

INTERNATIONAL STANDARD



**Printed electronics –
Part 204: Materials – Insulator ink – Measurement methods of properties of
insulator inks and printed insulating layers**



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INTERNATIONAL STANDARD



**Printed electronics –
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insulator inks and printed insulating layers**

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

PRINTED ELECTRONICS –

**Part 204: Materials – Insulator ink –
Measurement methods of properties of
insulator inks and printed insulating layers**

FOREWORD

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International Standard IEC 62899-204 has been prepared by IEC technical committee 119: Printed Electronics.

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

FDIS	Report on voting
119/256/FDIS	119/268/RVD

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

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

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

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

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- withdrawn,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

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

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INTRODUCTION

The IEC 62899 series deals mainly with evaluation methods for materials of printed electronics. The series also includes storage methods, packaging and marking, and transportation conditions.

The IEC 62899 series is divided into several parts according to each material. Each part is prepared as a generic specification containing fundamental information for the area of printing electronics.

The IEC 62899 series consists of the following parts:

Part 1: Terminology

Part 201: Materials – Substrates

Part 202: Materials – Conductive ink

Part 203: Materials – Semiconductor ink

Part 250: Material technologies required in printed electronics for wearable smart devices

Part 301-X: Equipment – Contact printing – Rigid master

Part 302-X: Equipment – Inkjet

Part 303-X: Equipment – Roll-to-roll printing

Part 401: Printability – Overview

Part 402-X: Printability – Measurement of qualities

Part 403-X: Printability – Requirements for reproducibility

Part 502-X: Quality assessment – Organic light emitting diode (OLED) elements

Furthermore, sectional specifications, blank detail specifications, and detail specifications for each material will be based on these parts.

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

PRINTED ELECTRONICS –

Part 204: Materials – Insulator ink – Measurement methods of properties of insulator inks and printed insulating layers

1 Scope

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

This document is applicable to insulator inks and printed insulating layers that are made from insulator inks used for printed electronics. The insulator inks include dielectric inks.

2 Normative references

The following documents are referred to in the text 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 60243 (all parts), *Electric strength of insulating materials – Test methods*

IEC 62631-2-1, *Dielectric and resistive properties of solid insulating materials – Part 2-1: Relative permittivity and dissipation factor – Technical frequencies (0,1 Hz to 10 MHz) – AC methods*

IEC 62899-201, *Printed electronics – Part 201: Materials – Substrates*

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 291, *Plastics – Standard atmospheres for conditioning and testing*

ISO 304, *Surface active agents – Determination of surface tension by drawing up liquid films*

ISO 489, *Plastics – Determination of refractive index*

ISO 758, *Liquid chemical products for industrial use – Determination of density at 20 °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 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 3664, *Graphic technology and photography – Viewing conditions*

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

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

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

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

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

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

ISO 14782, *Plastics – Determination of haze for transparent materials*

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

ISO 19403-1, *Paints and varnishes – Wettability – Part 1: Terminology and general principles*

ISO 19403-3, *Paints and varnishes – Wettability – Part 3: Determination of the surface tension of liquids using the pendant drop method*

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

NOTE The terms in italics are those defined in Clause 3.

3.1

insulator material

element of a printing or coating material, which itself is electrically insulating or becomes electrically insulating by the application of a post treatment such as heating

3.2

insulator ink

fluid in which one or several small molecules, polymers, or particles are dissolved or dispersed, and which becomes an electrically *insulating layer* (3.3) by the application of a post treatment such as heating

3.3

insulating layer

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

3.4

insulator film

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

3.5

solid content

mass fraction of an element which effectively functions as an insulating substance, in *insulator ink* (3.2)

3.6

non-volatile content

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

3.7

flash point

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

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

4 Atmospheric conditions for evaluation and conditioning

The standard atmosphere for evaluation (test and measurement) and storage of the specimen shall be a temperature of $23\text{ °C} \pm 2\text{ °C}$ and relative humidity of $(50 \pm 10)\%$, conforming to standard atmosphere class 2 as specified in ISO 291.

5 Measurement methods of properties of insulator ink

5.1 General

The insulator ink shall be tested by 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 insulator ink

Items		Documents in which each test method is defined
Evaluation of properties of insulator ink	Specimen	ISO 14488
	Density	Pycnometer method: ISO 758, ISO 1183-1, ISO 2811-1 Oscillation-type method: ISO 15212-1 Immersed body (plummet) method: ISO 2811-2
	Rheology	ISO 2555 (Brookfield-type rotational viscometer) ISO 2884-1 (cone-and-plate) ISO 3219 (rotational viscometer)
	Surface tension	ISO 304 (Wilhelmy method) ISO 19403-3 (constitution of the equipment, test method) ISO 19403-1 (pendant drop method)
	Flash point	ISO 2592 (open system) ISO 2719 (closed system) ISO 3679 (closed system)

5.2 Physical properties

5.2.1 Density

5.2.1.1 Measuring method

The measuring 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 measuring method to be used.

5.2.1.2 Equipment

Equipment shall be as specified in the measuring method (see 5.2.1.1) or shall be equipment considered equivalent or superior.

5.2.1.3 Report of the results

The report shall include the following:

- specimen identification;
- measuring method;
- measurement atmosphere (temperature and relative humidity);
- results.

5.2.2 Rheology

5.2.2.1 Measuring method

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

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

5.2.2.2 Report of the results

The report shall include the following:

- a) standard number of the measuring method;
- b) specimen identification;
- c) measuring temperature;
- d) viscometer model;
- e) viscosity expressed in millipascal second (mPa·s).

5.2.3 Surface tension

5.2.3.1 Measuring methods for surface tension

Surface tension shall be measured using the drawing up liquid film (Wilhelmy) method as specified in ISO 304 and the pendant drop method specified in 5.2.3.2.

5.2.3.2 Pendant drop method

5.2.3.2.1 Test equipment

The test equipment is composed of several parts such as a light source, graduated syringe, and needle. The composition of the equipment is specified in detail in ISO 19403-3.

5.2.3.2.2 Test method

The test method is determined in ISO 19403-3.

5.2.3.2.3 Measuring method

The surface tension of the ink is determined using the pendant drop method which shall be calculated with the Young-Laplace method specified in ISO 19403-1 and ISO 19403-3. The surface tension may also be calculated by the following method:

$$d_s/d_e$$

where d_e is the maximum diameter (cross sectional diameter) of the pendant drop and d_s is the diameter of the pendant drop where it is equal to the length of d_e from the bottom. This method analyses the interfacial tension by measuring the diameters d_e and d_s , as shown in Figure.1.

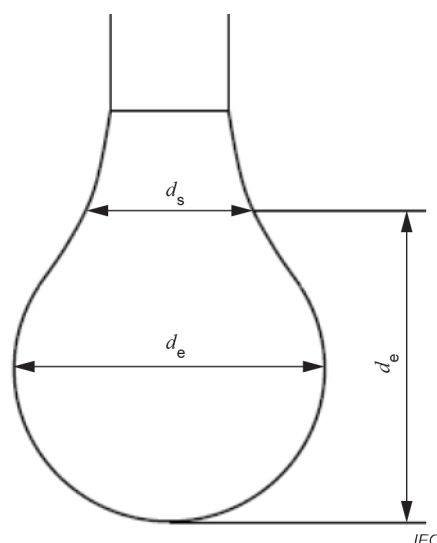


Figure 1 – Calculation of the surface tension of the droplet

The interfacial tension can be determined by the following formula:

$$\gamma = \Delta\rho g d_e^2 / H$$

where, $\Delta\rho$ is the difference of density between the drop phase and surrounding phase, g is the gravitational constant, and $1/H$ is the correction factor determined by the result of d_s/d_e . The correction factor is described in the references listed in the Bibliography.

5.2.3.2.4 Other methods

A temperature controller can be available to control the temperature of the atmosphere of the specimen. The property of the ink strongly depends on the temperature. The temperature controller is used to keep the temperature constant in the atmosphere of the measurement. The viscosity of the specimen shall be under 10 000 mPa·s (25 °C; shear rate 1.15 s⁻¹).

5.2.3.3 Report of the result

The report shall include the following:

- density of the specimen ink;
- average surface tension;
- temperature of the atmosphere;
- measuring method.

5.2.4 Flash point

5.2.4.1 Measuring method

The flash point shall be measured according to ISO 2592 (open system). The "open system" method is preferable as it is safer; however, "closed systems" are also widely used. The measuring method based on ISO 2719 (closed system) and ISO 3679 (closed system) may be applied if a closed system is required.

5.2.4.2 Report of the results

The report shall include the following:

- 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) others (special items).

6 Measurement of properties of the insulating layer

6.1 General

6.1.1 Overview

The insulating layer shall be tested by the methods specified in Table 2. Unless there is a prior agreement between the user and supplier, these methods shall be applied without modification. In case where the test has been modified, the changed condition shall be described in the report.

6.1.2 Test methods for insulating layer

Table 2 – Test methods for insulating layer

Items		Documents in which each test method is defined
Evaluation of properties of insulating layer	Test piece	6.1.3
	Substrate	6.1.4
	Insulator ink	6.1.5
	Dimensions of test piece	6.1.6
	Preparation of test piece	6.1.7
	Volume resistivity	IEC 62899-201 (film, coated on a support and calculation)
	Dielectric constant	IEC 62631-2-1 (dielectric constant, dielectric loss, sample preparation and measurement)
	Electric strength	6.3.3 or IEC 60243 (all parts) IEC 62631-2-1 (sample preparation)
	Luminous transmittance	ISO 13468-1 (single-beam method) ISO 13468-2 (double-beam method) Measuring equipment: ISO 13468-1:1996, Clause 4, ISO 13468-2:1999, Clause 4 ISO 13655, ISO 5-2
	Chromaticity	ISO 11664-4 Measuring equipment: ISO 5-2, ISO 5-3, ISO 3664, ISO 13655
	Uniformity of colour	6.3.4
	Haze	ISO 14782 (measuring method)
	Refractive index	ISO 489 (measuring method and contacting liquid)

6.1.3 Test piece

Test pieces are used for evaluating the insulating layer.

6.1.4 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 (user and supplier).

6.1.5 Insulator ink

Dilution is allowed, if necessary.

6.1.6 Dimensions of test piece

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

6.1.7 Preparation of test piece

The test piece shall be prepared using 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 alcohol;
- 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 insulating layer;
- d) the measurement shall be performed within 10 min of coating the film. When it is needed, more than 10 min before the measurement, the sample shall be kept in the desiccator, keeping the humidity under 10 %.

6.2 Electrical properties

6.2.1 Volume resistivity

6.2.1.1 General

Insulator inks are able to form two types of samples which are capable of measuring the volume resistivity. Two types of samples can be chosen depending on the use case and/or purpose of measurement.

When the volume resistivity is measured against the film which is formed by the insulator ink, the measurement shall be according to IEC 62899-201.

When the sample to be measured is a layer which is prepared by the printing or coating of the insulator ink to a support, the volume resistivity shall also be measured according to IEC 62899-201. However, the measuring electrode described in 6.2.1.2 may be used.

6.2.1.2 Measuring electrode for coating layer

6.2.1.2.1 Substrate and counter electrode

The metal layer shall be formed on the substrate. There are no restrictions with regard to the type of metal and the preparation process, but the resistivity of the metal layer shall be sufficiently lower than the insulating layer to be measured (such as more than 10^{-6} times lower).

Glass may be used as the substrate. The substrate shall have a resistance of more than $10^6 \Omega$ and no dent and distortion. An appropriate size may be used, but unless otherwise specified, a size of (60 to about 100) mm (horizontal length) × (60 to about 100) mm (vertical length) should be used.

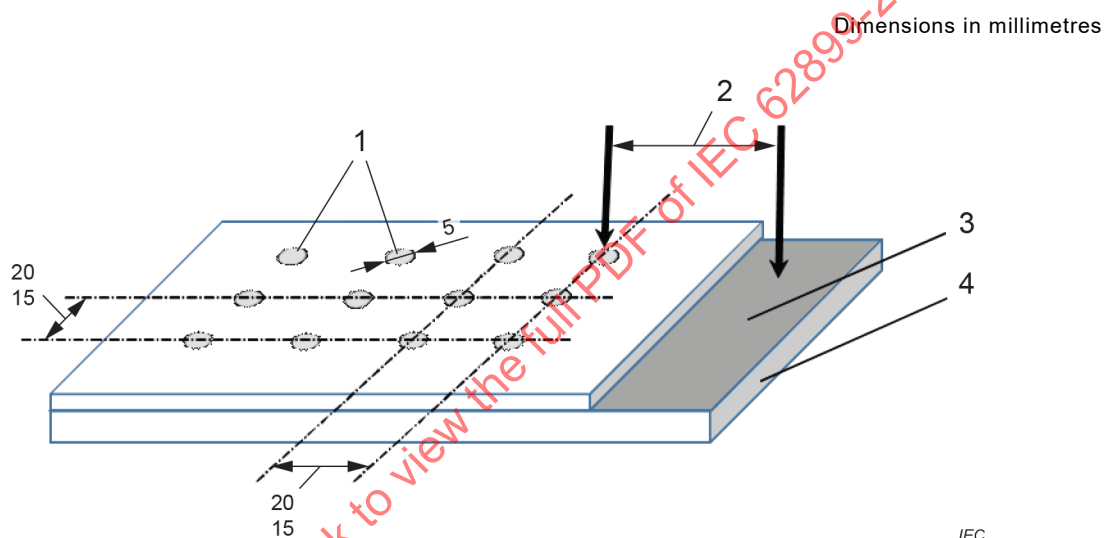
6.2.1.2.2 Insulating layer

The insulating layer for this method shall be prepared by the printing or coating on the metal layer specified in 6.2.1.2.1. It is necessary to secure the contact of the counter electrode; a part of the metal layer should not be covered with the insulating ink.

6.2.1.2.3 Measuring electrode

The measuring electrode shall be prepared so that the space between the circles with a diameter of 5 mm is in the range of 15 mm to 20 mm. There are no restrictions with regard to the type of metal forming the electrode. There are also no restrictions with regard to the preparation process. The requirement for the resistivity of the electrode shall be same as that for the counter electrode in 6.2.1.2.1.

The structure of the measuring electrode, insulating layer, and counter electrode is shown in Figure 2.



Key

- 1 Measuring electrode
- 2 Measured resistance
- 3 Counter electrode (metal layer)
- 4 Substrate

Figure 2 – Example of measuring electrodes

6.2.1.2.4 Measurement and calculation

The resistivity shall be measured between the counter electrode and the measuring electrode. The result shall be calculated using the method specified in IEC 62899-201.

6.2.1.3 Report of the results

The report shall include:

- a) identification of the sample substrate material;
- b) volume resistance and/or surface resistance;
- c) shape and dimensions of the specimen: length × width or diameter (mm), and thickness of insulating layer (μm);
- d) treatment and conditioning of the specimen (application of cleaning and method, conditioning atmosphere, etc.);

- e) test atmosphere (temperature and relative humidity);
- f) applied voltage (V).

6.2.2 Dielectric constant

6.2.2.1 General

The dielectric constant and dielectric loss shall be measured using the method as specified in IEC 62631-2-1 and the other method specified as follows.

6.2.2.2 Definitions

The definitions of the dielectric constant ϵ_0 and dielectric loss $\tan \delta$ are given in IEC 62631-2-1.

6.2.2.3 Preparation of specimens

The test sample shall be prepared as specified in IEC 62631-2-1 or in 6.2.1.2.

6.2.2.4 Measuring method

Methods for measuring the dielectric constant and dielectric tangent can be selected from the null methods or resonance methods described in IEC 62631-2-1.

The method for measuring the capacitance of the sample shall be a parallel plate capacitor method. The capacitor method is described as follows.

The model capacitor consists of two thin parallel conductive plates separated by dielectric constant ϵ . Assuming that the length and width of the plates are much greater than their separation d , the electric field near the centre of the device is uniform with the magnitude $E = \rho/\epsilon$. The voltage is defined as the line integral of the electric field between the plates:

$$V = \int_0^d E dx = \int_0^d \frac{\rho}{\epsilon} dx = \frac{\rho d}{\epsilon} = \frac{Qd}{\epsilon A} \quad (1)$$

Substituting V of Formula (1) in $C = Q/V$ reveals that the capacitance increases with the area of the plates, and decreases as the separation between the plates increases.

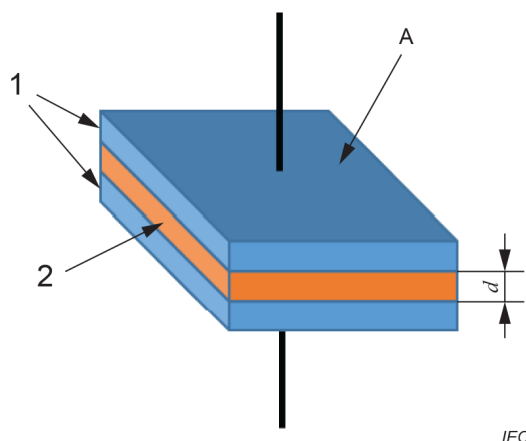
$$C = \frac{\epsilon A}{d} \quad (2)$$

The capacitance is therefore greatest in devices made from materials with a high dielectric constant, large plate area, and small distance between plates.

The results described in IEC 62631-2-1 are for cases with and without the guard-rings as follows. The dielectric constant ϵ_r of a specimen provided with its own electrodes is calculated according to the Formula (3). As the measured capacitance C'_x of a specimen without guard-rings contains a small amount of edge capacitance C_e , the dielectric constant is:

$$\epsilon_r = \frac{C'_x - C_e}{C_0} \quad (3)$$

where: C_0 and C_e can be calculated.



Key

- 1 Conductive plates
- 2 Dielectric
- A Area of the conductive plate
- d Thickness of the dielectric

Figure 3 – Calculation the dielectric constant

The dielectric constant ε_r of an insulator material is the ratio of the capacitance C_x of a capacitor, in which the space between and around the electrodes is entirely and exclusively filled with the insulating material in question, to the capacitance C_0 of the same configuration of electrodes in a vacuum: $\varepsilon_r = \frac{C_x}{C_0}$

The dielectric loss $\tan \delta$ of an insulator material is the tangent of the loss angle δ .

The dielectric loss $\tan \delta$ shall be calculated from the measured values in accordance with the formula given for the particular measuring arrangement used.

6.2.2.5 Test report

In the test report, the following information shall be given when it is relevant:

- a) type and designation of the insulator material as well as the form in which it is delivered. The method of sampling, shape, dimensions of the test specimen and date of sampling (statements on the specimen thickness and, if necessary, exact information on the treatment of the specimens at the contact areas of the electrodes are important);
- b) method and duration of the conditioning of the specimen;
- c) electrode arrangement and type of electrode, applied to the specimen, if any;
- d) measuring apparatus;
- e) temperature and relative humidity during the test and temperature of the specimen;
- f) applied voltage;
- g) applied frequency;
- h) dielectric constant ε_r (average value);
- i) dielectric loss $\tan \delta$ (average value);
- j) date of test.

The values of the relative dielectric constant and dielectric loss and the values calculated from them as loss index and loss angle shall be given, if necessary, in relation to temperature and frequency. Not all are necessary or even appropriate in all cases.

6.2.3 Electric strength

6.2.3.1 General

Electric strength shall be measured using the method as specified in IEC 60243 (all parts) and the other method specified as follows.

6.2.3.2 Preparation of specimen

The test sample shall be prepared as specified in IEC 62631-2-1 or in 6.2.1.2.

6.2.3.3 Measuring method

Electrical measurements are made according to the method employed, by following 6.2.3.5 and 6.2.3.6 and the recommendations of the makers of the equipment used.

6.2.3.4 Insulating layer

The insulating layer for this method shall be prepared by the printing or coating on the metal layer specified in 6.2.1.2.1. It is necessary to secure the contact of the counter electrode; a part of the metal layer should not be covered with the insulating ink.

6.2.3.5 Measuring electrode

The measuring electrode shall be prepared so that the space between the circles with a diameter of 5 mm is in the range of 15 mm to 20 mm. There are no restrictions with regard to the type of metal forming the electrode. There are also no restrictions with regard to the preparation process. The requirement for the resistivity of the electrode shall be same as for the counter electrode in 6.2.1.2.1.

6.2.3.6 Measurement and calculation

A voltage is applied between the main electrode and the counter electrode.

The voltage shall be increased gradually to the designated value (from 5 V to 500 V in 50 V intervals, for example) which depends on the sample property or the customer's demand. Then keep the voltage value constant for 45 s to ensure that the capacitor is charged up properly. If electrical breakdown of the insulating layer occurs, the voltage shall be increased to the next voltage increment. This process shall be continued until the insulating layer has broken or the insulating property has greatly decreased. The test shall be performed three times at different locations, and the average data shall be adopted.

The value of the apparent dielectric strength shall be calculated by the quotient of the breakdown voltage and the thickness of the insulating layer.

6.2.3.7 Report of the results

The report shall include:

- a) identification of the sample substrate material;
- b) breakdown voltage (V);
- c) shape and dimensions of the specimen length times the width or diameter (mm), and thickness of the insulating layer (μm);
- d) treatment and conditioning for the specimen (application of cleaning and method, conditioning atmosphere, etc);

e) test atmosphere (temperature and relative humidity).

6.3 Optical properties

6.3.1 Overview

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

6.3.2 Luminous transmittance

6.3.2.1 General

Luminous transmittance is presented as total luminous transmittance.

6.3.2.2 Measuring method

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

The detailed product specifications shall specify the applicable measuring method.

6.3.2.3 Measuring equipment

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

6.3.2.4 Wavelength or wavelength range used in the test

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

6.3.2.5 Report of the results

The report shall include the following items:

- a) measuring method and equipment;
- b) measuring wavelength or wavelength range;
- c) specimen thickness;
- d) luminous transmittance.

6.3.3 Chromaticity

6.3.3.1 General

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

6.3.3.2 Measuring method

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

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