left\{ \cosh \left[\eta \left(y_B - \frac{b}{2} \right) \right] - \cosh \left[\eta \left(y_C - \frac{b}{2} \right) \right] \right\} \times \cosh \left[\eta \left(y_D + \frac{b}{2} \right) \right] \right] \quad (2) \\
 & + \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \frac{4}{a\zeta \sinh(b\zeta)} \times \left[\left\{ \cos(\zeta x_B) \cosh \left[\zeta \left(y_B + \frac{b}{2} \right) \right] - \cos(\zeta x_C) \cosh \left[\zeta \left(y_C + \frac{b}{2} \right) \right] \right\} \times \cos(\zeta x_A) \cosh \left[\zeta \left(y_A - \frac{b}{2} \right) \right] \right. \\
 & \left. - \left\{ \cos(\zeta x_B) \cosh \left[\zeta \left(y_B - \frac{b}{2} \right) \right] - \cos(\zeta x_C) \cosh \left[\zeta \left(y_C - \frac{b}{2} \right) \right] \right\} \times \cos(\zeta x_D) \cosh \left[\zeta \left(y_D + \frac{b}{2} \right) \right] \right]
 \end{aligned}$$

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, and 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 $60 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ 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.

7.2.1.6 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 (for example company name, city, year).

7.2.2 Surface resistivity (based on the four-point probe method)

7.2.2.1 Determination of surface resistivity

Surface resistivity shall be obtained by dividing the volume resistivity measured in 7.2.1 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 7.2.1.5, shall be divided by the thickness of the conductive layer and the difference shall be calculated.

When the result of surface resistivity measurement is reported, the unit [$\Omega/\text{sq.}$] should be used in order to avoid a confusion with the unit of ordinary resistance [Ω] although the unit of surface resistivity is [Ω].

7.2.2.2 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 (for example company name, city, year).

7.2.3 Surface resistivity (based on the contactless method)

7.2.3.1 Determination of surface resistivity

In a continuous roll to roll production, there is a case where it is not possible to measure the conductivity by the contact method described in 7.2.2. In such case, the contactless method can be available. When using the contactless method, the equipment and procedures specified in IEC 62899-202-3 shall apply.

When the result of surface resistivity measurement is reported, the unit [$\Omega/\text{sq.}$] should be used in order to avoid a confusion with the unit of ordinary resistance [Ω] although the unit of surface resistivity is [Ω].

7.2.3.2 Report of the results

The report shall include the following items:

- a) specimen identification (sample ID, thickness of substrate and that of the conductive layer);
- b) test conditions (air pressure and drying temperature);
- c) test apparatus (model of the apparatus and gap between the ferrite core and sample);
- d) surface resistivity;
- e) tester, test place, and test time (for example company name, city, year).

7.3 Mechanical properties

7.3.1 Bending test

7.3.1.1 Test method

The method described in IEC 62899-202-5 shall be applied as the bending test. This method may be used to test the change in resistance or only the physical damage (or destruction) of the sample as well.

7.3.1.2 Report of the results

The report shall include the following items:

- a) specimen identification (including thickness of substrate, thickness of conductive layer, sample size);
- b) test conditions (air pressure and drying temperature);
- c) bending radius and bending strain;
- d) bending direction: inner or outer bending;
- e) linear motion length, interval of bending and number of bending cycles;

- f) initial resistance value and change in the resistance (optional);
- g) images of damaged film (optional);
- h) tester, test place, and test time (for example company name, city, year).

7.3.2 Abrasion resistance

7.3.2.1 Test method

The abrasion resistance shall be tested by using the rub tester specified in ISO 18947-1. The rub tester specified in ISO 18947-1:2021, Clause A.1, should be used, but other equipment specified in ISO 18947-1 may be used with the prior agreement of the user and the supplier. The equipment used shall be reported.

The receptor which rubs with the conductive layer should be the substrate used in the sample or PET, but another material most preferred for the purpose of the test may be used in the case where there is the prior agreement of the user and supplier.

Unless otherwise specified by prior agreement, the weight should produce a force equivalent to 0,6 N/cm², but it shall be at least more than 0,3 N/cm². The cycle of the test should be 1 000 cycles, but it shall be at least more than 100 cycles.

In most cases, the results of the abrasion resistance test are evaluated visually, but optical measurements specified in ISO 18947-1 may also be applied.

For a conductive layer for PCB, the test with an abrasive wheel may also be used. The test conditions on this test shall be decided by agreement between the user and supplier.

NOTE The method specified in ASTM D 4060 can be applied as the abrasion resistance test with abrasive wheel.

7.3.2.2 Report of the results

The report shall include the following items:

- a) specimen identification (including substrate type);
- b) test device (model number, manufacturer, and any unique modifications that have been made);
- c) test conditions (air pressure and drying temperature);
- d) sample size;
- e) a precise description of receptor such as material;
- f) the weight or force applied, the total number of cycles, the rate of cycling, and the line speed (or angular speed);
- g) the result of the test (for example any change such as damage, smudge on unprinted area, transfer of ink, of the sample and receptor, and the visual rank order of the samples);
- h) tester, test place, and test time (for example company name, city, year).

If instrumental evaluation is performed, in addition:

- i) the specific instruments used to measure optical density, colour, and lustre;
- j) abrasion resistance in terms of the change in colour, optical density or lustre of sample;
- k) ink transfer in terms of the change in optical density or colour of the receptor;
- l) scuff in terms of the change in lustre.

7.3.3 Adhesion strength

7.3.3.1 Test method

The tape test described in ISO 2409 shall be applied as the test for adhesion strength. Test results shall be indicated by the classification in accordance with ISO 2409:2020, Table 1. In this table, the results are shown in classes from 0 to 5, with 0 being the best. However, results of tape tests are generally represented by higher numbers as better results. To avoid confusion, the numbering of classification in ISO 2409:2020, Table 1, shall be reversed, with 5 being the best and 0 being the worst in this test.

NOTE ASTM D3359 or ASTM F1842 can be referred to if the procedures in ISO 2409 are insufficient.

7.3.3.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) test conditions (temperature and relative humidity during the test);
- c) the type of cutting tool used and the method of operation (manual or motor-driven);
- d) the results of the test (classification as indicated in ISO 2409:2020, Table 1);
- e) any deviations from the procedure specified;
- f) tester, test place, and test time (for example company name, city, year).

7.4 Optical properties

7.4.1 Overview

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

7.4.2 Luminous transmittance

7.4.2.1 General

Luminous transmittance is presented as total luminous transmittance.

7.4.2.2 Measurement 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 measurement method.

7.4.2.3 Measuring equipment

Measuring equipment shall be as specified in ISO 13468-1:19962019, Clause 4, for single-beam instruments, or ISO 13468-2:19992021, Clause 4, for double-beam instruments, as appropriate. Measuring equipment according to ISO 13655 and/or ISO 5-2 may be used.

7.4.2.4 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, or both.

7.4.2.5 Report of the results

The report shall include the following items:

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

7.4.3 Chromaticity

7.4.3.1 General

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

7.4.3.2 Measurement method

The measurement 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.

7.4.3.3 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, based on their application and purpose, and shall be specified in the detailed product specifications.

7.4.3.4 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.

7.4.3.5 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 (for example company name, city, year).

7.4.4 Uniformity of colour

7.4.4.1 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.

7.4.4.2 Measuring equipment

According to 7.4.3.3.

7.4.4.3 Illuminant (light source)

According to 7.4.3.3.

7.4.4.4 Measurement method

According to 7.4.3, 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.

7.4.4.5 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 following formula:

$$\Delta E_{ab}^* = \left[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2 \right]^{1/2} \quad (3)$$

where:

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

$\Delta L^*, \Delta a^*, \Delta b^*$ is the difference in L^* and difference in colour coordinates a^* and b^* of the chromaticity between the pair of points obtained in 7.4.3.

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

$$\Delta E_{ab}^* = \left[(\Delta L^*)^2 + (\Delta C_{ab}^*)^2 + (\Delta H_{ab}^*)^2 \right]^{1/2} \quad (4)$$

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 7.4.3;

ΔC_{ab}^* is the difference in ab chroma of the chromaticity between the pair of points obtained in 7.4.3;

ΔH_{ab}^* is the difference in ab hue of the chromaticity between the pair of points obtained in 7.4.3.

The colour difference formula in CIEDE2000 (see CIE Publication No. 142) may also be used for calculating the colour difference.

7.4.4.6 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 (for example company name, city, year).

7.4.5 Haze

7.4.5.1 Measurement 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).

7.4.5.2 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 (for example company name, city, year).

7.4.6 Refractive index

7.4.6.1 Measurement 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.

7.4.6.2 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 specified in ISO 489:1999/2022, 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.

7.4.6.3 Conditioning

Conditioning shall be performed at a temperature of $23\text{ °C} \pm 2\text{ °C}$ 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.

7.4.6.4 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 (for example company name, city, year).

8 Storage

8.1 Storage conditions

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

8.2 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 6.3.2.

8.3 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~~ 30 days (mPa·s);
- e) tester, test place, and test time (for example company name, city, year).

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Annex A (informative)

Example of 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.

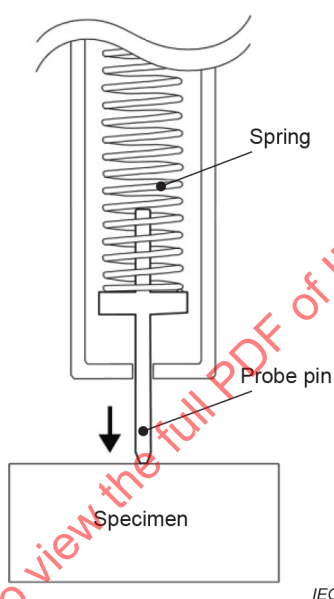


Figure A.1 – Example of the internal structure of probe

A.2 Example of the general overall view

An example of the general overall 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 a rectangular type probe is shown in Figure A.2 as an 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.

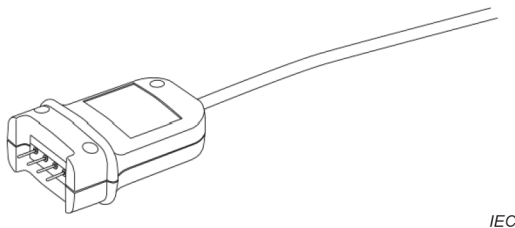


Figure A.2 – Example of the general overall view of the probe

Annex B (informative)

Formula of correction factor F

B.1 General

The resistivity ρ of the conductive layer is defined by the following formula:

$$\rho = Ft \frac{V}{I} \quad (\text{B.1})$$

where:

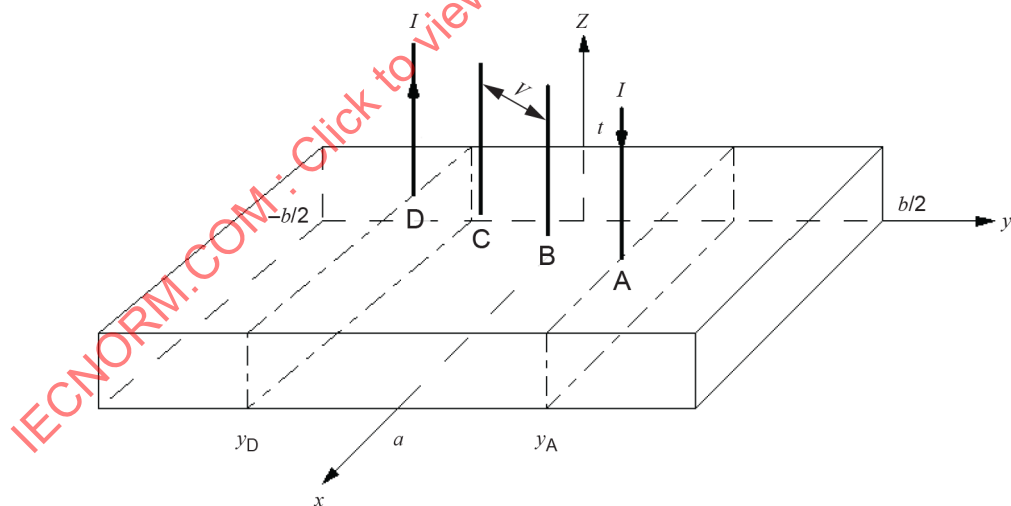
t is the thickness of the conductive layer (cm);

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 , and thickness t .

With the potentials of the probe B and the probe C expressed as φ_B and φ_C respectively, the potential difference V is represented as follows:

$$V = \varphi_B - \varphi_C \quad (\text{B.2})$$



IEC

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 formula, the following formula is established:

$$\nabla^2 \varphi(r) = 2\rho I [\delta(r - r_D) - \delta(r - r_A)] \quad (\text{B.3})$$

where:

- vector r is the coordinates (x, y, z) of any point in the conductive layer;
- r_A is the coordinates (x_A, y_A, t) of the contact point of the probe A and the conductive layer;
- r_D is the coordinates (x_D, y_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 $q = \epsilon^* \rho I / 2\pi$ 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, Formula (B.3) equals 0 due to the nature of the delta function, and Laplace's formula is obtained.

By applying the following three conditions in order to obtain the solution of Laplace's formula, 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 Formula of correction factor F

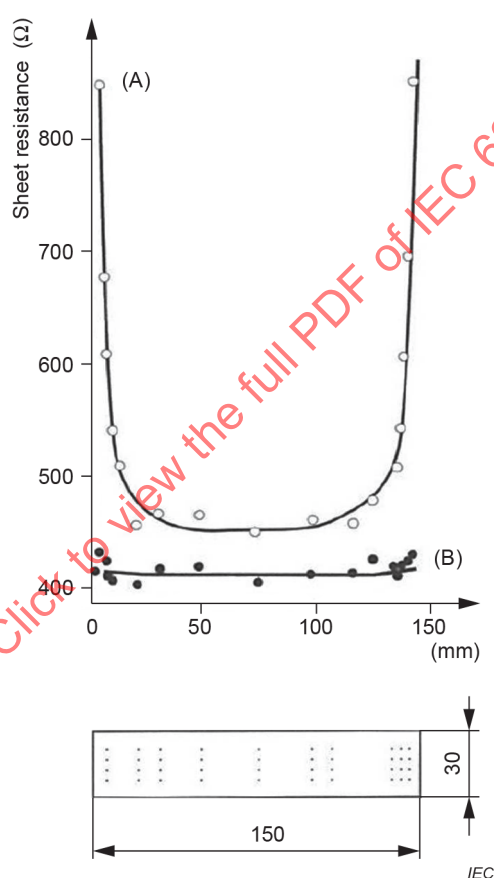
From these conditions, the correction factor F can be expressed as F^{-1} using Formula (2) in 7.2.1.5.2.

Annex C (informative)

Influence of the measuring position and size of the 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 formula 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 formula. 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 formula (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 formula is required in order to obtain stable and proper resistance.

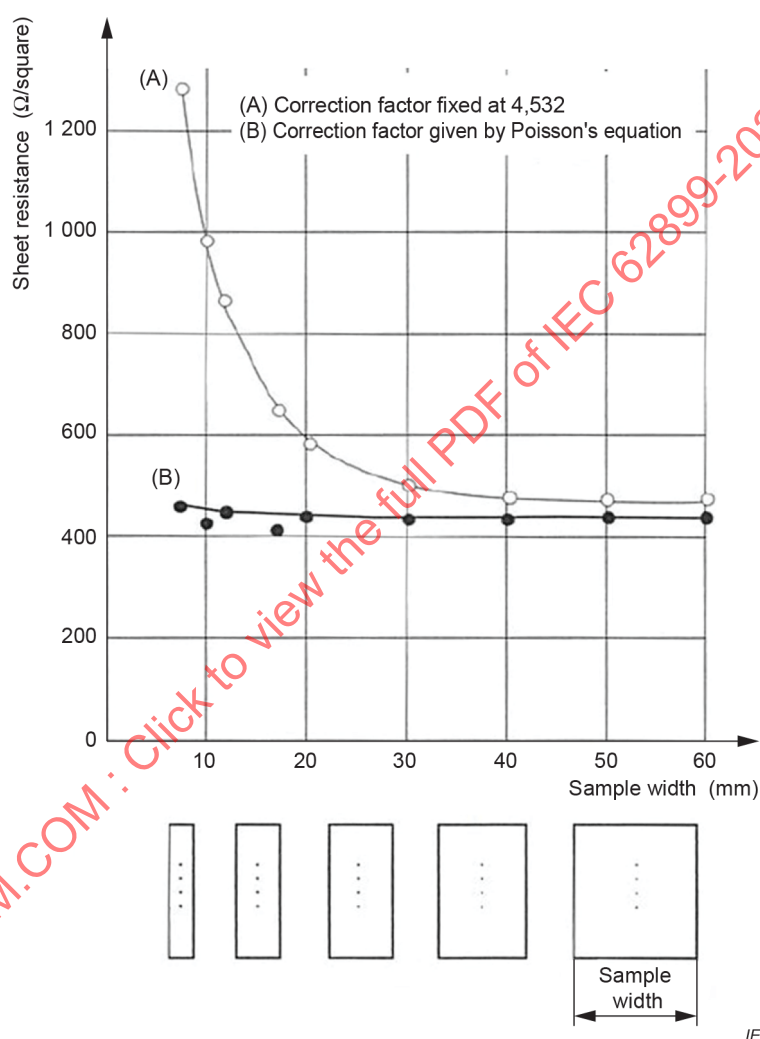


Figure C.2 – Model measurement of the influence of the specimen size

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 formula. The rectangle under the figure expresses the image of the measured specimen. The black points express the position of the probe.

Bibliography

IEC 60050 (all parts), *International Electrotechnical Vocabulary (IEV)*, available at www.electropedia.org

IEC 60050-212, *International Electrotechnical Vocabulary (IEV) – Part 212: Electrical insulating solids, liquids and gases*, available at www.electropedia.org

IEC 60695-4, *Fire hazard testing – Part 4: Terminology concerning fire tests for electrotechnical products*

ISO 862:1984, *Surface active agents – Vocabulary*

~~ISO 11664-4, *Colorimetry – Part 4: CIE 1976 $L^*a^*b^*$ Colour Space*~~

ISO/CIE 11664-6, *Colorimetry – Part 6: CIEDE2000 colour-difference formula*

ISO 22412, *Particle size analysis – Dynamic light scattering (DLS)*

ISO/TR 22814, *Good practice for dynamic light scattering (DLS) measurements*

CIE Publication No.142, *Improvement to Industrial colour – Difference Evaluation (2001)*

ASTM D3359 – 22, *Standard Test Methods for Rating Adhesion by Tape Test*

ASTM D 4060, *Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser*

ASTM F1842 – 15, *Standard Test Method for Determining Ink or Coating Adhesion on Flexible Substrates for a Membrane Switch or Printed Electronic Device*

IPC/JPCA-4591, *Requirements for Printed Electronics Functional Conductive Materials*

JEITA ET-4201, *Printed Electronics – Materials – Part 2-1: Ink of functional materials – Conductive material ink*

Masato Yamashita, Masahiro Agu, "Geometrical Correction Factor for Semiconductor Resistivity Measurements by Four-Point Probe Method" Jpn. J. Appl. Phys. Vol 23, 1984, 1499

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INTERNATIONAL STANDARD

**Printed electronics –
Part 202: Materials – Conductive ink**

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

PRINTED ELECTRONICS –

Part 202: Materials – Conductive ink

FOREWORD

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

This second edition cancels and replaces the first edition published in 2016. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

- a) definitions of conductive material, conductive ink and conductive layer have been revised;
- b) a summary of test methods is added;
- c) mechanical tests for conductive layer are added.

The text of this International Standard is based on the following documents:

Draft	Report on voting
119/423/FDIS	119/428/RVD

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

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

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 document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

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

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INTRODUCTION

Printed electronics is a technology that spans the printing and electrical/electronic area, and it provides a variety of products. Since participants in this industry come from different areas, their backgrounds and customs can be barriers to smooth communication and transactions in the supply chain. The printed electronics industry continues to grow, but many barriers still remain. Particularly, the lack of standardised terms and evaluation methods is one of the major factors that inhibit smooth communication.

This document focuses on measurement and evaluation methods for conductive inks and provides tools to promote the smooth communication within the supply chain.

This document specifies the basic items to be communicated and their measurement or evaluation methods. This document includes the measurement methods for the basic properties of inks and electrical conductivity, which is obtained by the post treatment of inks. Additionally, storage methods, packaging and marking, and transportation conditions are also included.

This document is part of the IEC 62899-202 series and similar documents are available for other materials used in printed electronics.

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

IEC 62899-201: Materials – Substrates

IEC 62899-202: Materials – Conductive ink

IEC 62899-203: Materials – Semiconductor ink

IEC 62899-204: Materials – Insulator ink

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

PRINTED ELECTRONICS –

Part 202: Materials – Conductive ink

1 Scope

This part of IEC 62899 defines the terms and specifies the standard test methods for characterization and evaluation of conductive inks.

This document also provides measurement methods for evaluating the properties of conductive layers made both from an additive process using conductive inks and from a subtractive process used in printed electronics.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 62899-202-3, *Printed electronics – Part 202-3: Materials – Conductive ink – Measurement of sheet resistance of conductive films – Contactless method*

IEC 62899-202-5, *Printed electronics – Part 202-5: Materials – Conductive ink – Mechanical bending test of a printed conductive layer on an insulating substrate*

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 489:2022, *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 pycnometer method and titration method*

ISO 2409:2020, *Paints and varnishes – Cross-cut test*

ISO 2471, *Paper and board – Determination of opacity (paper backing) – Diffuse reflectance method*

ISO 2555, *Plastics – Resins in the liquid state or as emulsions or dispersions – Determination of apparent viscosity using a single cylinder type rotational viscometer method*

ISO 2592, *Petroleum and related products – 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: Pycnometer 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 3251, *Paints, varnishes and plastics – Determination of non-volatile-matter content*

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 11664-4, *Colorimetry – Part 4: CIE 1976 L*a*b* colour space*

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 13468-1:2019, *Plastics – Determination of the total luminous transmittance of transparent materials – Part 1: Single beam instrument*

ISO 13468-2:2021, *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 powders in liquids*

ISO 15212-1, *Oscillation-type density meters – Part 1: Laboratory instruments*

ISO 18947-1:2021, *Imaging materials and prints – Abrasion resistance – Part 1: General rub testing methods*

ISO 20379, *Fine ceramics (advanced ceramics, advanced technical ceramics) – Measurement of thixotropic behaviour of ceramic slurry by use of a rotational viscometer*

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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

NOTE Words in italics are defined within Clause 3.

3.1

conductive material

ingredient of a printing or coating component with intrinsic property providing electrical conductivity

Note 1 to entry: The ingredient can be one or more small molecules, precursors, polymers, or particles.

Note 2 to entry: The ingredient can require post treatment to provide electrical conductivity

3.2

conductive ink

fluid in which one or more *conductive materials* (3.1) are dissolved or dispersed, and which is used to form an electrically conductive structure

3.3

conductive layer

film-like structure formed by printing or coating and *post treatment* (3.4) of *conductive ink* (3.2) on a substrate, which is electrically conductive

3.4

post treatment

process step following the deposition of ink to generate the intended functionality

Note 1 to entry: Process steps can be evaporation, annealing, curing or sintering

3.5

conductive film

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

3.6

solid content

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

3.7

non-volatile content

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

3.8

ash content

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

3.9

foreign matter

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

3.10

spherical particle

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

3.11

rod

substance with thin elongated cylindrical shape

3.12

wire

flexible cylindrical conductor, with or without an insulating covering, the length of which is large with respect to its cross-sectional dimensions

Note 1 to entry: The cross-section of a wire may have any shape, but the term "wire" is not generally used for ribbons or tapes.

[SOURCE: IEC 60050-151:2001, 151-12-28]

3.13

tube

substance with fiber-like hollow cylindrical shape

3.14

dispersion

system consisting of two or more phases one of which is continuous and at least one other is finely dispersed

[SOURCE: IEC 62899-101:2019, 3.31]

3.15

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 pre-conditioning

The standard atmosphere for evaluation (test and measurement) and storage of the specimen shall be a temperature of $23\text{ °C} \pm 2\text{ °C}$ 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 $23\text{ °C} \pm 1\text{ °C}$ and relative humidity of $(50 \pm 5)\%$, conforming to standard atmosphere class 1 specified in ISO 291. Atmospheric pressure in test and measurement may be specified in a prior agreement of trade partners, but it shall be reported.

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

5 Summary characteristics and evaluation method of conductive ink

The conductive inks used in printed electronics shall be tested in accordance with the methods specified in Table 1. Unless there is a prior agreement between the user and supplier these test methods shall be applied without modification. In cases where the test has been modified, the changed condition shall be described in the report.

Table 1 – Test methods for conductive inks used in PE

	Items	Standards for each test method
Ink properties	Non-volatile content	ISO 3251
	Ash content	ISO 3451-1 method A
	Foreign matter	ISO 4576
	Density	Pyknometer (specified in ISO 758, ISO 1183-1 or ISO 2811-1) Oscillation-type density meters (specified in ISO 15212-1) Immersed body (plummet) method (specified in ISO 2811-2)
	Rheology (viscosity)	Brookfield type rotational viscometer (specified in ISO 2555) Cone-and-plate viscometer (specified in ISO 2884-1) Rotational viscometer (specified in ISO 3219)
	Rheology (thixotropic index)	ISO 20379 using rotational viscometers
	Surface tension	Liquid film (Wilhelmy) method (specified in ISO 304)
	Size of conductive materials (spherical particles)	Electric sensing zone method (specified in ISO 13319) Laser diffraction method (specified in ISO 13320) Photon correlation method (specified in ISO 13321) Ultrasonic attenuation spectroscopy method (specified in ISO 20998-1)
	Size of conductive materials (rods, wires and tubes)	Static image analysis method (specified in ISO 13322-1)
	Size of conductive materials (other shapes)	ISO 9276-6
	Flashpoint	Open system; ISO 2592 Closed system; ISO 2719, ISO 3679
	Evaporation rate	Subclause 6.3.6 in this document
	Appearance of ink	Absorbance is measured by equipment specified in ISO 13468-1 or ISO 13468-2.

Items		Standards for each test method
Properties of a conductive layer (Electrical properties)	Volume resistivity	Four-point probe method (7.2.1 in this document)
	Surface resistivity (four-point probe method)	Subclause 7.2.2 in this document
	Surface resistivity (contactless method)	IEC 62899-202-3
Properties of a conductive layer (Mechanical properties)	Bending test	IEC 62899-202-5
	Abrasion resistance	ISO 18947-1
	Adhesion strength	ISO 2409
Properties of a conductive layer (Optical properties)	Opacity	ISO 2471 (diffuse reflectance)
	Luminous transmittance	Single-beam method (specified in ISO 13468-1) Double-beam method (specified in ISO 13468-2)
	Chromaticity	Presented as the CIE (1976) $L^*a^*b^*$ (according to ISO 11664-4)
	Uniformity of colour	Measure of the chromaticity at 10 points and express with colour difference
	Haze	ISO 14782
	Refractive index	ISO 489 method A

6 Evaluation of properties of conductive ink

6.1 Specimen

The specimen for evaluation shall be prepared in accordance with ISO 14488 or an equivalent method. If necessary, dilution by a compatible solvent is allowed.

6.2 Contents

6.2.1 Solid content

6.2.1.1 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.

6.2.1.2 Report of the results

The report shall include the following items:

- unique specimen identification;
- atmospheric conditions of test;
- solid content;
- tester, test place, and test time (for example company name, city, year).

6.2.2 Non-volatile content

6.2.2.1 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.

6.2.2.2 Test method

The test method shall be as specified in ISO 3251 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) 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 %.

6.2.2.3 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 (for example company name, city, year).

6.2.3 Ash content

6.2.3.1 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.

6.2.3.2 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 (for example company name, city, year).

6.2.4 Foreign matter

6.2.4.1 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.

6.2.4.2 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 different from that 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.

6.2.4.3 Report of the results

The report shall include the following items:

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

6.3 Physical properties

6.3.1 Density

6.3.1.1 Measurement method

The measurement method shall either be the pycnometer 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 measurement method to be used.

6.3.1.2 Equipment

Equipment shall be as specified in the measurement method (see 6.3.1.1) or shall be equipment considered equivalent or superior.

6.3.1.3 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 (for example company name, city, year).

6.3.2 Rheology

6.3.2.1 Measurement method for viscosity

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 measurement method and measuring temperature to be used.

6.3.2.2 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) type of liquidity (such as newtonian, non-newtonian, Ostwald, extended Ostwald, Bingham);
- f) viscosity expressed in millipascal second (mPa·s);
- g) tester, test place, and test time (for example company name, city, year).

6.3.2.3 Measurement for thixotropic index

The thixotropic index can be measured using the methods described in ISO 20379. The rotational viscometers in 6.3.2.1 can be used in this measurement, when the rotational viscometer used is reported.

6.3.2.4 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) measuring temperature;
- c) viscometer model;
- d) measurement conditions (maximum shear rate, standing time, time interval for ascending and descending operation, number of steps when ascending and descending stepwise, shear rate at each step, and sampling interval of data);
- e) viscosity expressed in millipascal second (mPa·s);

6.3.3 Surface tension

6.3.3.1 Measurement 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.

6.3.3.2 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 (for example company name, city, year).

6.3.4 Size of conductive materials

6.3.4.1 Sampling

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

6.3.4.2 Sphere

6.3.4.2.1 Measurement 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.

The DLS (dynamic light scattering) method according to ISO 22412 can be applied in a case where particle size is in submicrometre. However, since proper use of the instrument and interpretation of the result require certain precautions, good practices described in ISO/TR 22814 should be referred to when the DLS method is used.

6.3.4.2.2 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 (for example company name, city, year).

6.3.4.3 Rod, wire and tube

6.3.4.3.1 Measurement 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.

6.3.4.3.2 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 (for example company name, city, year).

6.3.4.4 Other shapes

6.3.4.4.1 Measurement method

For irregularly shaped particles which are not classified in 6.3.4.2 and 6.3.4.3, the mean particle size shall be calculated in accordance with the method specified in ISO 9276-6 or software based on it.

6.3.4.4.2 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 (for example company name, city, year).

6.3.5 Flashpoint

6.3.5.1 Measurement method

Flashpoint shall be measured in accordance with 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) or ISO 3679 (closed system) may be applied if a closed system is required.

6.3.5.2 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 (for example company name, city, year).
- f) others (special items).

6.3.6 Evaporation rate

6.3.6.1 General

The evaporation rate is a property which is necessary for the PE ink, but the details of the evaluation condition and the measurement method are significantly different for the ink. In this document, a common framework for 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.

6.3.6.2 Measurement method

The evaporation rate of solvent from an ink formulation can be determined by measuring the time taken to evaporate, for example, 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 be made to allow five or more points to be plotted on a graph of mass loss versus time for values of between 10 % and a higher value, such as 90 % loss of solvent from the ink. In the case where the test is stopped before 90 % loss of solvent from the ink, the test can be stopped when the rate of loss decreases almost to zero according to the graph plotted. The amount of solvent lost (%) should be added to the report in such case. The test should be repeated a total of three 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.

6.3.6.3 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 (for example company name, city, year).

6.3.7 Appearance of ink

6.3.7.1 Test method

For the inks used in PE, unlike graphic inks, the so-called "ink colour" does not pose a problem. However, the degree of suspension of the ink often becomes an issue. The degree of suspension of the ink is tested by using a spectrophotometer specified in ISO 13468-1 or ISO 13468-2. When the absorbance of the ink is 0,01 or less over the whole visible region, the ink can be said to be "transparent". The absorbance measurements using above ISO-specified equipment may also be used to monitor changes in ink condition. However, the relationship between the change of condition and the absorbance change varies depending on the ink, and there is no general principle yet. Details should be by agreement between user and supplier.

6.3.7.2 Report of the results

The report shall include the following items:

- a) specimen identification;
- b) test atmosphere (temperature and humidity);
- c) results (transparent, or absorbance at a certain wavelength);
- d) tester, test place, and test time (for example company name, city, year).

7 Evaluation of the properties of a conductive layer

7.1 Test piece

7.1.1 General

Test pieces are used for evaluating the conductive layer.

7.1.2 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).

7.1.3 Conductive ink

According to 6.1, except no dilution is allowed.

7.1.4 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 either smaller or thinner dimensions than specified, or both, may be used.

7.1.5 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.

7.2 Electrical properties

7.2.1 Volume resistivity

7.2.1.1 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 .

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

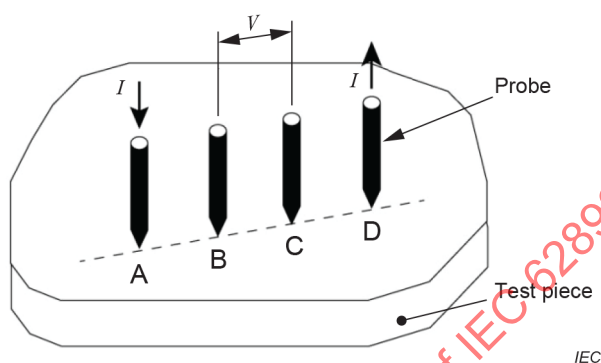


Figure 1 – Example of four-point probe measurement

7.2.1.2 Test equipment

7.2.1.2.1 Test equipment constitution

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

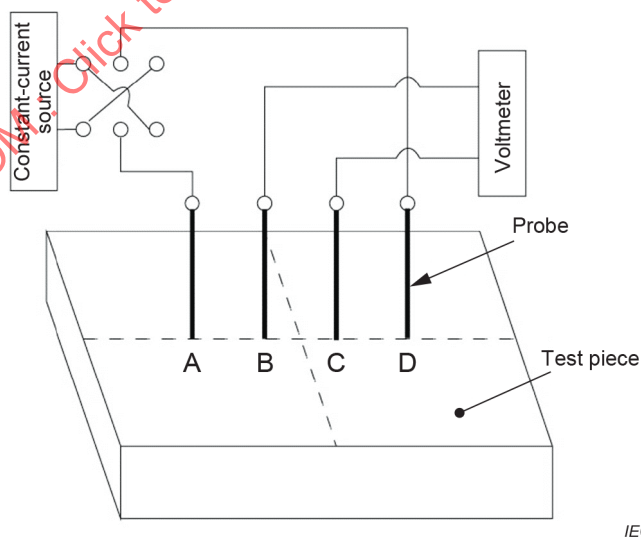


Figure 2 – Example of four-probe measurement equipment

7.2.1.2.2 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 $\pm 0,05$ % or less.

Table 2 – 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 k Ω	< 20 k Ω	< 200 k Ω	< 2 M Ω
Applied current (I_0)	1 000 mA	100 mA	10 mA	1 mA	100 μ A	10 μ A	1 μ A

7.2.1.2.3 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} Ω or higher.

7.2.1.2.4 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} Ω or higher. The spacing may be 0,5 mm, 1,0 mm, 2,2 mm, or 5,0 mm. 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 be measured and that its measured value be within ± 3 % of the known value.

7.2.1.3 Measuring equipment for specimen thickness

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

7.2.1.4 Evaluating the test piece

7.2.1.4.1 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 Ω ,
- 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.

7.2.1.4.2 Dimensions of the specimen

The dimensions of the specimen shall be as follows:

- a) The size of the specimen shall be selected from Table 3.

Table 3 – List of the size of the specimen

	Length (mm)	Width (mm)
Type A	$30 \pm 0,2$	$10 \pm 0,1$
Type B	$30 \pm 0,2$	$30 \pm 0,2$
Type C	$80 \pm 0,2$	$50 \pm 0,2$

- 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.

7.2.1.5 Test procedure

7.2.1.5.1 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

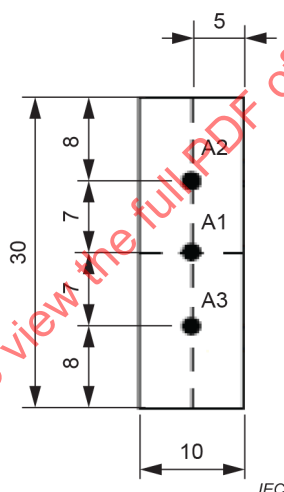


Figure 3 – Measuring positions of resistance (Type A)

Dimensions in millimetres

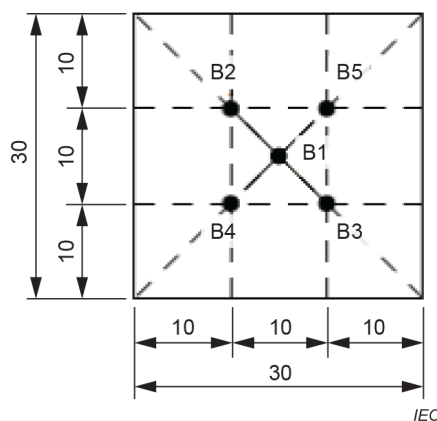


Figure 4 – Measuring positions of resistance (Type B)

Dimensions in millimetres

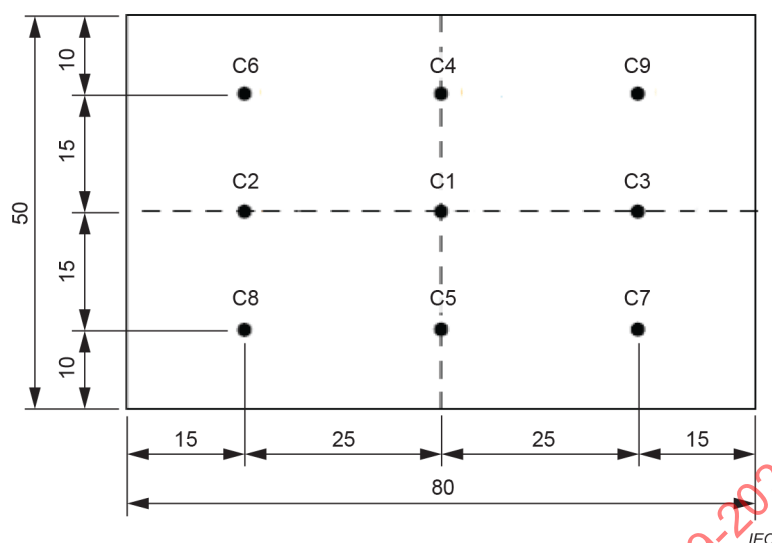


Figure 5 – Measuring positions of resistance (Type C)

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, the number of measuring points may be increased or decreased by consideration of the uniformity of the film and the past test results. In that case, the number of measurement points can be selected from 1 point (for Type A, B, and C), 5 points (for Type B and C), and 9 points (for Type C). When measuring only one point, A1, B1, or C1 shall be measured. When measuring five points, B1 to B5 or C1 to C5 shall be measured, respectively. When measuring 9 points, all C1 to C9 shall be measured. 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 a 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.

7.2.1.5.2 Calculating resistivity

Resistivity shall be calculated by using the following formula:

$$\rho = F \cdot t \cdot R \quad (1)$$

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 Formula (2). The same value as the solution of Poisson's formula may be used in the correction factor. (The formula 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.)

$$\begin{aligned}
 F^{-1} = & \frac{y_B - y_C}{a} + \sum_{m=1}^{\infty} \frac{2}{a\zeta \sinh(b\zeta)} \left[\left\{ \cos(\zeta x_B) \cosh \left[\zeta \left(y_B + \frac{b}{2} \right) \right] - \cos(\zeta x_C) \cosh \left[\zeta \left(y_C + \frac{b}{2} \right) \right] \right\} \times \cos(\zeta x_A) \cosh \left[\zeta \left(y_A - \frac{b}{2} \right) \right] \right. \\
 & \left. - \left\{ \cos(\zeta x_B) \cosh \left[\zeta \left(y_B - \frac{b}{2} \right) \right] - \cos(\zeta x_C) \cosh \left[\zeta \left(y_C - \frac{b}{2} \right) \right] \right\} \times \cos(\zeta x_D) \cosh \left[\zeta \left(y_D + \frac{b}{2} \right) \right] \right] \\
 & + \sum_{n=1}^{\infty} \frac{2}{a\eta \sinh(b\eta)} \times \left[\left\{ \cosh \left[\eta \left(y_B + \frac{b}{2} \right) \right] - \cosh \left[\eta \left(y_C + \frac{b}{2} \right) \right] \right\} \times \cosh \left[\eta \left(y_A - \frac{b}{2} \right) \right] \right. \\
 & \left. - \left\{ \cosh \left[\eta \left(y_B - \frac{b}{2} \right) \right] - \cosh \left[\eta \left(y_C - \frac{b}{2} \right) \right] \right\} \times \cosh \left[\eta \left(y_D + \frac{b}{2} \right) \right] \right] \quad (2) \\
 & + \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \frac{4}{a\zeta \sinh(b\zeta)} \times \left[\left\{ \cos(\zeta x_B) \cosh \left[\zeta \left(y_B + \frac{b}{2} \right) \right] - \cos(\zeta x_C) \cosh \left[\zeta \left(y_C + \frac{b}{2} \right) \right] \right\} \times \cos(\zeta x_A) \cosh \left[\zeta \left(y_A - \frac{b}{2} \right) \right] \right. \\
 & \left. - \left\{ \cos(\zeta x_B) \cosh \left[\zeta \left(y_B - \frac{b}{2} \right) \right] - \cos(\zeta x_C) \cosh \left[\zeta \left(y_C - \frac{b}{2} \right) \right] \right\} \times \cos(\zeta x_D) \cosh \left[\zeta \left(y_D + \frac{b}{2} \right) \right] \right]
 \end{aligned}$$

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, and 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 $60 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ 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.

7.2.1.6 Report of the results

The report shall include the following items:

- specimen identification;
- test conditions (air pressure and drying temperature);
- test piece size;
- volume resistivity and error;
- tester, test place, and test time (for example company name, city, year).

7.2.2 Surface resistivity (based on the four-point probe method)

7.2.2.1 Determination of surface resistivity

Surface resistivity shall be obtained by dividing the volume resistivity measured in 7.2.1 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 7.2.1.5, shall be divided by the thickness of the conductive layer and the difference shall be calculated.