Labthink Instruments CO.,LTD.
144 Wuyingshan Road, Jinan, China
Tel: +86-531-85811021 85864214
Fax: +86-531-85812140
Website: www.labthink.cn
E-mail: trade@labthink.cn

Standard of Coulometric Film Oxygen Permeability Testing

ASTM D 3985

Abstract: based on standard ASTM D 3985, this article briefs on coulometric oxygen permeability testing of film and sheet materials. It also presents a detailed introduction about the principal and operating procedure of coulometric testing.

Key words: ASTM D 3985, coulometric sensor, transmission rate, Faraday's law, and coulometric analysis.

With the accelerated development of trade globalization, some testing technology used in American commerce and trade gradually appeared in our country. After China's entering of WTO, two sensor permeability testing methods (coulometric sensor method for film oxygen permeability testing and electrolytic method for film water vapor permeability testing) are added to the testing standards of medicine package. Since these two methods are not methods adopted by national standards, they are less applied in our country.

Introduction about coulometric method is rather rare in YBB00082003. The following is a detailed introduction about that based on the reference standard ASTM D 3985 of YBB00082003.

1. Application Fields

This test method covers a procedure for determination of the steady-state rate of transmission of oxygen gas through plastics in the form of film, sheeting, laminates, coextrusions, or plastic-coated papers or fabrics. It only applies to oxygen permeability testing of specimen under drying condition(Relative moisture less than1% RH). Test performed under wet condition can refer to ASTM F 1927. Oxygen permeability of most film will vary greatly with the changing of humidity.

2. Testing Method

The specimen is mounted as a sealed semi-barrier between two chambers at ambient atmospheric pressure. One chamber is slowly purged by a stream of nitrogen and the other chamber contains oxygen. As oxygen gas permeates through the film into the nitrogen carrier gas, it is transported to the coulometric detector where it produces an electrical current, the magnitude of which is proportional to the amount of oxygen flowing into the detector per unit time. Among that, oxygen gas is the testing gas and nitrogen gas is the carrier gas. Oxygen gas concentration of upper chamber is higher than that of lower chamber, due to which certain concentration difference is formed between both sides of specimen. During the transmission process, oxygen gas transmits from upper chamber through specimen into lower chamber.

Labthink Instruments CO.,LTD.
144 Wuyingshan Road, Jinan, China
Tel: +86-531-85811021 85864214
Fax: +86-531-85812140
Website: www.labthink.cn
E-mail: trade@labthink.cn

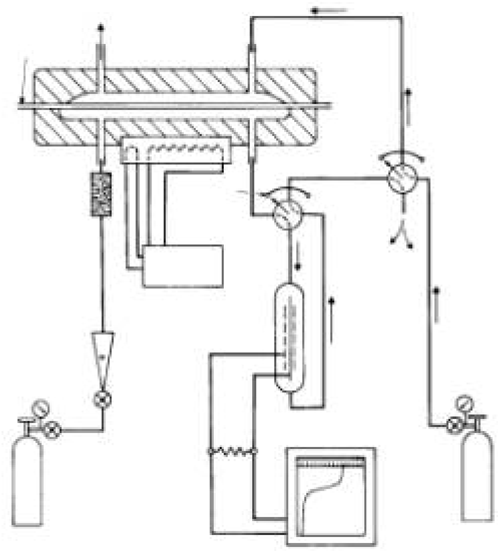

Fig.1 Instrument Principle of Coulometric method

3. Testing interference

The presence of certain interfering substances in the carrier gas stream may give rise to unwanted electrical outputs and error factors. Interfering substances include free chlorine and some strong oxidizing agents. Exposure to carbon dioxide should also be minimized to avoid damage to the sensor through reaction with the potassium hydroxide electrolyte.

4. Instrument Calibration

The oxygen sensor used in this test method is a coulometric device that yields a linear output as predicted by Faraday's Law. In principle, four electrons are produced by the sensor for each molecule of oxygen that passes into it. Experience has shown… under some circumstances the sensor may become depleted or damaged to the extent that efficiency and response are impaired. For that reason, this test method incorporates means for a periodic sensor evaluation. standard film is needed in instrument calibration. Since the data of standard film can directly influence the determination of calibration coefficient Q, special attention should be paid to film preparation and calibration repeatability. In this standard, NIST standard material is used for calibration testing.

5. Testing Process

Preparation of apparatus… Heating the apparatus will speed the drying and outgassing process… Inserting the specimen… Purging the system… purge air from the upper and lower diffusion cell chambers, using a flow rate of 50 to 60 mL. After 3 or 4 min, reduce the flow rate to the desired value between 5 and 15 mL/min. Maintain this configuration for 30min… INSERT THE

Labthink Instruments CO.,LTD.
144 Wuyingshan Road, Jinan, China
Tel: +86-531-85811021 85864214
Fax: +86-531-85812140
Website: www.labthink.cn
E-mail: trade@labthink.cn

SENSOR so that the carrier gas which has passed through both sides of the diffusion cell is diverted into the sensor… Establish E 0 … Switch OXYGEN into the test-gas side of the diffusion cell… The sensor output current should increase gradually, ultimately stabilizing at a constant value. While some thin films with high diffusion coefficients may reach equilibrium in 30 to 60 min, thicker or more complex structures may require a number of hours to reach a steady state of gas transmission. The steady-state voltage value of the oxygen transmission rate shall be recorded and labelled Ee . Appropriateness of carriers gas flow is of critical importance in the testing, which must be adjusted accurately according to the standard.

Oxygen diffusion coefficient can be calculated according to the formula in standard with the E_0 and Ee obtained during testing.

6. Other Cautions

First, oxygen sensor is of self-consumption type. The sensor should be replaced when there is obvious attenuation of output signal while cannot be offset by calibration. Back diffusion of air into the unit is undesirable. That is oxygen in the air will deplete the sensor and therefore shorten its lifespan.

Secondly, High oxygen concentrations in the carrier gas, from the testing of poor barriers, will tend to produce detector saturation. This will shorten the lifespan of sensor. However, this problem can be solved through two ways. One is to reduce oxygen concentration (reduce concentration difference between two sides of the specimen) in testing gas by mixing oxygen and nitrogen. Another way is to reduce specimen surface area through sticking barrier board made of metal or aluminum foil to two sides of specimen with adhesive or ethoxyline resin. In this way, specimen area is equal to the opening area on barrier board.

Again, testing temperature should be controlled. Temperature is a critical parameter affecting the measurement of O_2GTR. Strict temperature controlling can reduce the influence of temperature fluctuation on testing results.

7. Conclusion

ASTM D 3985 is the commonly used standard for film oxygen permeability testing using oxygen sensor at present. It is also the reference standard of coulometric analysis method in YBB00082003. A deep understanding of this standard will help the better implementing of YBB00082003. Temperature and humidity are critical parameters influencing final test data. That is why efficiency controlling of testing environment is a way to improve testing accuracy.