## INTERNATIONAL STANDARD

ISO 21760-1

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# Adhesives for organic electronic devices — Determination of water vapour transmission rate —

## Part 1: **Adhesive film preparation methods**

Teh STAdhésifs pour dispositifs électroniques organiques — Détermination du taux de transmission de vapeur d'eau —

Partie 1: Méthodes de préparation du film adhésif

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#### **Foreword**

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see <a href="www.iso.org/directives">www.iso.org/directives</a>).

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The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

A list of all parts in the ISO 21760 series can be found on the ISO website.6-4d1a-ad96-

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Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <a href="https://www.iso.org/members.html">www.iso.org/members.html</a>.

## Adhesives for organic electronic devices — Determination of water vapour transmission rate —

#### Part 1:

### Adhesive film preparation methods

#### 1 Scope

This document specifies six methods for determining the water vapour transmission rate of adhesive films coated on a plastic substrate.

The adhesive is used in organic electronic devices such as organic light-emitting diodes.

The methods provide rapid measurement over a wide range of water vapour transmission rates.

#### 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.

(Standards.iten.al)

ISO 2808, Paints and varnishes — Determination of film thickness

ISO 4593, Plastics — Film and sheeting — Determination of thickness by mechanical scanning https://standards.itel.avcatalog/standards/sist/4cc/0d91-0736-4d1a-ad96-

ISO 15106 (all parts), Plastics — Film and sheeting 44 Determination of water vapour transmission rate

#### 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:

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

#### 3 1

#### water vapour transmission rate

amount of water vapour transmitted per unit area of *test specimen* (3.2) per unit time under specified conditions

Note 1 to entry: It is expressed in grams per square metre per 24 h [g/( $m^2 \times 24$  h)].

#### 3.2

#### test specimen

supporting substrate with coating of adhesives applied to it

#### 4 Principle

A test specimen consists of a non-self-supporting adhesive film on a plastic substrate. The test specimen is mounted in a transmission cell forming a sealed barrier between two chambers.

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The upstream chamber is usually the wet side and the downstream chamber is usually the dry side.

The amount of water vapour transmitted through the specimen is detected by a sensor capable of providing an electrical output signal.

Since the water vapour transmission rate of a non-self-supporting adhesive cannot be measured directly, it is necessary to measure the water vapour transmission rate of the adhesive plus substrate and the substrate alone.

When calculating the water vapour transmission rate of a non-self-supporting adhesive, the water vapour transmission rate of the substrate shall be taken into account. The reciprocal number of the water vapour transmission rate of the adhesive is obtained by subtracting that of the substrate alone from that of the substrate plus adhesive.

Materials such as a light-emitting layer in the flexible organic electronic device are degraded by the penetration of a very small amount of water vapour. Both the plastic substrate and adhesive film (seal material) are required to have high barrier properties. The method for determining the water vapour transmission rate of plastic substrates is specified in accordance with one of ISO 15106-1, ISO 15106-2, ISO 15106-3, ISO 15106-4, ISO 15106-5, or ISO 15106-6.

The water vapour transmission rate of self-supporting adhesive film shall be determined in accordance with ISO 15106 (all parts).

#### 5 Test specimens

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5.1 Substrate for non-self-supporting adhesives (standards.iteh.ai)

Any homogeneous material which has a water-vapour transmission rate larger than 1 g/( $m^2 \cdot 24$  h) is suitable for use as the substrate for non-self-supporting adhesives, for instance poly(ethylene terephthalate) film. https://standards.itch.ai/catalog/standards/sist/4cc70d91-0736-4d1a-ad96-

Determine the mean thickness,  $d_s$ , of the substrate using a suitable method selected from those specified in ISO 4593 or ISO 2808.

The thickness of the PET substrate,  $d_s$ , shall be selected from the following values: 12  $\mu$ m, 25  $\mu$ m, 50  $\mu$ m or 100  $\mu$ m, because these are easy to obtain and commercially available.

The substrate should not be porous material.

#### 5.2 Preparation of test specimens

#### 5.2.1 Preparation of non-self-supporting adhesives on a substrate

The substrate shall be clean and dry.

Apply the adhesive material to be tested to the substrate in accordance with the manufacturer's application instruction.

The optimum thickness of the adhesive is dependent on the coating method. The total thickness of the adhesive shall be in the range of 3  $\mu m$  to 60  $\mu m$ . It is recommended to be selected from the following values: 5  $\mu m$ , 10  $\mu m$ , 20  $\mu m$  or 50  $\mu m$ .

Several coating methods are available. These are described in the following annexes:

- Annex A: Spin coating application method;
- Annex B: Blade coating application method;
- Annex C: Spray coating application method.

To improve the barrier properties, not only resins (such as epoxy, urethane and acrylic), but also resins and multi-layered films containing an inorganic filler or desiccant have been used as a seal material. In this case, the same test procedure can be applied.

Methods other than these shall be agreed upon by the interested parties.

#### 5.2.2 Test specimen

The specimens shall be representative of the material, free from wrinkles, creases and pinholes, and have uniform thickness. Each specimen shall have a larger area than the transmission area of the cell.

NOTE Uneven specimens are not suitable due to water vapour leakage.

#### **5.2.3** Number of specimens

Three specimens shall be tested unless otherwise specified or agreed between the interested parties.

#### 6 Conditioning

The specimens shall be conditioned at  $23 \pm 2$  °C and  $50 \pm 5$  %RH for a length of time appropriate to the material under test, unless otherwise agreed between the interested parties.

#### 7 Determination of the thickness of the adhesive

#### iTeh STANDARD PREVIEW

#### 7.1 General

### (standards.iteh.ai)

The thickness, *d*, of the adhesive is not used to calculate the water vapour transmission rate; it is necessary when comparing the transmission/coefficient. It may be determined by calculation or by optical, mechanical operators of the suitable/methods/dards/sist/4cc70d91-0736-4d1a-ad96-

NOTE Optical determination of thickness of the adhesive can also be used to check the test piece for pores, holes, etc., and determine the depth of penetration of the adhesive material into the substrate.

#### 7.2 Determination of the thickness of the adhesives by calculation

Calculate the dry-film thickness, *d*, in micrometres, from the application rate (the amount of adhesive material applied), using <u>Formula (1)</u>:

$$d = \frac{c \times V_{\rm n}}{100} \tag{1}$$

where

- c is the application rate, in millilitres per square metre (cubic centimetres per square metre);
- $V_{\rm n}$  is the non-volatile-matter content, expressed as a percentage by volume, determined in accordance with ISO 3233-1[1].

### 7.3 Determination of the thickness of the adhesive by optical, mechanical or other suitable methods

Determine the mean thickness of the adhesive using a suitable method selected from those specified in ISO 4593 or ISO 2808.

So as not to break the adhesive, it is preferable to determine the thickness after measurement of the water vapour transmission rate. However, if the adhesive swells, it shall be measured first.

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The thickness of any adhesive material shall be determined by optical or other suitable methods.

#### 8 Test methods

Several methods are available for measuring the amount of water vapour permeating through a specimen. Six of these examples are specified in the corresponding ISO 15106 part.

- ISO 15106-1: Humidity detection sensor method (equal pressure method);
- ISO 15106-2: Infrared detection sensor method (equal pressure method);
- ISO 15106-3: Electrolytic detection sensor method (equal pressure method);
- ISO 15106-4: Gas-chromatographic detection sensor method (differential pressure method);
- ISO 15106-5: Pressure sensor method (differential pressure method, high sensitivity);
- ISO 15106-6: Atmospheric pressure ionization mass spectrometer method (equal pressure method, high sensitivity).

If the water vapour transmission rate of a specimen is 1 g/(m $^2 \cdot 24$  h) or more, it is recommended to use the methods of ISO 15106-1, ISO 15106-2 and ISO 15106-4. When it is less than 1 g/(m $^2 \cdot 24$  h), it is preferable to use the methods of ISO 15106-3, ISO 15106-5 and ISO 15106-6.

Fragile test specimens are not suitable to be tested according to ISO 15106-4 and ISO 15106-5.

The method used depends on Teh STANDARD PREVIEW

- the water vapour transmission rate of the material tested; h.ai)
- the sensitivity required;

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— the fragility of the material tested. https://standards.iteh.ai/catalog/standards/sist/4cc70d91-0736-4d1a-ad96-d4572f5db164/iso-21760-1-2019

#### 9 Procedure

#### 9.1 General

Since the water vapour transmission rate of a non-self-supporting adhesive film cannot be measured directly, it is necessary to measure the water vapour transmission rate of the adhesive film plus substrate and the substrate individually.

#### 9.2 Water vapour transmission rate, $W_{AS}$ , of the substrate plus adhesive film

Measure the water vapour transmission rate,  $W_{AS}$ , of the substrate plus adhesive film using a method selected from ISO 15106-1 to ISO 15106-6.

#### 9.3 Water vapour transmission rate, $W_S$ , of the substrate

Measure the water vapour transmission rate,  $W_S$ , of the substrate using a method selected from ISO 15106-1 to ISO 15106-6.

It is preferable to use the same method to measure  $W_S$  and  $W_{AS}$ , if measurement of both  $W_S$  and  $W_{AS}$  is possible.

#### 10 Calculation

#### 10.1 General

When calculating the water vapour transmission rate of a non-self-supporting adhesive film, the water vapour transmission rate of the substrate shall be taken into account. The water vapour transmission rate of the adhesive is obtained by subtracting that of the substrate plus adhesive from that of the substrate alone.

#### 10.2 Water vapour transmission rate, $W_A$ , of non-self-supporting adhesive films

The water vapour transmission rate,  $W_A$ , of the adhesive film is calculated from the difference between the water vapour transmission rate of the substrate,  $W_S$ , and that of the substrate plus adhesive film,  $W_{AS}$ , as shown in Formula (3), which is derived from Formula (2):

$$1/W_{A} = 1/W_{AS} - 1/W_{S} \tag{2}$$

$$W_{\mathbf{A}} = (W_{\mathbf{AS}} \times W_{\mathbf{S}}) / (W_{\mathbf{S}} - W_{\mathbf{AS}}) \tag{3}$$

where

 $W_A$  is the water vapour transmission rate of the adhesive film, expressed in grams per square metre per 24 h [g/(m<sup>2</sup>×24 h)]; DARD PREVIEW

 $W_{AS}$  is the water vapour transmission rate of the substrate plus adhesive film, expressed in grams per square metre per 24 h [g/(m<sup>2</sup> × 24 h)];

Ws is the water vapour transmission rate of the substrate, expressed in grams per square metre per 24 h [g/(m²axi24bì)]h.ai/catalog/standards/sist/4cc70d91-0736-4d1a-ad96-d4572f5db164/iso-21760-1-2019

#### 11 Test condition

The test conditions should preferably be chosen from those given in <u>Table 1</u>.

**Test conditions Temperature Relative humidity**  $^{\circ}C$ %  $25 \pm 0.5$  $90 \pm 3$ 1 2  $40 \pm 0.5$  $90 \pm 3$ 3  $60 \pm 0.5$  $90 \pm 3$ 4  $85 \pm 0.5$  $85 \pm 3$ 

Table 1 — Choice of test conditions

Test conditions other than these shall be agreed upon by the interested parties.

#### 12 Test result

Express the test result as the arithmetic mean of the results obtained for each test specimen, rounding to two significant figures.

If the measured value of the specimens is near the background value, both values shall be reported.