The benefits of using coulometric sensors in oxygen transmission rate testing

Introduction

Coulometric sensors determine the number of molecules transformed during an electrolysis reaction by measuring the amount of electricity (in coulombs) consumed or produced.

This method yields a linear output as predicted by Faraday's Law. When oxygen enters the coulometric sensor, it reacts to release four free electrons. This 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 time. Every oxygen molecule that enters the sensor is analyzed, allowing a measurement of the oxygen transmission rate (OTR) that is 95-98% efficient.

Test methods

There are three ASTM test methods for analyzing OTR using a coulometric sensor.

ASTM D3985

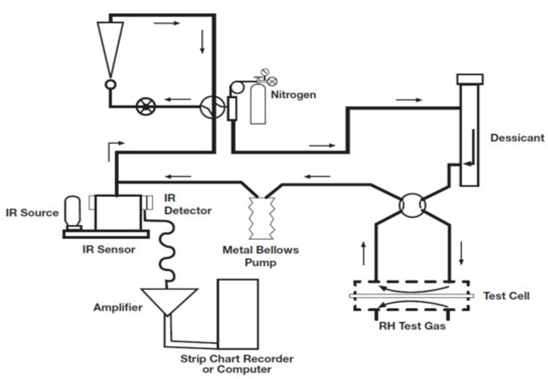

Figure 1. ASTM D3985.

This is the standard test method for determining the OTR through plastic film and sheeting. The test barrier is mounted in a test chamber. One chamber is purged of oxygen by a stream of nitrogen, after which a stream of oxygen is introduced to the other side and allowed to permeate to equilibrium. The oxygen that permeates through is sent, along with the nitrogen gas, to the sensor, where it produces an electrical current.

ASTM F1927

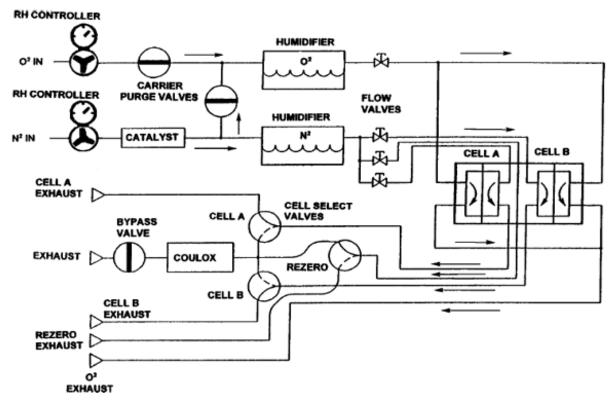

Figure 2. ASTM F1927.

This test method is the same as D3985, except it allows for different relative humidities during testing. This is important because the permeation rate of some materials is affected by relative humidity, causing the OTR to change. This must be taken into account when testing these materials or the OTR may not accurately reflect the material's performance under all conditions it will be exposed to.

ASTM F1307

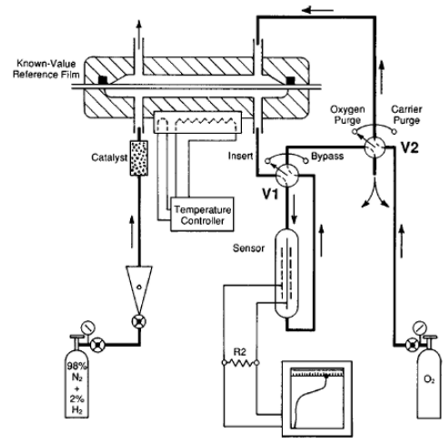

Figure 3. ASTM F1307.

While the previous two test methods are only for testing films, this method allows for the testing of packages. The standard is specifically for dry testing, but it is possible to modify it to test at different relative humidities.

Because these test methods are considered "intrinsic" standards that do not require calibration, they can be used as reference methods to ensure the accuracy of non-coulometric OTR testing.

Coulometric vs. non-coulometric sensors

Most sensors that claim to be coulometric do not actually fit the definition because only a small amount of permeant is sent to the sensor (Figure 4). With these instruments only about 3% of the permeant that passes through the test barrier is being analyzed, and the total transmission rate must then be calculated either manually or with computer software. These instruments have difficulty measuring the OTR of high oxygen barriers because the amount of oxygen sent to the sensor is so small.

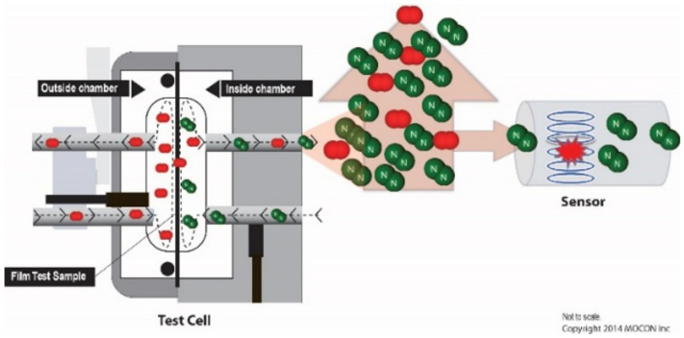

Figure 4. A test cell with a sensor that is not truly coulometric.

Coulometric sensors are much larger than non-coulometric ones because they are designed to handle a higher volume of test gas. Since they are larger, they are capable of containing more reagent, so they 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 for up to two years, decreasing the cost associated with permeation testing.

An obstacle when using a non-coulometric sensor is that to get to the sensor the permeant must pass through a film, which is itself a barrier. To ensure that the total transmission rate is calculated accurately, the sensor must be calibrated to determine how much of the test gas is actually passing through the film into the sensor.

Problems associated with calibration

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. One option for improved accuracy is to use different calibration films for test barriers with different permeabilities, but even so, non-coulometric instruments are unable to accurately measure the OTR of high oxygen barriers. This is not an issue with coulometric sensors, since every molecule of oxygen that passes through the test film is sent to the sensor for analysis (Figure 5).

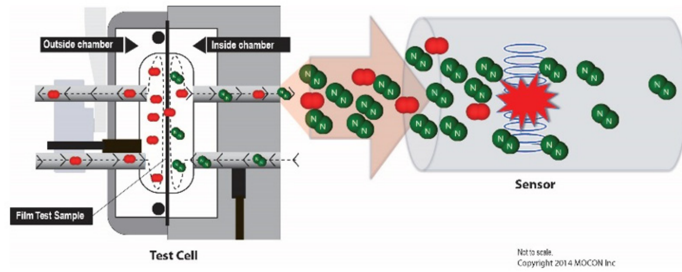

Figure 5. A test cell with a coulometric sensor.

Coulometric sensors do not require calibration the way non-coulometric ones do. Calibration can be operator-dependent and therefore have poor reproducibility, particularly when calibrating for high oxygen barriers.

The process of calibration can take a significant amount of time when calibrating for high barriers. All testing must be put on hold during this time, which is an inconvenience for companies with a high test volume. It could create a backlog of materials to be tested and potentially lead to lost revenue.

Conclusion

In order for a sensor to be considered coulometric, 100% of the oxygen that passes through the film or package must be analyzed. If an instrument claims to have a coulometric sensor it is essential to establish if 100% of the oxygen does in fact pass through the sensor. If this is not the case, they do not fully comply with the ASTM standards and will require calibration with all its associated problems.

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