

Testing Water Vapor Transmission Rates for Flexible Barrier Materials Including Metalized Structures

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ABSTRACT

As new materials with increased barrier properties are developed, the instruments and test methods used for measuring the barrier properties must be modified. Metalized structures are a perfect example. Many challenges arise when measuring the water vapor transmission rate of metalized films, from erratic data to flaking of the metalization. Additionally, no ASTM method exists for determining the water vapor transmission rate of flexible pouches. This paper reviews methods for analyzing the water vapor transmission rates of metalized structures, both flat films and flexible packages.

INTRODUCTION

Advancements in barrier quality of materials are occurring at a rapid pace. With more stringent packaging requirements and longer desired product shelf-life, packaging engineers must create materials with unprecedented barrier levels. Testing the barrier properties of packaging materials has become commonplace. ASTM has published several standard methods for evaluating barrier properties of materials including F1249 for water vapor and D3985 for oxygen. As the barriers improve, slight modifications of the methodology must also be made in order to achieve lower transmission rate results.

ASTM F1249 outlines an instrumented procedure for determining the rate of water vapor transmission through flexible barrier materials. In the method, a pressure-modulated infrared sensor is used to measure the water vapor concentration. Problems can arise when testing metalized films in accordance with this method due to the bellows pump incorporated in the system. This paper reviews some techniques to solve these issues.

Currently, there is no ASTM method documenting an instrumented technique for testing water vapor transmission rates for packages. This paper also reviews a successful technique to test the water vapor transmission rate of barrier packages.

METHOD REVIEW:

ASTM F 1249 is a standard test method for measuring the water vapor transmission rate through barrier materials. In this method a “dry chamber is separated from a wet chamber of known temperature and humidity by the barrier material to be tested.... Water vapor diffusing through the film mixes with the gas in the dry chamber and is carried to a pressure-modulated infrared sensor. The sensor measures the fraction of infrared energy absorbed by the water vapor and produces an electrical signal, the amplitude of which is proportional to water vapor concentration. The amplitude of the electrical signal produced by the test film is then compared to the signal produced by measurement of a calibration film of known water vapor transmission rate. This information is then used to calculate the rate at which moisture is transmitted through the material being used.” (ASTM F1249).

INSTRUMENT OPERATION:

Figure 1 illustrates the measuring system outlined in ASTM F1249. The flow passes through a metal bellows pump after the test chamber. Additionally, due to the sensor's mode of operation, the pressure-modulated infrared sensor is flow dependent, requiring a lower flow for better barriers or lower transmitters. At low flow rates however, the

bellows pump can create a slightly negative pressure on the dry chamber. The bellows pump can also create slight perturbations in a flexible sample.

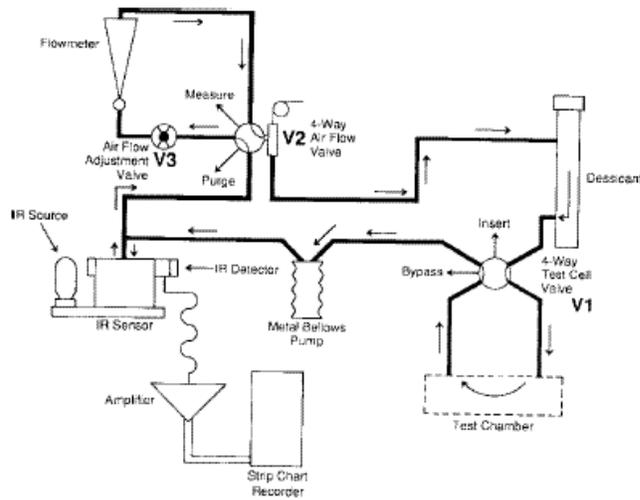


FIGURE 1. ASTM F1249 measuring system (ASTM F1249).

METALIZED FLAT FILM TESTING:

Metalized materials provide excellent barrier properties to packaging applications. Because most metalized structures are excellent barriers or low transmitters to water vapor, a low flow rate must be used when utilizing ASTM F1249, thus creating a slightly negative pressure in the dry chamber. Some metalized materials can be prone to flaking or degradation when subjected to the combination of water vapor and perturbations of the sample. Thus testing metalized films in accordance with ASTM F1249 can be present some issues. The following are some suggestions to help minimize these effects:

Metalization side to dry chamber:

Orienting the film in the diffusion cell such that the metalization side is facing the dry chamber will limit the exposure of the metalization to water vapor, thus decreasing the likelihood of flaking.

Masking:

A foil mask is typically used to mount samples smaller than the required 50cm² diffusion chamber area or to reduce the area available for permeation for high transmitters. It consists of two square pieces of adhesive backed foil with circular cut-outs in the center. The sample is placed between the two pieces, attached by the adhesive. The cut-out can range in size from 40cm² to 1 cm². Masking a metalized sample with a 40cm² mask will increase the overall rigidity of the sample, thus decreasing the effects of the perturbations. Although the sample rigidity is increased, it is important to note that the area is reduced by masking, thus decreasing the area available for permeation and ultimately reducing the sensitivity of the test.

Screen on dry chamber side:

A screen is available as a support for a metalized film. Figure 2 illustrates how the screen is inserted into the ports on the dry chamber side of the diffusion cell. The screen supports the metalized film and decreases the effect of the perturbations caused by the bellows pump on the instrument. It is recommended to calibrate with the screen prior to using.



Figure 2. Screen placed in dry chamber of diffusion cell

Flow rate modification:

Although the instrument recommends testing low transmitters at a flow rate of 10 sccm, other flow rates are possible. A flow-rate of 25 sccm has shown to have limited perturbation effects due to the bellows pump but still provide the sensitivity needed for low transmitters. It is crucial that the instrument be calibrated at a flow rate of 25 sccm if this technique is used.

Single cell analysis:

The instruments manufactured in accordance with ASTM F 1249 typically have two diffusion cells, enabling the analysis of two samples concurrently. The instrument can be set to analyze one sample while the other is being vented to room air. The process of switching cells, however, can result in additional back pressure on the sample. To avoid this, test only one sample at a time.

FLEXIBLE PACKAGE TESTING:

Currently, no instrumented ASTM method exists for testing the water vapor transmission rate of flexible packages. This is largely due to issues that can be created by the bellows pump used in ASTM F1249. At low flow rates, the slight negative pressure created by the bellows pump creates a vacuum in the flexible pouch, causing it to collapse. For higher transmitters, ASTM F1249 can be used in conjunction with ASTM F 1307, “Test Method for Oxygen Transmission Rate Through Dry Packages Using a Coulometric Sensor”, (reinstated 2002) to successfully test the water vapor transmission rate of flexible packages. The packages are simply mounted or fixtured in accordance with ASTM F1307, attached to the instrument used in ASTM F1249 and tested. It is important to maintain a constant temperature and water vapor driving force throughout the experiment. Environmental chambers are often used for this purpose.

For better barriers requiring lower flow rates, nafion tubing can be used to remove the effects of the vacuum. Figure 3 illustrates the set-up with nafion tubing. The tubing is attached to both inlet and outlet carrier gas lines and inserted into the package. The lines are then attached to the instrument. The nitrogen gas flows through the inlet tubing to the nafion where it picks up any permeating water vapor and then exits through the outlet tubing, to the instrument and the sensor. Because the tubing is not porous, there is no vacuum effect on the package. The justification for the transmission rate calculation is shown below.

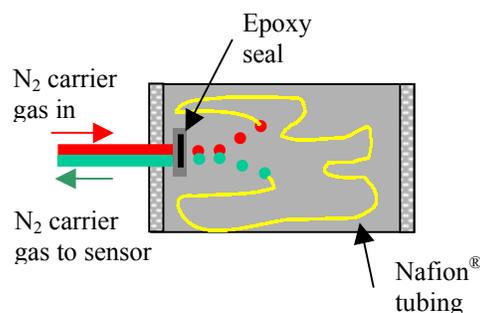


Figure 3. Flexible package set-up to test water vapor transmission rate.

Nafion tubing:

Nafion is a commercially available perfluorosulphonate cation exchange membrane with an extremely high water vapor transmission rate. Nafion tubing is manufactured in a variety of diameters.

Multiple barrier relationship:

When evaluating the transmission rate of multiple barriers, an analogy to electric circuits can be applied. The parallel resistance equation, where the inverse of the total resistance is equal to the sum of the inverses of the individual resistances, can be applied to transmission rates as shown in Equation 1. TR_{total} is the transmission rate through both the flexible package and the nafion tubing, $TR_{package}$ is the transmission rate of the package and TR_{nafion} is the transmission rate of the nafion tubing.

$$\frac{1}{TR_{total}} = \frac{1}{TR_{package}} + \frac{1}{TR_{nafion}} \quad \text{Equation 1.}$$

Since the water vapor transmission rate of nafion (TR_{nafion}) is extremely high, $\frac{1}{TR_{nafion}}$ is a very small number, negligible in fact. The resulting relationship is shown in Equation 2.

$$\frac{1}{TR_{total}} = \frac{1}{TR_{package}} ; TR_{total} = TR_{package} \quad \text{Equation 2.}$$

EXPERIMENTAL DATA:

Two unknown types of flexible bags were tested for water vapor transmission rate using both the traditional method of combining ASTM F1249 and ASTM F1307 and the nafion tubing modification. Due to the difficulty of testing the packages at a low flow rate of 10 sccm, the flow rate was set to 50 sccm, the same flow rate where the calibration was conducted. Figure 4 (a and b) illustrate the experimental set-up used. Duplicates were tested for each case. As shown in Table 1, the results from the two methods correlated very well.



Figure 4. Experimental set-up used to compare water vapor transmission rate results with and without nafion tubing.

Table 2. Water vapor transmission rate results comparing two methods.

Package	Average Water Vapor Transmission Rate (g/day)	
	With Nafion	Without Nafion
A	0.20	0.19
B	0.32	0.33

CONCLUSION

Several methods were to discussed to successfully test the water vapor transmission rate of metalized films in accordance with ASTM F1249. These techniques included:

- Metalization side to dry chamber
- Masking
- Screen on dry chamber side
- Flow rate modification
- Single cell analysis

A method for determining the water vapor transmission rate of flexible barrier packages was presented. The method involved the use of nafion tubing and the combination of ASTM F1249 and ASTM F1307. Results showed excellent correlation when using the addition of nafion.

REFERENCES

1. ASTM F1249
2. ASTM F1307