IEC 62951-7

Edition 1.0 2019-02

INTERNATIONAL STANDARD

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**Semiconductor devices – Flexible and stretchable semiconductor devices –
Part 7: Test method for characterizing the barrier performance of thin film encapsulation for flexible organic semiconductor**

**Dispositifs à semiconducteurs – Dispositifs à semiconducteurs souples et extensibles –
Partie 7: Méthode d'essai pour caractériser la performance des barrières en couches minces utilisées pour l'encapsulation des semiconducteurs organiques souples**

IEC 62951-7:2019-02(en-fr)

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SEMICONDUCTOR DEVICES –
FLEXIBLE AND STRETCHABLE SEMICONDUCTOR DEVICES –

Part 7: Test method for characterizing the barrier performance of thin film encapsulation for flexible organic semiconductor

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

FDIS	Report on voting
47/2533/FDIS	47/2542/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 62951 series, published under the general title *Semiconductor devices – Flexible and stretchable semiconductor devices*, 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- withdrawn,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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SEMICONDUCTOR DEVICES – FLEXIBLE AND STRETCHABLE SEMICONDUCTOR DEVICES –

Part 7: Test method for characterizing the barrier performance of thin film encapsulation for flexible organic semiconductor

1 Scope

This part of IEC 62951 specifies evaluation conditions and gives a method of measurement as well as a test set-up for the measurement of barrier performance for thin-film layer with ultra-low permeation rate under both flat and bending conditions. This document also includes the preparation of specimen, electrical contacts, sensor films and calculation procedures. For these purposes, this document provides terms, definitions, symbols, configurations, and test methods including test conditions such as temperature, relative humidity, testing time.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of 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
water vapour transmission rate
WVTR
amount of water vapour transmitted through unit area of test specimen per unit time under specified conditions

Note 1 to entry: Its unit is generally g/m^2 day or g/(m^2·24 h) and it means how much water vapour permeates through a 1 m by 1 m square for 1 day.

[SOURCE: ISO 15106-1:2003, 3.1, modified – the note has been expanded.]

3.2
four-wire resistance measurement
electrical resistance measuring technique that uses separate pairs of current-carrying and voltage-sensing electrodes to make more accurate measurements than the simpler and more usual two-terminal sensing

3.3
organic thin film transistor
OTFT
thin film transistor fabricated by using an organic semiconducting material

Note 1 to entry: This note applies to the French language only.

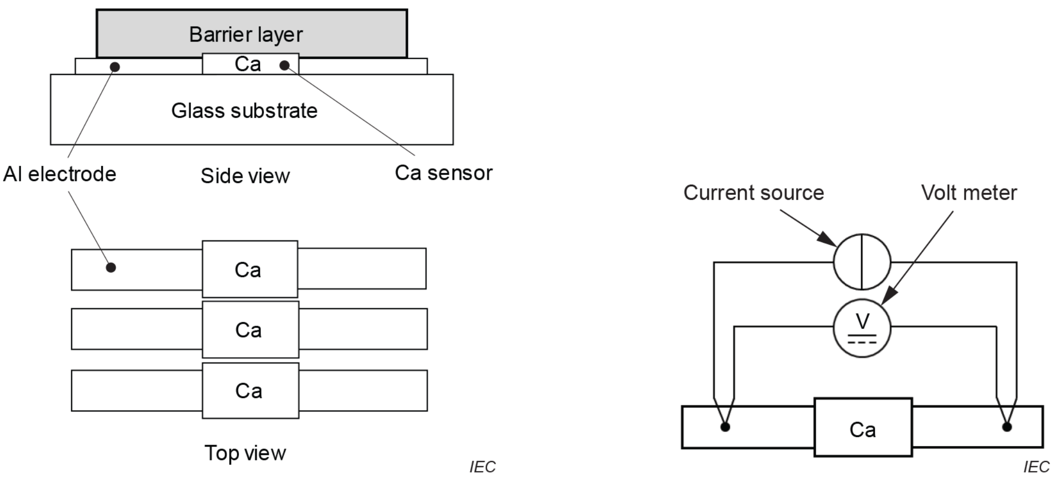
4 Test method

4.1 General

In the case of organic semiconductor devices, one of the main reliability issues is their susceptibility to water vapour. Hence, a high performance encapsulation layer is needed to suppress the ingress of water vapour into devices. The requirement of these barrier layers is less than 10^{-4} g/m^2 day to provide enough protection for organic devices such as OTFTs and organic diodes from the operating environment. Commercially available and published standard for WVTR measurement is only available for a barrier having 10^{-3} g/m^2 day or 10^{-4} g/m^2 day. Hence, this cannot detect the ultra-low permeation of water vapour. This document covers the measurement method for ultra-low permeation rate and general test set-up. The WVTR can be measured if the change of resistance of the Ca sensor is measured. In addition, WVTR under bending conditions needs to be measured for flexible organic electronics.

4.2 Experimental apparatus

Due to the detection limit, the test method based on the infra-red sensor for water vapour transmission rate in the published standard cannot be applied to barrier layer materials for organic semiconductors. Ca test is a widely known and used method for measuring ultra-low permeation rate of the thin-film barriers. Calcium is an electrically conducting and optically opaque metal which becomes non-conducting and transparent after oxidation by humidity. Hence, the measurement of Ca conductance or transparency changes provides an indirect method to determine the oxidation and corrosion rates of Ca induced by permeated water vapour through the barrier layer. In order to measure the electrical conductance change as a function of time, the schematics in Figure 1 a) and b) are suggested. Metal electrodes such as aluminium and Ca sensor are fabricated on the impermeable substrate against water vapour. The dimension including the thickness of the Ca sensor and electrode shall be changed according to the applications. The fabrication of each layer is described in Annex A as an example. The barrier layer which is used to measure WVTR is fabricated on top of the Ca sensor and electrodes. Figure A.2 in Annex A shows typical conductance change as a function of time measured in the test set-up illustrated in Figure 1.



a) Side and top view of Ca test cell and Al electrical contacts

b) Top view of Ca test cell and four-wire resistance measurement

Figure 1 – Experimental set-up for measuring resistance by the four-wire resistance measurement method

4.3 WVTR calculation

By using the experimental set-up described, the change of electrical resistance can be measured by the four-wire resistance measurement method. An example of data can be found in Annex A. The WVTR can be determined by monitoring the time rate of change of the electrical conductance of Ca (data in Figure A.2 in Annex A) through the use of Formula (1) which has been widely used.

$$WVTR = n\delta_{\mathrm{Ca}}\rho_{\mathrm{Ca}} \frac{dG}{dt} \frac{l}{w} \frac{M_{\mathrm{H_2O}}}{M_{\mathrm{Ca}}} \frac{A_{\mathrm{Ca}}}{A_{\mathrm{w}}} \quad (1)$$

where

n is the molar equivalent of the reaction (assumed as n = 2);

δ_{Ca} is the calcium resistivity ($3{,}4 \times 10^{-8}$ Ω m);

ρ_{Ca} is the calcium density (1,55 g/cm^3);

G is the electrical conductance of calcium (the value of $\frac{dG}{dt}$ in Formula (1) is from the linear fitting in the conductance change versus time as shown in Figure A.2 in Annex A);

M is the molar mass ($M_{\mathrm{H_2O}}$ and M_{Ca} are the molar masses of water vapour and of Ca, respectively);

l is the length of the calcium sensor;

w is the width of the calcium sensor;

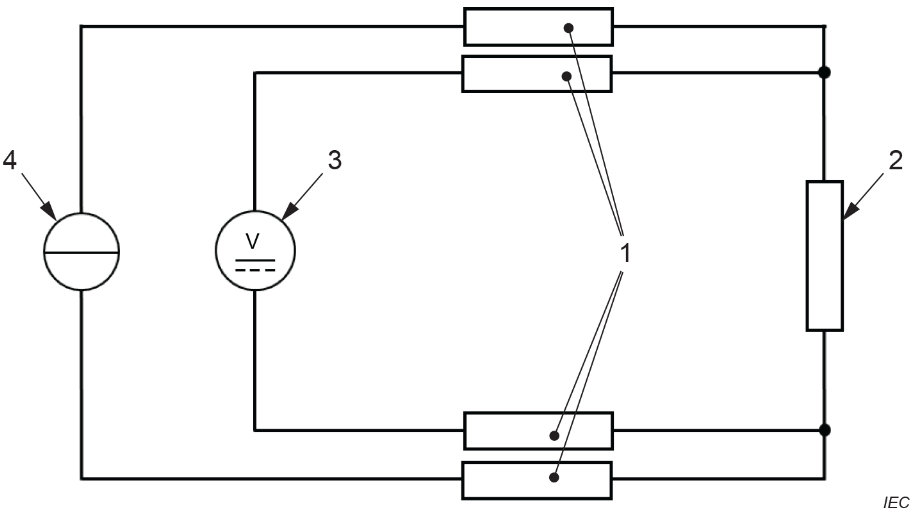
A_{ca} is the area of the Ca sensor;

A_{w} is the area of the window.

The ratio of the area of the Ca sensor to the area of the window for water permeation and the ratio of the length is unity due to the geometry of the experimental set-up.

4.4 Measurement of Ca resistance

In order to measure the electrical conductance change accurately, the four-wire resistance measurement method is suggested to measure the resistance change, as shown in Figure 2. The four-wire resistance measurement method is an electrical impedance measuring technique that uses separate pairs of current-carrying and voltage-sensing electrodes to make more accurate measurements than the simpler and more usual two-terminal (2T) sensing. Due to imperceptible change in resistance during the Ca test, the four-wire resistance measurement method should be used.



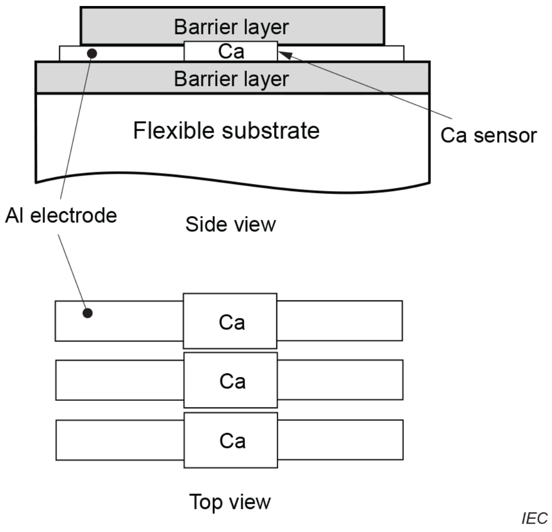
Key

1. resistance of wire;
2. resistance of Ca sensor;
3. volt meter;
4. current source.

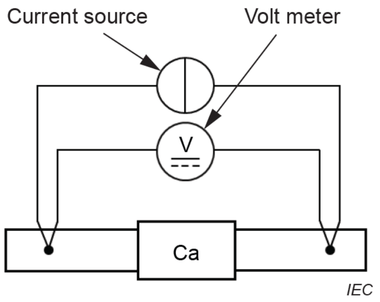
Figure 2 – Four-wire resistance measurement method for measuring resistance change

4.5 Application to flexible organic semiconductor devices

Subclauses 4.2 to 4.4 explain the basic concept of the experimental apparatus for measurement of permeated water vapour through thin film barrier layer in one direction. Subclause 4.5 is about the experimental apparatus for measurement of permeated water vapour through both sides as shown in Figure 3. Due to the limitation of glass substrate to flexible applications, flexible substrates such as PET (polyethylene terephthalate) have been widely used. However, most of flexible substrates are permeable to water vapour. Hence, in order to decrease the amount of permeated water vapour through bottom substrate, a barrier layer should be deposited on top of the flexible substrate, as illustrated in Figure 3. Water vapour can permeate through the top barrier layer as well as the bottom flexible substrate with a barrier layer. The measurement procedure for resistance change is identical to that described in 4.2 to 4.4.



a) **Side and top view of Ca test cell and Al electrical contacts for flexible substrate**

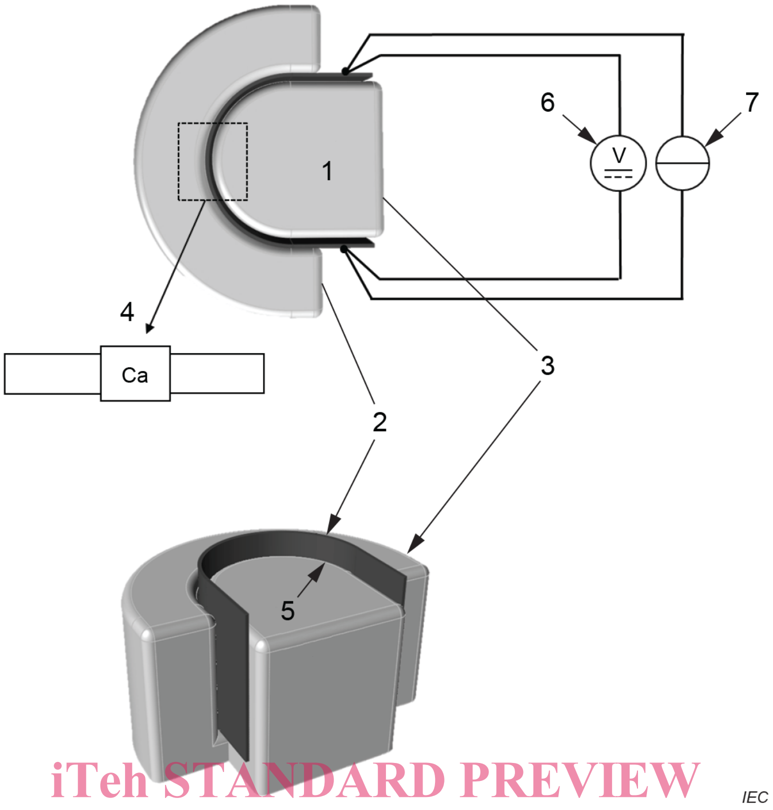


b) **Top view of Ca test cell and four-wire resistance measurement**

Figure 3 – Experimental set-up for measuring resistance by the four-wire resistance measurement method for flexible substrate

4.6 Measurement of barrier performance under the bending condition

Subclauses 4.2 to 4.5 explain the basic concept of the experimental apparatus for measurement of permeated water vapour through both the thin film barrier layer and the flexible substrate. Subclause 4.6 is about the experimental apparatus for measurement of permeated water vapour through both sides under the bending condition as shown in Figure 4. Due to the actual application of flexible electronics, the barrier performance needs to be measured under the bending condition. By using the experimental apparatus described in Figure 4, the radius of the curvature for the thin film barrier layer can be precisely realized.



Key

1 bending radius

2 flexible barrier layer

3 bending test jig

4 Ca sensor

5 bending radius

6 volt meter

7 current source

Figure 4 – Experimental set-up for measurement of barrier performance under the bending condition

5 Test report

It should be noted that the value of WVTR is significantly dependent on the test environments such as temperature and relative humidity. Hence, it is noted that the value of WVTR should be reported with the test environment. In this document, it is suggested that the test should be carried out either in a controlled environmental chamber or under standard laboratory conditions. The following information shall be included in the test report:

a) reference to IEC 62951-7

b) the shape and the dimensions of experimental apparatus

- Ca sensor
- electrode
- barrier layer