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

Part 1: Adhesive film preparation methods

Adhésifs 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

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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.

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 www.iso.org/members.html.

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.

ISO 2808, *Paints and varnishes — Determination of film thickness*

ISO 4593, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning*

ISO 15106 (all parts), *Plastics — Film and sheeting — 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 https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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 × 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.

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

5.1 Substrate for non-self-supporting adhesives

Any homogeneous material which has a water-vapour transmission rate larger than 1 g/(m^2 · 24 h) is suitable for use as the substrate for non-self-supporting adhesives, for instance poly(ethylene terephthalate) film.

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 µm, 25 µm, 50 µm or 100 µm, 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 µm to 60 µm. It is recommended to be selected from the following values: 5 µm, 10 µm, 20 µm or 50 µm.

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 ± 2 °C and 50 ± 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

7.1 General

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 or other suitable methods.

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 Formula (1):

$$d = \frac{c \times V_n}{100} \tag{1}$$

where

c is the application rate, in millilitres per square metre (cubic centimetres per square metre);

V_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.

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 · 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 · 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:

— the water vapour transmission rate of the material tested;

— the sensitivity required;

— the fragility of the material tested.

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):

$$1/W_A = 1/W_{AS} - 1/W_S \tag{2}$$

$$W_A = (W_{AS} \times W_S)/(W_S - W_{AS}) \tag{3}$$

where

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

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^2 × 24 h)];

W_S is the water vapour transmission rate of the substrate, expressed in grams per square metre per 24 h [g/(m^2 × 24 h)].

11 Test condition

The test conditions should preferably be chosen from those given in Table 1.

Table 1 — Choice of test conditions

Test conditions	Temperature °C	Relative humidity %
1	25 ± 0,5	90 ± 3
2	40 ± 0,5	90 ± 3
3	60 ± 0,5	90 ± 3
4	85 ± 0,5	85 ± 3

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.

13 Precision

The precision of this test method is not known because interlaboratory data are not available at the time of publication.

14 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 21760-1:2019;

b) the test conditions;

c) the name of the apparatus used for W_S with a reference to the relevant part of ISO 15106;

d) the name of the apparatus used for W_{AS} with a reference to the relevant part of ISO 15106;

e) all details necessary for identification of the sample tested;

f) the method of preparation of the test specimens;

g) the side of the specimen which faced the supply of water vapour, if necessary;

h) the transmission area of the specimens;

i) the mean thickness of the adhesive tested;

j) the type of substrate (including the primer, if used), its average thickness d_S and its water vapour transmission rate W_S;

k) the number of specimens tested;

l) details of specimen conditioning;

m) the test result (water vapour transmission rate of the adhesive film W_A, water vapour transmission rate of the adhesive film plus substrate W_{AS},);

n) the date of the test (or the period of the test).

Annex A
(informative)

Spin coating application methods

A.1 General

Spin coating is a useful application when it is desirable to coat only one side of a film. Spin coating is typically used for small or medium-sized film that is flat and symmetrical in shape.

Typical coating thicknesses range from 200 nm to 25 µm.

A.2 Apparatus

The basic apparatus for spin coating consists of a spinning device, a coating delivery device, a collection tank/reservoir, a pump, and a filtering element.

A.3 Procedure

A.3.1 The coating thickness is a function of the coating solids content, solvent composition, temperature and viscosity.

In a spin coating application, it is generally most effective to use the coating solids content (or viscosity) and spin rate to make adjustments to the coating thickness, while keeping the other coating properties and environmental factors constant.

A.3.2 The liquid solution is applied to the substrate either during slow spinning or with a stationary sample.

A.3.3 The sample is rorated at a slow spin speed to distribute the solution.

A.3.4 The rotation speed is increased to give the desired thickness.

A.3.5 The rotation of the substrate should be maintained while the solvents evaporate.

Annex B
(informative)

Blade coating application methods

B.1 General

Blade coating, also known as knife coating or doctor blading, is a processing method for fabrication of large area films on rigid or flexible substrates. The well-defined thickness is mainly controlled by the gap size between the blade and the surface. For lab-scale processing, the blade is moved over a flat surface. For large-scale roll to roll processes, the blade is fixed above the moving surface.

B.2 Apparatus

The apparatus for lab-scale blade coating consists of a film applicator with a blade, a pushing machine and a waste plate.

B.3 Procedure

B.3.1 The substrate film is layed on the glass bed of the pushing machine as flat as possible.

B.3.2 The film applicator is placed on the substrate film.

B.3.3 Both micrometre screws on the film applicator is adjusted to control the gap between the blade and the surface of the substrate.

B.3.4 The coating solution is poured onto the substrate.

B.3.5 The coating speed is set before running the pushing machine.

B.3.6 The applicator is pushed by the pushing machine to distribute the coating solution on top of the substrate with a constant thickness.