IEC 62899-202-6

Edition 1.0 2020-12

INTERNATIONAL STANDARD

colour inside

Printed electronics –
Part 202-6: Materials – Conductive ink – Measurement method for resistance changes under high temperature and humidity – Printed conductive layer on a flexible substrate

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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PRINTED ELECTRONICS –

Part 202-6: Materials – Conductive ink – Measurement method for resistance changes under high temperature and humidity – Printed conductive layer on a flexible substrate

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International Standard IEC 62899-202-6 has been prepared by IEC technical committee 119: Printed Electronics.

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

FDIS	Report on voting
119/323/FDIS	119/331/RVD

Full information on the voting for the approval of this International 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 document will remain unchanged until the stability date indicated on the IEC website under "http://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- withdrawn,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 printing process is a highly promising technology for fabricating flexible devices due to its high conductivity and productivity. In particular, a printed conductive layer on a flexible substrate will be widely employed as an electrode or an interconnect for flexible devices. It will be dealt with and commercialized as a sort of composite material in which the conductive layer is formed on the substrate as a conductor.

For conductive films, silver/copper nanowires or metal mesh on flexible substrate are a key component for many recently developed electronic products, from smart phones to the keypads of appliances such as refrigerators and washing machines. Although the conventional material for transparent conductive films is indium tin oxide (ITO), transparent conductive films, enabled by printed electronics technologies, have arisen as a replacement. For application of conductive films, the electrical property under environmental conditions such as temperature, humidity, light, etc., is very important because it is highly sensitive to the environment because of oxidation, dissolution, melting, etc [1] [1]. The conductive films should be stored on a shelf and should be environmentally stable during their operation in electronic devices. Therefore, a method and environment for transferring, storing, and processing the conductivity film are sometimes provided by the supplier and include environmental measurements for printed devices. Although some environmental conditions for testing already exist, the unique characteristics of the printed conductive films should be considered, because they are fabricated on a polymer substrate which is susceptible to temperature, humidity, light, etc., unlike the conventional ITO transparent conductive films.

In this document, an environment reliability test is proposed to evaluate the electrical property and resistance change of a printed conductive film on a flexible substrate.

1 Numbers in square brackets refer to the Bibliography.

PRINTED ELECTRONICS –

Part 202-6: Materials – Conductive ink – Measurement method for resistance changes under high temperature and humidity – Printed conductive layer on a flexible substrate

1 Scope

This part of IEC 62899 provides a method of in-situ measurement for the resistance change of a conductive layer formed by printing methods on a flexible substrate under specified temperature and humidity conditions.

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

IEC 62899-202, *Printed electronics – Part 202: Materials – Conductive ink*

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

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

3.1
conductive ink
ink using metal particles as a main conductive component, in which one or more small molecules, oligomers, polymers, or particles are dissolved or dispersed, and which becomes an electrically conductive layer by post-treatment such as annealing by heating, lighting or chemical treating

Note 1 to entry: Metal particles are specified in IEC 62899-202.

3.2
conductive layer
electrically conductive film-like layer fabricated with conductive ink, which is printed or coated on a substrate, followed as necessary by post-treatment such as annealing by heating, lighting or chemical treating

3.3
flexible substrate
flexible substrate on which conductive ink is printed, such as a plastic film

4 Test sample and measurement equipment

4.1 General

The test sample for environmental testing shall be prepared using a conductive ink on a substrate.

4.2 Sample shape and size for measuring resistance change

A sample with a rectangular shape with a ratio over 20 is recommended.

The tolerance of width and length is 5 % (see IEC 62899-201 and IEC 62899-202).

The recommended sample size is 200 mm × 10 mm.

Another sample size of which the ratio of the longer side over the shorter side is at least 20 may be used for in situ environmental test.

5 Test conditions

5.1 General

The electrical resistance changes of a conductive film shall be measured during the environmental test inside a temperature and humidity controlled chamber. The test shall be measured under atmospheric pressure.

5.2 Test conditions under temperature and humidity

Unless otherwise specified, the test temperature and humidity shall be (85 ± 2) °C and (85 ± 5) %, respectively (see Annex A).

When testing at another temperature and humidity, the tolerance range for the test temperature shall be within ±2 °C and the tolerance for the test humidity shall be within ±5 %.

5.3 Test conditions at high temperature

Unless otherwise specified, test temperature shall be (120 ± 2) °C.

However, the test temperature shall not exceed the glass transition temperature (T_g) of the polymer used in the substrate of the test specimen. When T_g is lower than 120 °C, the test temperature shall be 15 °C lower than T_g in the substrate, with a tolerance of ± 2 °C.

5.4 Test conditions for thermal cycling

Unless otherwise specified, the thermal cycling test shall be carried out at the following temperature cycle:

1) keep at (-20 ± 2) °C for 30 min,
2) increase to (80 ± 2) °C within 30 min,
3) keep at (80 ± 2) °C for 30 min,
4) decrease to (-20 ± 2) °C within 30 min.

Repeat 1) to 4).

The relative humidity shall be kept at (50 ± 5) % throughout the test and condensation on the sample should be avoided.

6 Test method and test apparatus

6.1 General

A printed conductive film is placed in a chamber which can control the temperature and humidity. Place the sample paying attention to the humidity distribution in the chamber.

To measure resistance change, test sample can be placed on a rigid plate and fixed with screws for connection with the measuring equipment, as shown in Figure 1 to Figure 3. The test sample is connected with two or four conductive wires to the measuring equipment.

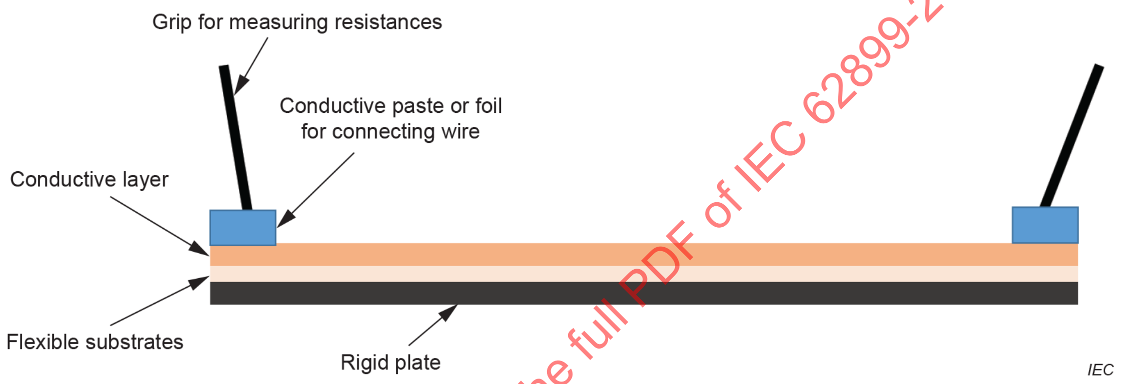

Figure 1 – Schematic diagram of environmental test jig of printed conductive film for two-wire measurement

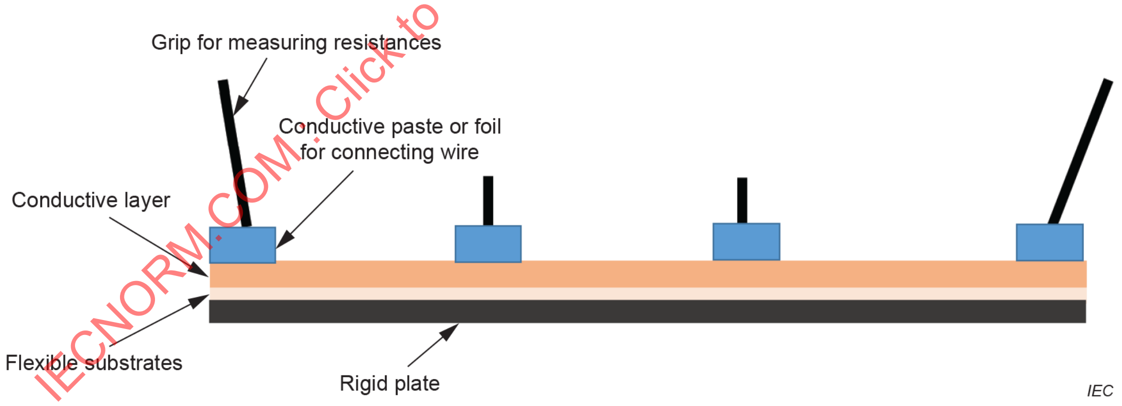

Figure 2 – Schematic diagram of environmental test jig of printed conductive film for four-wire measurement

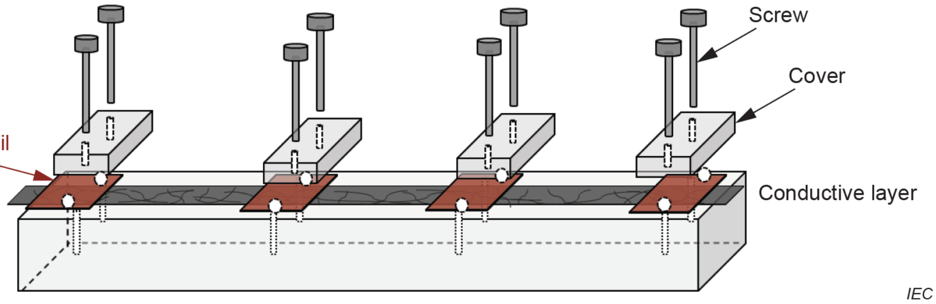

Figure 3 – Schematic diagram of environmental test jig of printed conductive film with screws for a four-wire measurement

NOTE The sample is placed paying attention to the humidity distribution in the chamber.

6.2 Test apparatus

As shown in Figure 1, for the environmental test, the conductive film shall be placed on a plate to reduce the interference from flexibility.

The plates shall be clean, smooth, and rigid to avoid damaging the samples. For the electrical measurement, the grip, shown in Figure 4, shall be made from a conductive metal grip for electrical connection, but the plate shall be made from insulating materials such as resin, polycarbonate, mono cast nylon, etc. The covers and the screws in Figure 1 to Figure 3 shall have the same properties as the plates. The plate and cover shall have a resistance larger than 10^6 Ω, and no deformation shall be allowed during the temperature and humidity test.

Figure 4 – Image of metal grip for connection

All plates and materials shall be stable at a temperature from –30 °C to 150 °C with 90 % relative humidity for the test duration.

6.3 Test procedure

The test procedure is as follows:

a) Prepare the conductive film.

b) Place the conductive film on a rigid plate and grip the sample.

c) Place the sample inside the chamber with controllable temperature and humidity.

d) Connect the two- or four-wire electrical connections for measuring resistance to the measurement equipment, which should be outside of the chamber.

e) Measure the initial resistance.

f) For the test method given in 5.2 to 5.4, set up the temperature and humidity to reach the target temperature and humidity.

g) Measure the resistance.

h) If a cycle is necessary, the temperature and humidity are controlled with the test conditions given in 5.2 to 5.4.

i) The duration time and interval of the collecting data shall be chosen from Table 1.

Table 1 – Options for duration time and interval of collecting data

Option number		**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
For 5.2 and 5.3	Duration time	2 h	16 h	72 h	96 h	168 h	240 h	336 h	1 000 h
	Maximum time interval	30 min	4 h	12 h	24 h	48 h	60 h	72 h	84 h
For 5.4	Duration time	6 h	16 h	24 h	48 h	72 h	96 h		
	Maximum time interval	1 h	1 h	2 h	2 h	2 h	2 h		

j) The termination for the test shall be included in the graph or table of the report. Since the necessary criteria vary greatly depending on the application, it is necessary to agree in advance the final criteria between trade partners. The appropriate duration time shall be set according to the termination criteria. In the case where is no agreement between partners in advance, the duration may be selected from Table 1. The maximum interval of collecting data shall be set so that at least three data measurements shall be made during the test.

6.4 Measurement

The electrical resistance changes of a conductive film shall be measured over time under the environmental test. For a more accurate evaluation, the electrical resistance can be measured periodically under the temperature and humidity test conditions. The electrical resistance of a conductive film shall be measured using the four-wire measurement method with direct current. The conditions for electrical measurement are listed in Table 2.

Table 2 – Resistance range of the test piece and applied current (see IEC 62899-202)

Resistance range of the test piece	< 20 mΩ	< 200 mΩ	< 20 Ω	< 2 kΩ	< 20 kΩ	< 200 kΩ	< 20 MΩ
Applied current	1 A	100 mA	10 mA	1 mA	100 µA	10 µA	1 µA

If the resistance is larger than 1 kΩ, the two-wire measurement method can also be used, because the impact of the contact problem can be considered small and the change of electrical resistance is more important in this test than the absolute value.

Using the metal grips, the electrical resistance of the conductive film can be measured in-situ under environmental conditions.

The measurement shall be based on IEC 62899-202 and IEC 62899-202-5.

7 Data analysis

7.1 Reporting the electrical property

The electrical resistance shall be measured as a function of time at given environmental conditions.

The initial value (R_0) and the relative ratio of the resistance change ($\Delta R/R_0$) of the electrical resistance shall be reported:

$$\Delta R/R_0 = \left(R - R_0\right)/R_0 \tag{1}$$

where

ΔR is the change of electrical resistance;

R is the resistance after the known time;

R_0 is the initial resistance before the environmental test.

The initial value (R_0) is required to report the absolute electrical property of the conductive film and to avoid the confusion resulting from the normalized process. The relative ratio ($\Delta R/R_0$) is an important factor in electronic devices to quantify the long-term reliability.

7.2 Reporting of results

The report shall include the following items:

a) specimen identification (including film thickness, sample size)
b) test condition
c) test specification
d) number of cycles
e) temperature
f) humidity
g) resistance

Annex A
(informative)

An example of environmental test

1) Sample size: 200 mm × 10 mm (see Figure A.1).

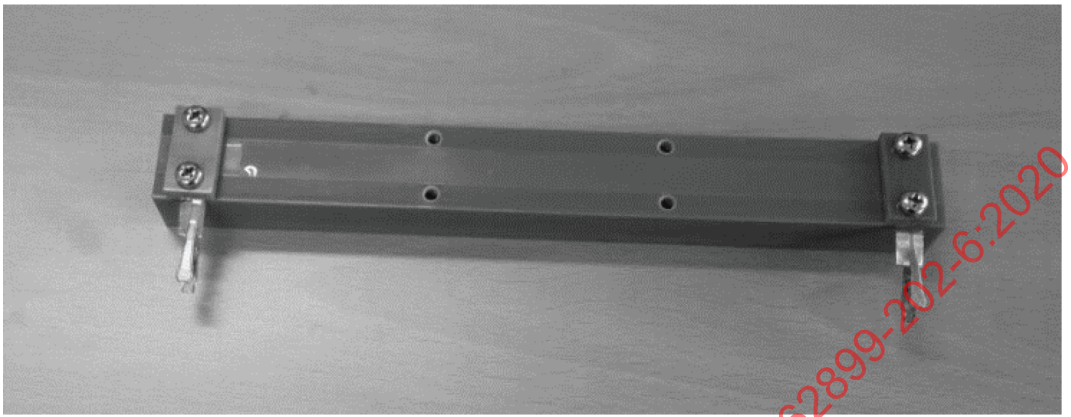

Figure A.1 – Image of environment reliability test sample

2) Test condition (see Figure A.2):
   temperature 85 °C, humidity 85 %.
3) Test result (see Table A.1).

Table A.1 – Test result

Time (h)	Temperature (°C)	Humidity (% RH)	Resistance (Ω)	Resistance change (%)
0	85	86,2	1036	0
1	85	85,2	1025,5	-1,0
2	85	85,2	1046,5	1,0
4	85	87	927,5	-10,5
5	85	85	889	-14,2
6	85	84,5	836,5	-19,3
24	85	85	1001	-3,4
48	85	84,6	1176	13,5
72	85	84	1074,5	3,7
96	85	85,1	1281	23,6
120	85	88,8	983,5	-5,1
144	85	85,1	1046,5	1,0
168	85	85,8	1053,5	1,7
192	85	85,3	1071	3,4
216	85	85,3	1088,5	5,1
240	85	85,5	1928,5	86,1
264	85	85,4	1589	53,4
288	85	85,3	1165,5	12,5
312	85	85,3	1123,5	8,4
336	85	85,3	1305,5	26,0
360	85	85,4	1487,5	43,6
384	85	85,4	1529,5	47,6
408	85	85,4	1557,5	50,3
432	85	85,4	1613,5	55,7
456	85	85,4	1659	60,1
480	85	85,5	1802,5	74,0
504	85	85,5	1830,5	76,7
528	85	85,5	1953	88,5
552	85	85,6	2201,5	112,5
576	85	85,6	2135	106,1
600	85	85,6	2135	106,1
624	85	85,5	2086	101,4
648	85	85,5	2030	95,9
672	85	85,5	2016	94,6
696	85	85,7	2730	163,5
720	85	85,7	2695	160,1

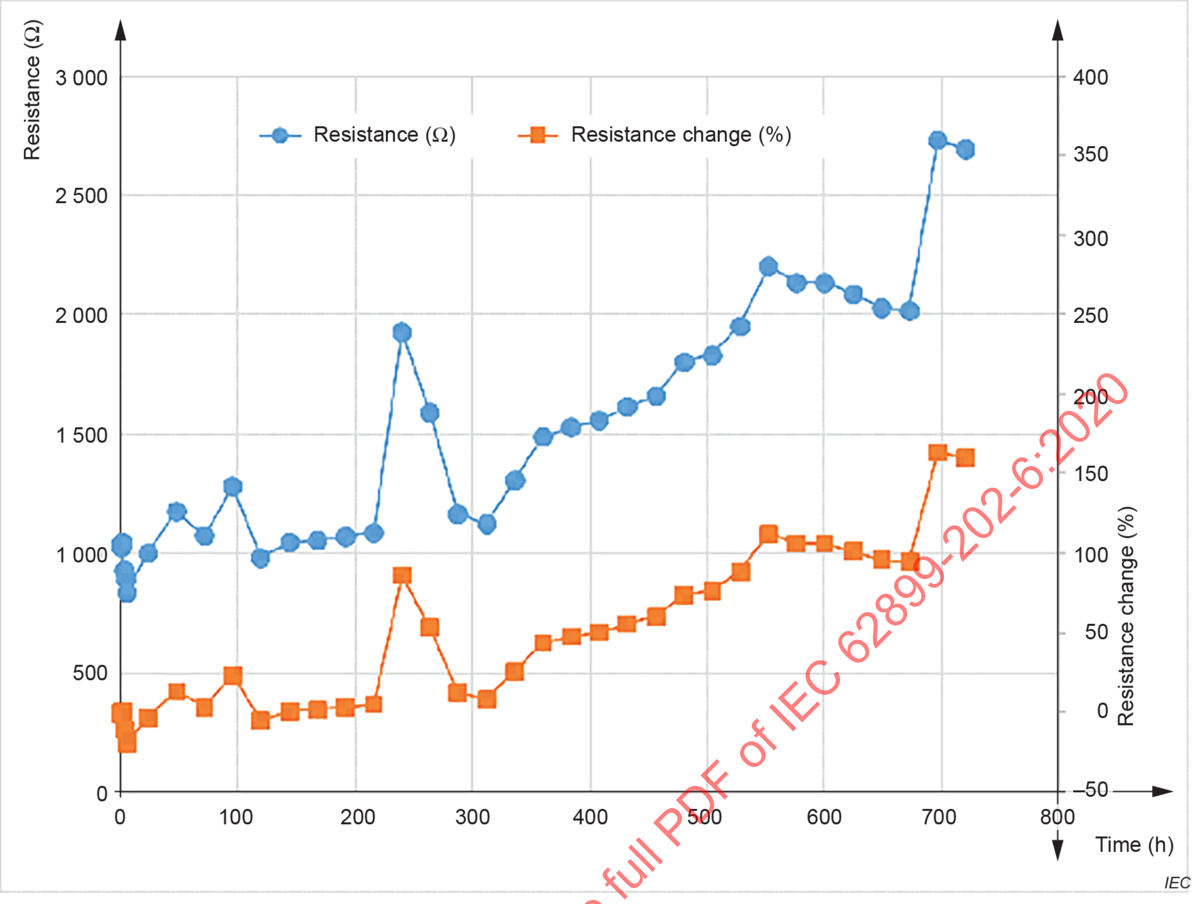

Figure A.2 – Resistance change at a temperature of 85 °C and a humidity of 85 %