IEC 62788-6-2

Edition 1.0 2020-03

INTERNATIONAL STANDARD

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Measurement procedures for materials used in photovoltaic modules – Part 6-2: General tests – Moisture permeation testing of polymeric materials

Procédures de mesure des matériaux utilisés dans les modules photovoltaïques – Partie 6-2: Essais génériques – Essais de perméation à l'humidité des matériaux polymères

IEC 62788-6-2:2020-03(en-fr)

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FOREWORD

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

FDIS	Report on voting
82/1659/FDIS	82/1690/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 62788 series, published under the general title *Measurement procedures for materials used in photovoltaic modules*, 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- withdrawn,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

This part of IEC 62788 describes methods to measure the permeation properties of polymeric materials. The degradation of PV modules is known to go through many different corrosion processes. These degradation processes may depend upon moisture ingress into the encapsulant, edge seal, frontsheet, or backsheet materials. Typical polymeric materials used include (amongst other polymers) ethylene-vinyl acetate (EVA) and polyolefins for encapsulants, polyisobutylene (PIB) for edge seals, and polyethylene terephthalate (PET), polyvinyl fluoride (PVF), or polyvinylidine fluoride (PVDF) for backsheets. Therefore, knowing the moisture permeation characteristics of polymeric materials is relevant for module design. These properties can be determined as a function of temperature and relative humidity. With these parameters, simple scaling rules for time and distance can be used to extrapolate to the use environments.

MEASUREMENT PROCEDURES FOR MATERIALS USED IN PHOTOVOLTAIC MODULES –

Part 6-2: General tests – Moisture permeation testing of polymeric materials

1 Scope

This document provides methods for measuring the steady-state water vapour transmission rate (WVTR), water vapour permeability (P), diffusivity (D), solubility (S), and moisture breakthrough time (T_{10}) (defined as the time to reach 10 % of the steady state WVTR) for polymeric materials such as encapsulants, edge seals, frontsheets and backsheets. These measurements can be made at selected temperatures and humidity levels as deemed appropriate for evaluation of their performance in PV modules. Measurement is accomplished by inspection of the transient WVTR curve and by fitting it to a theoretical Fickian model. This document is best applied to monolithic films. If multilayer films are used, the D and S values are only apparent values, but the steady-state values can still be measured.

This document was written for the measurement of water permeation, but it can equally be used for other permeants such as O_2. In this case the same diffusion equations, fitting procedures, and scaling arguments are used.

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 TS 61836, *Solar photovoltaic energy systems – Terms, definitions and symbols*

ISO 2528, *Sheet materials – Determination of water vapour transmission rate (WVTR) – Gravimetric (dish) method*

ISO 9932, *Paper and board – Determination of water vapour transmission rate of sheet materials – Dynamic sweep and static gas methods*

ISO 15106-1, *Plastics – Film and sheeting – Determination of water vapour transmission Rate – Part 1: Humidity detection sensor method*

ISO 15106-2, *Plastics – Film and sheeting – Determination of water vapour transmission Rate – Part 2: Infrared detection sensor method*

ISO 15106-3, *Plastics – Film and sheeting – Determination of water vapour transmission Rate – Part 3: Electrolytic detection sensor method*

ISO 15106-4, *Plastics – Film and sheeting – Determination of water vapour transmission Rate – Part 4: Gas-chromatographic detection sensor method*

ASTM F1249-06, *Standard test method for water vapour transmission rate through plastic film and sheeting using a modulated infrared sensor*

3 Terms, definitions and symbols

For the purposes of this document, the terms and definitions the terms and definitions given in IEC TS 61836 and the following 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 Terms and definitions

3.1.1
edge seal
polymeric material designed to be placed between two impermeable (or with extremely low permeability) frontsheet and backsheet materials to restrict moisture ingress from the sides

3.1.2
Fickian
material for which the diffusivity is constant, independent of concentration of the permeant within the experimental uncertainty

3.1.3
permeability
state or quality of a material or membrane that causes it to allow liquids or gases to pass through it

3.1.4
diffusivity
measure of the capability of a substance to be diffused or to allow something to pass by diffusion

3.2 Symbols

T	temperature [°C] or [K]
t	time from application of moisture or the start of the experiment [h]
T_{delay}	instrumental delay time [h]
T_{10}	time for WVTR to reach 10 % of its steady-state value [h]
$T_{1/2}$	time for WVTR to reach 50 % of its steady-state value [h]
l	sample thickness [mm]
H	relative humidity [%]
T_R	water vapour transmission rate [$g \cdot m^{-2} \cdot day^{-1}$]
P	water permeability [$g \cdot mm \cdot m^{-2} \cdot day^{-1}$]
D	water diffusivity [$cm^2 \cdot s^{-1}$]
S	water solubility [$g \cdot cm^{-3}$]
K_{10}	moisture ingress breakthrough constant [$cm \cdot h^{-0,5}$]
P_o	Arrhenius permeability prefactor [$g \cdot mm \cdot m^{-2} \cdot day^{-1}$]
Ea_P	Arrhenius permeability activation energy [$kJ \cdot mol^{-1}$]
D_o	Arrhenius diffusivity prefactor [$cm^2 \cdot s^{-1}$]
Ea_D	Arrhenius diffusivity activation energy [$kJ \cdot mol^{-1}$]

S_o Arrhenius solubility prefactor [g·cm^{-3}]

Ea_S Arrhenius solubility activation energy [kJ·mol^{-1}]

$K_{o,10}$ Arrhenius moisture ingress breakthrough constant prefactor [cm·h$^{-0,5}$]

Ea_{K10} Arrhenius moisture ingress breakthrough constant activation energy [kJ·mol^{-1}]

4 Apparatus

Any instrument capable of measuring the transient permeation through a membrane shall be used. Many examples of apparatus that are suitable for these measurements are described in ASTM F1249-06, ISO 2528, ISO 9932, ISO 15106-1, ISO 15106-2, ISO 15106-3, and ISO 15106-4. The key characteristics to look for are high precision and small timescales for the response to changes. At low permeation rates, the amount of moisture adsorbed onto instrument surfaces becomes a problem, because it limits the useful precision of some instruments.

5 Test specimens

The suitable polymer film sample thickness will depend on the transient WVTR and the instrumental setup. Typically, samples are between 0,25 mm and 2 mm thick, but other thicknesses may be used. Very low-permeability materials, such as polychlorotrifluoroethylene (PCTFE) may require much thinner films so that permeated water is detectable at low temperatures. Conversely, high-diffusivity materials (such as silicones) may need to be several millimetres thick to accurately separate the lag time, associated with transient diffusion in the polymer, from the instrumental delay. For very thick samples, care shall be taken to assess and/or minimize moisture ingress from, or egress to, the external environment from the sides of the sample. Thickness variation shall be less than ±5 % over the sample area of interest. The suggested sample test area is between 5 cm^2 to 100 cm^2, but other areas may be used if desired.

NOTE Masks can be provided by permeation equipment manufacturers and are typically made of an Al foil with an adhesive on one side. These masks are typically designed to reduce the transmission area from 50 cm^2 to 5 cm^2.

Samples shall be processed or cured (if applicable) in accordance with the manufacturer's specification. Verify that the sample does not change during measurement through loss of volatiles or other chemical degradation processes in a way that could change the permeation properties relative to the intended use environment. This can be verified by comparing initial measurements with another set of measurements, made under the same conditions, on the same film, at a later date. If permeation changes with aging, one shall either only use fresh samples or aged samples and note the sample history in the test report.

Sample surfaces shall be smooth and flat with uniform thickness (< 5 % variation in thickness). This can require curing (or thermal treatment) while being held between flat, planar surfaces. Masking the samples or supporting them with a mesh can be necessary during testing at higher temperatures to prevent sag or other deformations of the sample.

Some materials, particularly polyisobutylene-based edge seals, are very tacky and prone to stick to the surfaces of the measurement instrument. In this case, a thin (preferably < 0,05 mm), highly permeable (50 g·m^{-2}·day^{-1}), non–stick supporting film may be used. If a film like this is used, verify that it does not impact the results by measuring films of at least two different thicknesses. If the films do not impact the measurement, the steady-state WVTR will scale as $1/l$ and the transient time should scale as $1/l^2$, where l is the sample thickness.

If a thicker specimen is needed, multiple layers of film may be simply stacked together in the test fixture as long as the air gap between layers is minimized. It is recommended that the laminate be run through a roll laminator or some other means to create good contact between films. This method yields acceptable results because the volume of the air gap is very low, the mole fraction of water in air is typically low, and the diffusivity of moisture in air is very high. If applying this method to other permeants, such as oxygen, small gaps between layers are likely to affect the results. Here, one shall verify the WVTR and the transient time scale as l^{-1} and l^{-2} respectively.

When measuring multilayer laminate films containing layers of different materials (e.g. a backsheet consisting of a fluoropolymer/polyethylene terephthalate/ethylene vinyl acetate), this film layering procedure shall not be used to increase the delay time for moisture ingress to determine diffusivity and solubility. Provided sufficient instrument sensitivity is present in accordance with the restrictions outlined in Clause 7, the steady-state values for WVTR and permeability can still be obtained without layering, but, in some cases, estimates of the apparent diffusivity and solubility might not be possible for a given instrument with the restrictions outlined for the time scales on the transient curves.

6 Procedure

For moisture ingress testing, sample drying in a desiccated atmosphere (RH < 1 %) prior to testing can be used to reduce the equilibration drying time in the instrument. The required drying time and temperature will depend on the sample diffusivity. A drying temperature of 40 °C to 50 °C is recommended for at least 48 h to 72 h for most materials. Thicker samples, or samples with low D, can take longer to dry. For samples such as edge seals with desiccants, drying might not be possible. In this case, the materials shall be used as received while taking precautions to minimize the exposure to moisture prior to testing. While being conditioned, samples should not be stacked flat on top of each other, but air should be allowed to circulate between them. Keep the sample dry until it is placed in the diffusion cell, which has been previously dried by removing residual moisture and/or purging with dry nitrogen or similar dry gas. After placing the sample in the diffusion cell, allow time for equilibration of the diffusion cell to the desired temperature.

Pass dry gas (e.g. N_2) over both sides of the sample to remove any residual moisture. Care should be taken to ensure that the gas is at the desired temperature before passing it over the film. When the cell is within ±0,5 °C of the desired temperature and the WVTR is at a steady state near zero, within the detection limits of the instrument, turn off the external purge gas on the permeant source side and start monitoring moisture permeation on the detector side (see Figure 1).

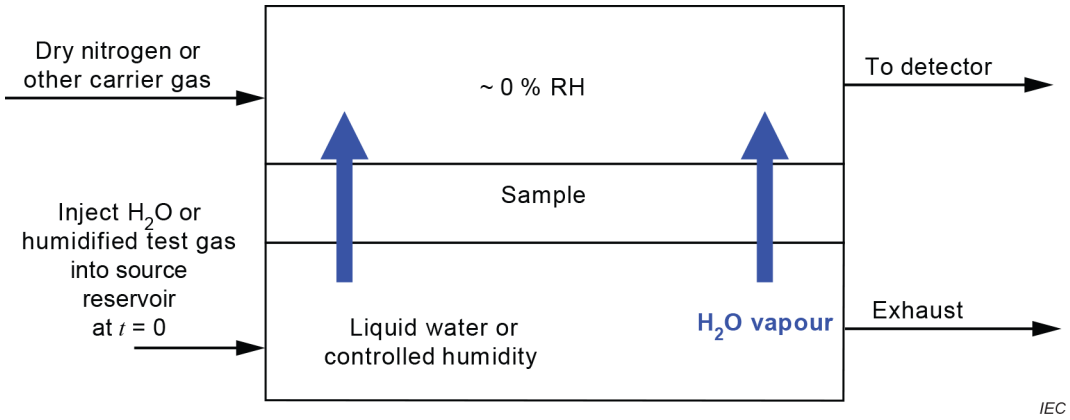

Figure 1 – Diagram of a diffusion cell

For testing at 100 % RH, inject (or otherwise apply moisture as a step change) 3 ml to 10 ml of H_2O into the permeant source chamber of the cell and monitor the WVTR over time noting the time H_2O was added. Water shall not be added to the point of filling up the chamber and contacting the sample. The water should be equilibrated to the desired temperature within ±10 °C. More than 10 ml of water may be necessary for larger films so that there is enough liquid to cover the bottom of the chamber. When the WVTR has reached steady state, the experiment can be terminated.

Experiments may also be conducted at humidity levels below 100 %. Any method that can produce a step change in humidity may be used. The step change in humidity shall be short enough to reach at least 90 % of the desired value in a time less than 0,05 · $T_{1/2}$, where $T_{1/2}$ is the time at which WVTR ($T_{1/2}$) is half of the WVTR at steady state.

EXAMPLE Controlled humidity step change can be accomplished by placing the test cell in a chamber with controlled temperature and humidity. Dry nitrogen or air is blown through the side of the film where the water will be sourced. Care is taken such that the tubes go through the chamber for a sufficient length of time to thermally equilibrate the gas before it contacts the test cell. This could require one to place a thermocouple in the N_2 flow to make this determination. Once thermal equilibration is achieved, and the permeated moisture rate is below detection, air is pulled out of the water source side using a pump allowing the humidified air in the chamber to enter the test cell. The vacuum pressure must be low enough that it will not deform the sample..

The time to be considered adequately close to the steady state shall be at least 4 · $T_{1/2}$, but preferably greater than 8 · $T_{1/2}$ for Fickian materials. For non-Fickian materials (e.g. edge seal materials loaded with desiccant), this steady-state time may not need to be as long because a more abrupt curve is typically obtained. For non-Fickian materials, a duration of at least 4 · $T_{1/2}$ shall elapse, or alternatively if the WVTR has not changed by more than 5 % over a time period of 0,5 $T_{1/2}$, then sufficient data has been collected and the test can be terminated.

The diffusivity and solubility of moisture in the material, along with the instrument capabilities affect the thickness of the sample needed for measurement. The half-time $T_{1/2}$ shall be 10 times greater than the instrument delay time (T_{delay}) to determine the diffusion coefficient or the breakthrough time T_{10}. If the half-time $T_{1/2}$ < 10 T_{delay}, then increase the sample thickness and retest. The $T_{1/2}$ should vary with the square of the sample thickness so a fourfold increase in $T_{1/2}$ should be obtained for a doubling of the sample thickness. Conversely, the sample thickness may need to be reduced if the permeation rate is below the detection limit of the instrument or if adsorption of moisture on instrument surfaces contributes unpredictably to the instrumental delay time (T_{delay}).

7 Calculations

7.1 Determination of diffusivity and solubility of moisture

An example of how to calculate diffusivity and solubility is given in Annex A. The instrument delay time T_{delay} accounts for the fact that for a specific instrument, with a specified flow rate, at a given permeation rate, there will be a delay between when moisture permeates a film and when it is detected. This value should vary approximately inversely with the carrier gas flow rate, and typically (but not always) is independent of sample thickness and permeation properties. For very low permeation rates, T_{delay} may further be affected by adsorption on the instrument surfaces. This value shall be determined using one of several methods: as a constant for an instrumental configuration by measuring two samples of a material with different thicknesses (l) at the same conditions and fitting the same set of values for D, S, and T_{delay} to the two data sets. Alternatively, T_{delay} may be determined by measuring the same Fickian film at several temperatures and fitting them using the same value for T_{delay} after fitting to an Arrhenius curve to obtain a straight line. An upper limit for T_{delay} can be determined by measurement of a thin film with a very short transient.

The diffusion shall be considered Fickian if (ignoring instrumental re-zeroing or similar noise) the WVTR (T_R) data closely fits Equation (1),

$$T_R(t) = \frac{DS}{l}\left[1 + 2\sum_{n=1}^{\infty}(-1)^n e^{\left(-\frac{Dn^2\pi^2 t}{l^2}\right)}\right] \quad (1)$$

where l is the film thickness and t is the time defined as the difference between the time the moisture was added and T_{delay}.

When using Equation (1), the terms for n shall be used up to at least n = 15, but values of 50 to 100 are recommended. This equation is only applicable for Fickian materials where the diffusion constant is independent from concentration.

The best–fit to Equation (1) can be determined by minimizing the sum of the square residuals, Σ{[measured $T_R(t)$] – [$T_R(t)$ from Equation 1]}^{2}. A visual fit only of the data is acceptable also. Either of these methods is easily accomplished by using a spreadsheet. It is suggested that the spreadsheet be set up allowing the data to be fitted by adjusting the parameters $D \cdot S / l$ and D / l^2. These parameters can be independently and visually fit to the equation more easily. The quantity $D \cdot S / l$ is the steady-state WVTR and is fitted first. This is followed by fitting the quantity D / l^2, which is the characteristic diffusion time and is adjusted to match the lag in permeation.

Values for D and S shall only be determined with this method for Fickian materials. When making this determination, a minimum of 10 data points shall be taken, at even intervals, before 4 · $T_{1/2}$. A material is considered Fickian if the difference between the measured and calculated values has an error that is less than 5 % of the steady-state WVTR at all points (instrumental transients, such as those caused by re-zeroing, may be ignored in this determination) (see Figure A.1 and Figure A.2 for examples). Often, a laminate material may have a single low-permeability layer that dominates the transient permeation resulting in a composite structure with the appearance of being Fickian. In the case of any laminate film, the diffusivity and solubility shall be reported as being only apparent properties (D_{app}, and S_{app}).

Permeability (P), or specific T_R, is defined as $P = D \cdot S = T_R \cdot l$. Because this quantity is not dependent on sample thickness, on the transient permeation time, or on the non–Fickian nature of a material, it can often be a better property (than WVTR) for intercomparison of different materials. Permeability values shall be reported after the material has reached steady state.

7.2 Determination of breakthrough constant

Examples of how to calculate the breakthrough time are given in Annex A. For materials used as edge seals, or for low-diffusivity encapsulants intended to be used to similarly reduce moisture ingress, the 10 % breakthrough constant K_{10}, relates the distance, X, for moisture permeation at a constant temperature and relative humidity to elapsed time as,

$$X = K_{10}\sqrt{\tau} \quad (2)$$

This value is determined by substituting the film thickness l for X as