

“Inverted-Wet-Cup Method”

Water Vapor Permeability of Packaging Materials

– Application Note 24-01

Scope

In this application note, the Inverted-Wet-Cup method is presented using the example of a packaging material and a nitrile rubber material in combination with liquids.

Introduction

The water vapor permeability of materials is an important property, for example in packaging materials, such as paper, plastic films or cardboard, but also in the area of building materials such as vapor barrier films or protective coatings. Especially in the case of packaging material, water vapor permeation both into and out of the packaging is an important criterion regarding shelf life.

Therefore, knowledge and control of water vapor permeability are essential to evaluate and optimize a product regarding the intended application.

To determine water vapor permeability, proUmid offers a precise and convenient method with the Permeability Kit [1].

For this purpose, a permeability dish is covered with the film to be tested. By filling those dishes with either a desiccant (*Dry-Cup-Method*), a saturated salt solution or water (*Wet-Cup-Method*), a difference to the partial water vapor pressure of the environment is generated, resulting in a migration of water molecules through the film. The measurement of water vapor migration is carried out gravimetrically.



Fig. 1: Inverted-Cup. The drip dish below protects the instrument from any liquid that may leak from the sample dish.

As additional option, the Inverted-Cup Method (Fig.1) is available as a modification of the Wet-Cup-Method.

This method is particularly useful when a packaging material is in direct contact with a liquid product. The inverted arrangement eliminates the influence of the air layer on water vapor permeation and ensures a more realistic measurement result.

The proUmid Inverted-Cup-Kit contains a sample tray with six positions (Fig. 2), including a reference cup and five sample positions, each with a test area of 15.02 cm². The dishes are made of Ni-coated aluminum to ensure high stability of the material also against aggressive liquid components.

Sample preparation

For measurement, the sample is cut according to the diameter of the raised edge of the clamping ring (Fig. 2). To ensure tightness, PTFE paste can be applied to the contact area between dish and sample.

Then, the permeability dish is filled with the test liquid (Fig. 3) and covered with the sample material. Locating pins can be used to fix the sample in place and prevent it from slipping (Fig 4).

The clamping ring is fixed on the dish with screws and spacers (Fig. 5) and finally the prepared sample dish and placed upside-down in the drip dish and positioned in the sample tray (Fig. 6)

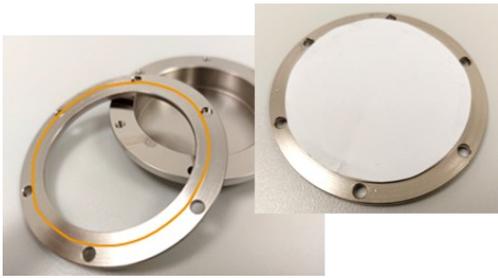


Fig. 2



Fig. 3



Fig. 4



Fig. 5



Fig. 6

Measurement Principle

The Inverted-Cup method is described in standard ASTM E96 [2].

The transfer of water vapor through the sample material is caused by different partial vapor pressures inside and outside the sample cup. In the wet cup arrangement, the liquid in the sample cup normally creates an atmosphere of high relative humidity - e.g. in the case of pure water, close to 100 % RH. By setting a lower relative humidity in the climatic chamber of the SPS/Vsorp device, the migration of water molecules through the sample in the direction of the driving pressure gradient is induced. As a result, a change in the weight of the sample cup can be measured with the analytical balance of the DVS device. In the case of the "wet cup" arrangement, a continuous weight loss is measured, which is due to water vapor migration from the liquid through the sample material into the climate chamber (Fig. 7).

Calculation of the Water Vapor Transmission Rate

The WVTR is calculated from the slope of the linear section of the mass change over time (Fig. 7). The WVTR is then related to the area of the sample and usually to a time unit of one day [d]. [Eq. 1]

$$WVTR = \frac{\Delta m}{\Delta t \cdot A \cdot 24} \quad \text{Eq. 1}$$

Δm : Weight change in [g]; Δt : Time interval in [h]
 A : Sample surface in [m²]

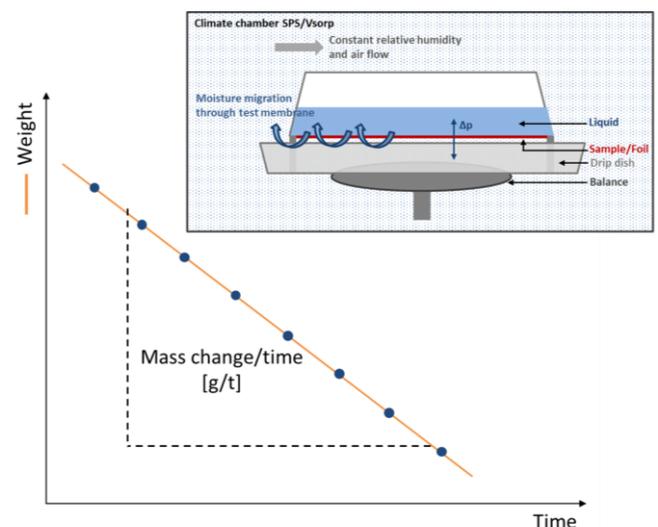


Fig. 7: Principle, schematic set-up and exemplary result of an Inverted-Cup measurement.

Experiments

To test the vapor tightness, the cups were filled with water and covered with an aluminum disc. Silicon paste was applied on the contact area between cup and sample to improve tightness of the set-up. Fig. 8 shows the results of the experiment. In addition, a standard sorption measurement of an aluminum disc was done to analyze moisture sorption behavior in comparison. Both curves show the similar course with a first decrease of weight due to settling of the relative humidity in the climate chamber down to ~0% RH and associated desorption processes from the sample surface. However, afterwards no further weight loss and thus no permeation of water vapor through the set-up could be observed which confirms tightness of the set-up.

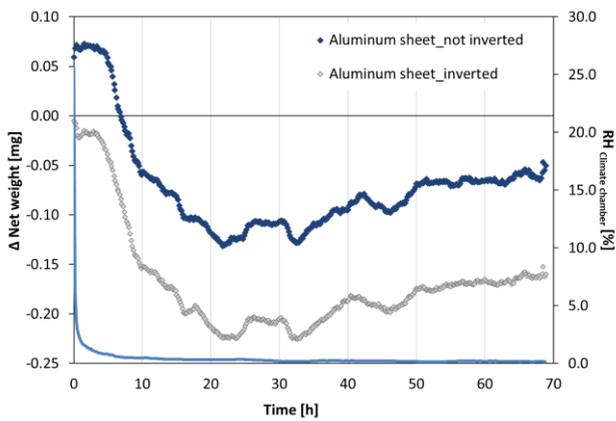


Fig. 8: Test of tightness of the experimental set-up.

Fig. 9 shows exemplary results of an Inverted-Cup experiment using coated cardboard samples in contact with several aqueous solutions.

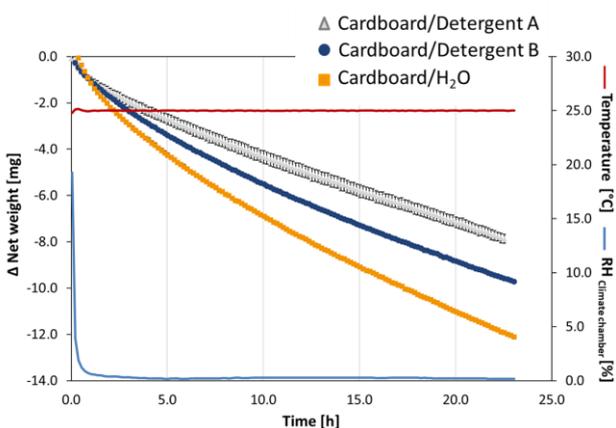


Fig. 9: Weight loss over time due to vapor permeation through coated cardboard samples depending on the test solution.

Tests were performed on a SPSx-1 μ High Load. Within the climate chamber of the SPS a relative humidity of 0% was set. Different test solutions were filled into the permeability dishes to analyze the resistance of the cardboard sample against moisture migration depending on the test solution.

Table 1 summarizes the experimental results. It shows that different water vapor transmission rates depending on the test liquid used were obtained. A WVTR of 6.7 g/m²/day was measured for pure water, for detergent A and B of 4.6 g/m²/day and 5.4 g/m²/day, respectively.

Table 1: WVTR of different test solutions through cardboard samples obtained from Inverted-Cup analysis

Sample	WVTR [g/m ² /day]	Δ RH [%]
Cardboard/Detergent A	-4.557 +/- 0.020	87
Cardboard/Detergent B	-5.345 +/- 0.005	96
Cardboard/H ₂ O	-6.721 +/- 0.057	100

An explanation for the differences in water vapor transmission rates is the properties of the liquids, which differ primarily in their viscosity and their water activity. Higher viscosity decreases the tendency of water vapor evaporation and dependent on the a_w -value of the liquid the partial vapor pressure differences between the inside of the dish and the SPS climate chamber varies.

This influences the driving force for water vapor permeation through the material and accordingly, as the results in Table 1 show, WVTR is highest for water with a Δ RH of ~100% followed by the detergents B and A with a Δ RH of 96% and 87%.

As a further example, Fig. 10 shows results from vapor transmission through a nitrile rubber material. Tests were done with several test solutions to get an estimate of the resistance of the material.

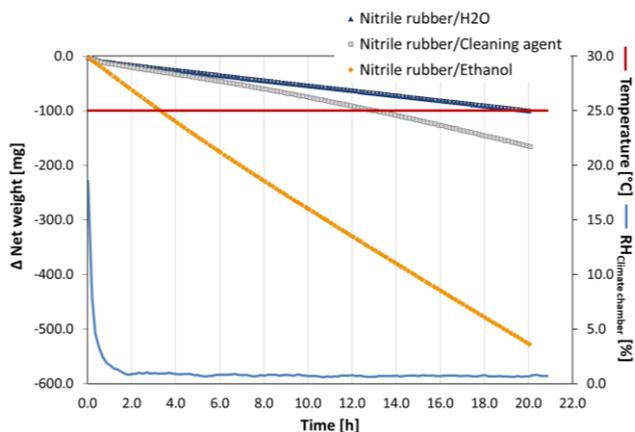


Fig. 10: Weight loss over time due to vapor permeation through nitrile rubber material depending on the test solution.

Results summarized in Table 2 show significant difference in material resistance against the tested solutions. The highest transmission rate of 410 g/m²/day was measured for the ethanol solution which can be explained by the relatively higher vapor pressure compared to water. However, even for water a quite high WVTR of 73 g/m²/day was detected.

Table 2: WVTR of different test solutions through nitrile rubber material obtained from Inverted-Cup analysis

Sample	WVTR [g/m ² /day]
Nitrile rubber/ H ₂ O	-73.717 +/- 1.633
Nitrile rubber/ Cleaning agent	-140.830 +/- 10.830
Nitrile rubber/ Ethanol	-410.324 +/- 5.102

Conclusion

Water vapor permeability is an important parameter for the characterization of packaging materials. For example, conclusions can be drawn about the protective properties of packaging materials in relation to the shelf life of the packaged product. The special design of the Inverted-Cup arrangement additionally provides valuable insights into the properties of materials that are in direct contact with a packaged liquid. The special arrangement avoids the influence of the air barrier and provides a realistic image of the actual packaging situation.

The Inverted-Cup kit developed by proUmid offers a simple and reliable method for characterizing water vapor transmission. The multi-sample option with

simultaneous measurement of up to 5 samples also ensures time-saving analysis.



References

- [1] proUmid Application note 12-01, Water vapor Permeability of Films
- [2] ASTM E96/E96M – 10; Standard Test Methods for Water Vapor Transmission of Material