WHITE PAPER

ASTM E96 VS. F1249 WHICH PROVIDES MORE ACCURATE TEST RESULTS?

Modern barriers require more sensitive and accurate test methods. Does E96 stand up to the test?

MOCON, Inc.
May 16, 2017

Contents

mocon® measuring the invisible

CONTROLLING MOISTURE IS KEY TO PRODUCT QUALITY

Abstract

ASTM International (formerly known as the American Society for Testing Materials) is recognized as a global leader in the development of international voluntary consensus standards.

ASTM has approved two test methods to track water vapor transmission from materials: ASTM E96 – Water Vapor Transmission of Materials Using Gravimetric Method (adopted in 1941), and ASTM 1249 – Water Vapor Transmission Rate Through Plastic Film Sheeting Using a Modulated Infrared Sensor (adopted in 1990). This white paper explores these methods to determine which provides better water vapor transmission rate (WVTR) test results to meet today's packaging needs.

Packaging plays an important role in protecting product quality.

Introduction

Product packaging plays an important role in protecting product quality for the duration of its desired shelf life and provides packaged product stability. Controlling moisture into and out of packaging is key to ensuring products meet increasingly longer shelf lives, and prevents products from drying out or becoming soggy before they are consumed.

At the same time, manufacturers seek to avoid the expense of over-packaging their products by finding a material that meets their needs without employing overly expensive packaging solutions. In today's global economy where products are often manufactured and shipped to different regions that can have widely varying environmental conditions, selecting the right packaging material often involves weighing economic, technical and geographic factors.

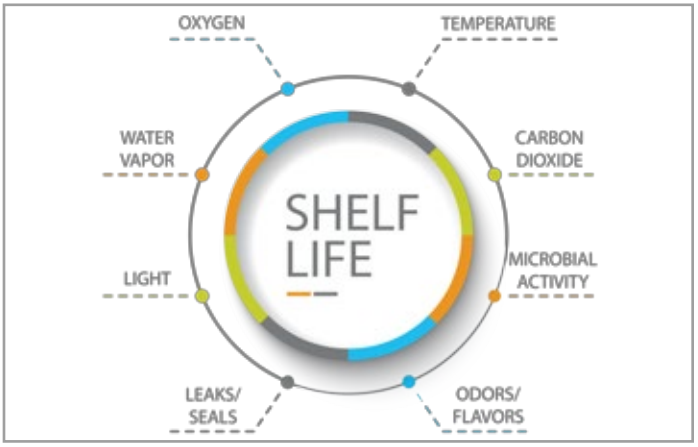
Package engineers need precise, repeatable and sensitive WVTR test results that prevent both over-packaging expenses and costly product shelf life failures. They also need testing methods that can produce accurate results quickly, without adversely affecting product development or time-to-market schedules.

Manufacturers seek to avoid the expense of over-packaging their products by finding a material that meets their needs without being overly expensive.

BOTH TEMPERATURE AND RH AFFECT PERMEATION

Water Vapor Permeation

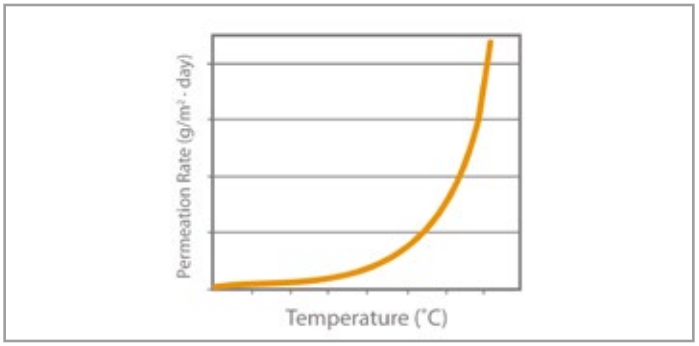
When considering packaging materials, it's important to remember that all materials permeate. Measuring permeation in various packaging materials is useful because a number of factors can affect shelf life, resulting in spoilage and/or a bad brand experience by consumers.



The following equation describes units of permeation:

$$\frac{\text{Volume of gas at STP} \quad \text{Material Thickness}}{\text{Area} \quad \text{Time} \quad \text{Pressure drop, driving force}} \qquad \frac{cm^3(273.15K, 1.013\times10^5\,Pa)\times cm}{cm^2\times s\times Pa}$$

Permeation is affected by temperature and relative humidity (RH). Typically, permeation rates change 5% to 7% per degree C. Consequently, precise control of both factors is critical for repeatable results.



In some materials, RH can also affect the permeation rate of gases. Proper RH generation and measurement are necessary to obtain accurate permeation results. To ensure proper testing conditions, RH must always be measured at the film.

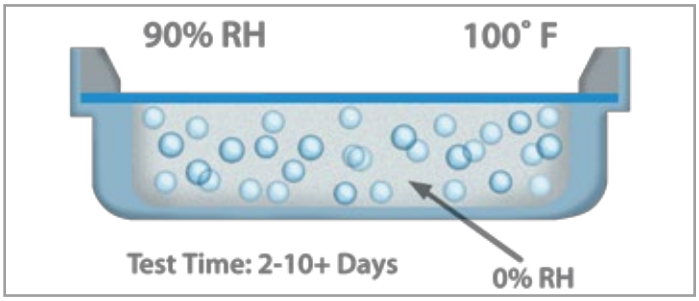
Effects of Humidity on O_2 Transmission Rate of Materials

Polymer	0% RH	100% RH
ABS	0.06	0.06
HDPE	6.6	6.6
LDPE	28.8	28.8
Nylon 6	0.06	0.3
PVA	3.3	9.0
PVOH	0.0006	1.5
Uncoated	0.0078	12.0
Cellophane	0.0078	12.0

(10 x 10 ^11 ml x cm/cm2 x sec x cmHg) analyzed at 25°C Source: K. Cooksey, Important factors for selecting food packaging materials based on permeability.

ASTM E96

Known as the gravimetric or cup test method, ASTM E96 (E96) is used to determine WVTR through a barrier film or sheet. The two test procedures associated with this method are the desiccant method and the water method.



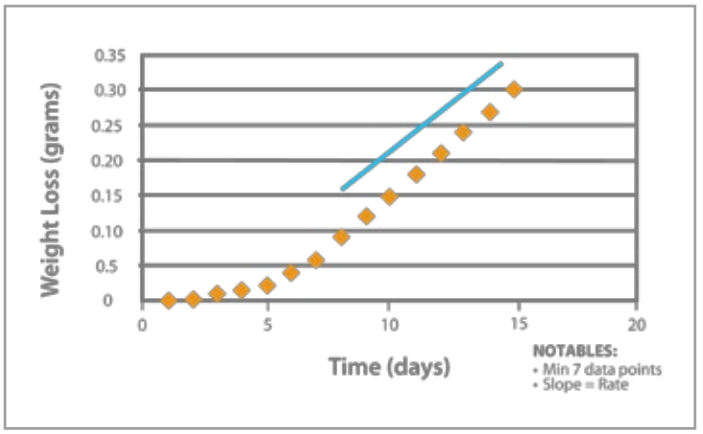
During the gravimetric test, a cup or dish is filled with desiccant or water, placed in a temperature and humidity controlled chamber, and weighed periodically. Depending on the barrier properties and sample thickness, the gravimetric method could require a test time of one day to two to three weeks (or longer). Enough data points should be collected until weight gain (or

F1249 AUTOMATES THE EXACT CONDITIONS REQUIRED FOR TESTING

loss) trend reaches steady state or equilibrium.

It is possible to test only one sample per test dish, so multiple test setups are required to analyze multiple samples. To obtain accurate results, test conditions must remain constant. Slight changes in RH and temperature can significantly affect test results – even removing the dishes from the environmental chamber for weighing can cause fluctuations that impact the results.

"The sample to be tested using E96 must be prepared by setting up the external test/challenge conditions (e.g., 37.8°C and 90 percent RH). This requires an environmental chamber/oven, a sealed container, perhaps an extra gas line, and desiccant or saturated salt solution to drive the desired challenge," says MOCON Scientist Michael Kragness. "The test also is limited to the sensitivity of the analytical balance used."



The ASTM E96 specification itself states, "A permeance value obtained under one set of conditions may not indicate the value under a different set of conditions. For this reason, the test conditions should be selected that most closely approach the conditions of use." Therefore, it is important to always consider the product's destination environmental average temperature and relative humidity, and use these as test conditions to ensure results that meet real world packaging needs.

E96 is highly labor-intensive and generally provides decent in-lab repeatability on a small scale. However, when multiple environmental chambers are used and/or test results are compared between labs, results are less reproducible. Typically, test methods are considered suspect when it is difficult to obtain similar results for similar films in different labs. Due to the challenges in accurately controlling RH and temperatures, this method provides excessive lab-to-lab variability.

E96 test results are also highly operator-dependent. Technicians with less experience may achieve different results when testing the same materials. Experienced technicians, however, may develop techniques that work for them personally. Through round-robin testing, ASTM reports E96 has about 20 percent lab-to-lab variability.

In addition, unless the technician has access to a humidified walk-in chamber, the samples must be removed from the external test environment when taking weight measurements for E96. This has the potential to cause issues ranging from condensation resulting in erroneous weight measurements to compromising material mechanical integrity. An example of the latter occurs when testing certain medical thin films; headspace within the cup contracts, pulling a slight vacuum that stresses the sample.

Due to the limitation of the sensitivity, this method is only suitable for testing poor to medium barrier materials. For modern high barrier materials, this method is not sensitive enough to measure the results, or efficiently obtain results in a reasonable time period.

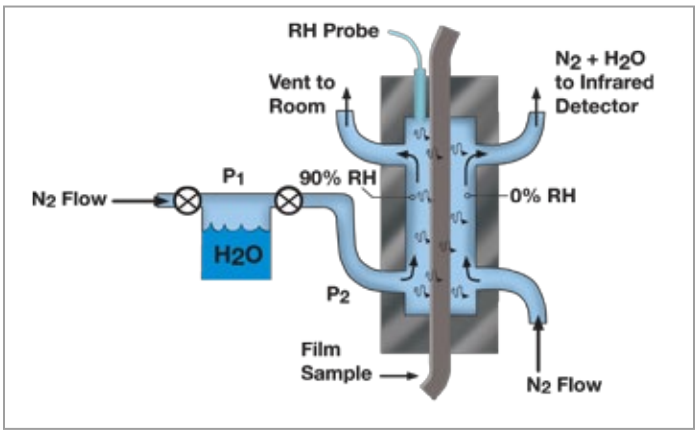
ASTM F1249

The purpose of the ASTM F1249 (F1249) test method is to obtain accurate data for the WVTR of plastic sheeting and film. This method is suitable to test flexible barrier films and sheets consisting of single and multilayer natural or synthetic polymers and foils, including coated materials.

This popular test standard is used to reliably measure WVTR

MORE SENSITIVE METHODS ARE REQUIRED AS BARRIERS IMPROVE

through flexible barrier materials in a variety of industries. In this method, "a dry chamber is separated from a wet chamber of known temperature and humidity by the barrier material to be tested. The dry chamber and the wet chamber make up a diffusion cell in which the test film is sealed. Water vapor diffusing through the film mixes with the gas in the dry chamber and is carried to a pressure-modulated infrared sensor. This 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 tested." (ASTM F1249).



"As packaging materials moved from traditionally 'great barriers' (cans and glass) and 'poor barriers' (paper) to a variety of polymers, the duration and variability of E96 testing was a huge factor in slowing down research and quality control operations," said Kragness. "An instrumental method with less human interaction and more precision was needed. The MOCON name came from our founding name, Modern Controls, which referenced automating the manual E96 testing with an infrared detector (IRD) method."

F1249 relies on instrumentation provided by the MOCON PERMATRAN-W® product line, which includes a patented pressure-modulated infrared (PMIR) sensor that can accurately detect down to one part per million (ppm) of water vapor.

This family of test equipment can quickly and accurately test multiple samples. Personnel in 13 ASTM member labs conducted extensive round-robin testing, and achieved an impressive between-lab precision rate of ±3 percent, depending on the WVTR range. Originally issued in 1992, F1249 is now widely used in Asia, Europe and North America, and is recognized as standard method JIS K-7129 in Japan.

According to Robert Demorest, President & CEO, the main advantage of F1249 over E96 is speed. "General Mills had a cup test cabinet with 100 cups in it, and technicians would spend two days setting them up, weighing each cup, and getting them into the cabinet. Then, over the next 10 days they would remove each cup, weigh it and log the weight, and return the cup to the cabinet," said Demorest. "Technicians could determine the equilibrium WVTR from the 10 day curves. The entire cycle took two weeks, and they would have the WVTR on 100 samples. However, the PERMATRAN-W was much faster, providing an answer for five samples in one day, or a bit slower when the IRD was used."

Also, results were more accurate as barriers improved, technicians were able to see better barriers, and they could log the results into their data storage system.

In North America, commonly reported barrier test conditions are:

Industry	Temperature	Relative Humidity
Food	100°F (37.8°C)	90%
Pharmaceutical	77°F (25°C)	60%

Testing directly at both the specified humidity and the specified temperature in WVTR testing always yields more precise results.

The PERMATRAN-W Model 3/34 accurately generates and maintains desired RH which is why it produces precise measurement results. Materials can be tested for exact RHs between 0 percent and 100 percent, enabling operators to meet specific test conditions required by various companies and industries.

Key Benefits of ASTM F1249 Method

F1249 has many clear advantages over E96, including precision, repeatability, accuracy and sensitivity. The inability to achieve precise and repeatable packaging test results may result in package failure when confronted with demanding environmental conditions.

Under-packaging products can result in costly product shelf life failure, as well as potential lost goodwill among retailers and consumers. Similarly, over-packaging results in wasteful and unnecessary spending for packaging materials – money that could be better spent developing new products.

The importance of achieving the same test results by different people, using different instruments in different locations – can't be overstated. F1249 eliminates operator-dependent variables and environmental factors for more accurate test results which enable more informed packaging decisions.

Technological advances have greatly improved the ability to quantify differences in materials that might otherwise appear to have the same, or very similar, characteristics. F1249 can accurately differentiate barrier attributes of materials and packaging below the sensitivity limits of E96.

As the speed of business continues to accelerate, and being first to market conveys increasing rewards, the cost of time to test and verify packaging options cannot be overlooked. E96 can take significantly longer to measure WVTR to equilibrium than F1249 test methodology. By producing actionable results in less time, F1249 accelerates product and packaging development, and time to market is similarly reduced.

"High transmitters (i.e., >0.5 g/pkg-day) may complete in one day using both E96 and F1249 test methodologies. However, the time benefit associated with using F1249 dramatically increases as the barrier performance improves," says Kragness. "Most analytical balances don't have the sensitivity to see minute changes in weight associated with permeation rates, whereas the PERMATRAN can see levels of moisture correlating to 0.005 $g/m^2 \cdot$ day. To put this in perspective, the analytical balance would need a resolution repeatable at 2.5 x 10^5 g using a $50cm^2$ test area to be considered comparable to the PERMATRAN."

Reducing labor costs and increasing productivity are never-ending challenges for companies. E96 is a manual, labor-intensive methodology compared to F1249, an automated, instrumented test methodology. Using F1249 allows companies of all sizes to accomplish more with fewer resources and provides better, more precise results.

Conclusion

F1249 provides more precise and repeatable WVTR testing data compared to E96, and provides that information in less time. Greater sensitivity provided by F1249 methodology ensures lab and business managers have the information they need to make packaging decisions that meet requirements in challenging environmental conditions globally.

For more information on these test methods, contact your local MOCON Representative.

MOCON, Inc.
7500 Mendelssohn Ave N
Minneapolis, MN 55428
USA
info@mocon.com
www.mocon.com

MOCON, Inc. North America is ISO 9001:2015 Certified
Certificate No: 216208-2017-AQ-USA-ANAB