ASTM D3985 VS. ASTM F2622

CHOOSING THE RIGHT ASTM METHOD FOR YOUR OTR APPLICATION

WHITE PAPER

This paper provides you with the information necessary to decide which permeation instrument is best suited for your unique application, ensuring you get the most accurate, reliable results to safeguard your brand.

MOCON, Inc.
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NOT ALL PERMEATION SENSORS ARE EQUAL... THEREFORE TWO METHODS WERE DEVELOPED

Abstract

In the world of oxygen permeation testing, not all instruments are created equal. This is why ASTM[1] approved two distinct test methods for determining oxygen transmission rates of packaging materials: ASTM D3985 – 05 (Reapproved 2010) – "Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor" and ASTM F2622 – 08 – Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using Various Sensors. This white paper explains the difference between these two methods, and discusses when either method could be used and when one method would be superior to the other.

Introduction

Oxygen adversely impacts the shelf life of many foods by causing oxidation, bacteria growth, and other changes in product quality. To protect the product's quality, oxygen barrier packaging materials are used. Consequently, accurate Oxygen Transmission Rate (OTR) measurement is important when assessing oxygen barrier properties during the selection of packaging materials. Appropriate sensor technology is imperative for accurate, repeatable and reliable OTR results from which important commercial and quality control decisions are made. The ASTM methods differ, depending upon the sensor used. Only MOCON oxygen permeation instruments with the patented COULOX® coulometric sensor comply with ASTM D3985 as reapproved by ASTM Committee F02 on Flexible Barrier Materials in 2010.

Theory of the Coulometric Sensor

The Coulometric or Coulox® oxygen sensor is a fuel cell that performs in accordance with Faraday's Law. When oxygen enters the coulometric sensor, the sensor reacts with the oxygen molecules to release four free electrons. This reaction is detected by the sensor as an electrical current, the magnitude of which is proportional to the amount of oxygen flowing into the sensor per unit of time. Each and every oxygen molecule

To get reliable permeation data of high or ultra-high barrier materials requires an accurate and repeatable test method such as ASTM D3985.

Developed by Subcommittee: F02.10, ASTM test method D3985 was created as a standard method using coulometric sensor technology. ASTM F2622 was created for use of all other sensor types as they are not as reliable as the coulometric sensor.

that enters the sensor is analyzed, allowing a measurement of the oxygen transmission rate (OTR) that is 95-98% efficient; therefore, no calibration is required.

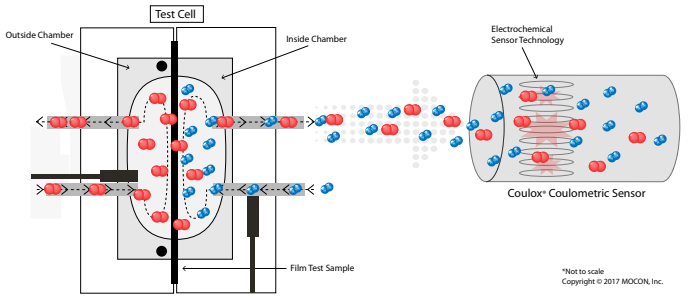

Figure 1. Example of a Coulometric Sensor

For a sensor to be considered coulometric, 100% of the oxygen that passes through the test sample must be analyzed. If an instrument claims to have a coulometric sensor, it is imperative to confirm whether 100% of the oxygen does in fact pass through the sensor (Figure 1). Because these test methods are considered "intrinsic" standards that do not require calibration, they can be used as reference methods to verify the accuracy of non-coulometric OTR test results.

ASTM D3985 was devised for dry testing, but can be modified for testing at different relative humidity conditions when combined with its sister method ASTM F1927-07 "Standard Test Method for Determination of Oxygen Gas Transmission Rate, Permeability and Permeance at Controlled Relative Humidity Through Barrier Materials Using a Coulometric Detector."

Non-coulometric Sensors

ASTM F2622 was drafted to allow for the use of various non-coulometric sensors, devices, and procedures. The sensor could be electrochemical or other type of detector. Certain electrochemical sensors are configured with a membrane over the sensor to protect the sensor from seeing all the oxygen molecules (Figure 2). Only a fraction or portion of the stream is analyzed. Although, as the name implies, an electrochemical reaction takes place and the number of electrons are measured to relate the quantity of oxygen in the stream, those electrochemical sensors only measure the concentration of oxygen. They are not 100% efficient. Therefore these instruments do not fit the definition of coulometric rather the method described in ASTM F2622. They could be called "non-coulometric" as they require calibration. To repeat, a coulometric oxygen sensor is one which is at least 95% efficient or one which literally sees and counts all the oxygen permeating through

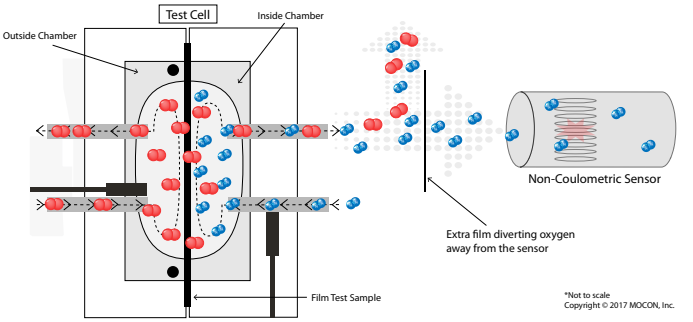

Figure 2. Example of a Non-coulometric Sensor

the test sample. To reach the sensor, the permeant must pass through a film - that itself is a barrier. Consequently, these instruments have difficulty measuring the OTR of high oxygen barriers because the amount of oxygen received by the sensor is so small. Also, to ensure the total transmission rate is calculated accurately, the electrochemical sensor must make a comparison to a known amount of oxygen which makes the system calibration-dependent. When calibrating, it is important to use a material that has a similar OTR to the test material to ensure the accuracy of the test results. However, for calibration-dependent systems, the nearest reliable calibration level is magnitudes higher than many barriers. Moreover, the calibration process could be operator-dependent. Thus, when measuring high oxygen barriers, large errors and poor reproducibility can result.

Other Aspects to Consider

The following aspects also should be considered when

SENSOR TYPE CAN BE THE DIFFERENCE BETWEEN A RIGHT AND WRONG ANSWER

comparing the sensors used in each method.

1. Sensor Size

Coulometric sensors are much larger than non-coulometric ones because they are designed to handle a higher volume of test gas. As they are larger, they can contain more reagent and last much longer. While a non-coulometric sensor may only last a few months, depending on how often it is used, coulometric sensors can last up to two years. This decreases the cost of ownership associated with permeation testing.

2. Effects of Ambient Conditions

True coulometric sensors such as the COULOX® used in most MOCON oxygen permeation systems are not affected by flow, pressure or temperature changes. However, calibration-dependent non-coulometric sensors are affected by these variables. As these variables are constantly in flux, a system that is affected by them will exhibit reduced repeatability. When a secondary film is used to protect the sensor, environmental conditions that would affect the barrier property of this protective film alter the number of oxygen molecules seen by the sensor. Consequently, the accuracy of measurements from non-coulometric sensors is reduced.

Which Method is Right for Your Application?

Coulometric and non-coulometric methods have different applications. For example, if the application is produce/fruit packaging where high levels of oxygen are needed for products' respiration, or other products that are not oxygen sensitive, then the packaging material used is usually polyolefin or other low oxygen barrier material. In this case, instruments with non-coulometric sensors suit the purpose. An example is the recently developed MOCON OX-TRAN® 2/12.

When working with high barrier materials to package foods or other products that are easily oxidized, the low OTR level packaging material requires a more accurate sensor. Best practice is to select an instrument which conforms to the coulometric method. Non-coulometric instruments require refinements to be made to temperature, relative humidity, system leaks and other parameters which can make the difference between a right and wrong answer. When business decisions are based on results generated from a permeation instrument, then accuracy and repeatability of the results must be reliable.

Appendix: Summary of Sensor Comparison

Sensor Type	Coulometric	Non-Coulometric
ASTM Method	D3985	F2622
Calibration Required?	NO	YES
Carrier Gas Dependent?	NO	YES
Sensor Response Linearity	Linear for whole range	Not Linear (may need to calibrate at different levels)
Affected by Ambient Temperature?	NO	YES
OTR level	High to Ultra-high barriers	Medium to High barriers
Sensor Cost	Usually higher cost but longer life	Usually cheaper but shorter life
NIST Traceability	YES	NO

Footnotes

1. 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.

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