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Enactment of the National Standard on Electrolytic Sensor Method Water Vapor Permeability Testing

Abstract: this article details on the enactment background and progressing of electrolytic sensor method water vapor permeability testing standard. Based on actual demand of application in our country, it also introduces the calibration as well as the application advantages of this standard.
Key Words: water vapor permeability, electrolytic sensor method, calibration, and electrolytic cell

Test methods for water vapor permeability transmission rate through films can be divided into gravimetric method and sensor method. At present, only gravimetric method is used for domestic film water vapor permeability testing, which is not propitious for product evaluation of international trade. In addition, although electrolytic sensor method has been adopted in some industrial standards (for example: medical packaging standards), there is no corresponding international support, which also impedes the popularization of industrial standard. Comparing with gravimetric method, electrolytic method has more advantages and can better satisfy present test requirements on high barrier packaging materials, the enactment of corresponding test standards is of urgent necessity. In early 2007, the enactment starts formally. Now it has entered the standard approving stage.

1． The Enactment of Film Water Vapor Permeability Test Standard Using Electrolytic Sensor Method

As the natioanl packaging standardization committee specified in its requirment ‘about enactment and revision of naitional standard project’ , China package scientific and research testing center will undertake the standard enactment task entitled test method for water vapor trasnmission rate through plastic films and sheets-electrolytic sensor method. National package quality inspection center(Jinan) and Jinan Labthink mechanical and elctronic coopration are specially invited to draft this standrad. The standard making team, formed by technicians from these three institutes, drafted the recommending national standard proposal and submitted it to national package standardization committee after investigated, analyzed and discussed the significance, feasibility, the international and foreign standards involved as well as the social and economical benefits. Based on ISO15106-3:2003 *Plastics - Film and sheeting - Determination of water vapor transmission rate - Part 3: Electrolytic detection sensor method* and referring to the rich barrier property research experience of Labthink, the standard making group finished draft standard for comment that completely conforms to application characteristics of plastic packaging industry. After soliciting opinions from all domestic enterprises, colleges and industry experts, the draft standard for comment is revised for many times. Content of draft standard for approval was finalized on 24th July 2007 and the draft standard has been submitted for investigation. This standard will be formally issued within the next few days.

Test principle of electrolytic method is as below: electrolytic cell is used as humidity sensor. Permeation cell of instrument is divided into a dry cell and a humidity-controlled wet cell by film or sheet. Water vapor transmitting from wet cell through film into dry cell is carried away by carrier gas to electrolytic cell, where the electric signal being measured and output can be used to calculate transmission rate of water vapor through specimen.

2． Calibration of Electrolytic Sensor Method

Electrolytic sensor method, together with dynamic relative humidity testing method and infrared testing method, belongs to sensor method water vapor permeability testing. It was gradually introduced since 1970.

Requiring the same test environment as that of water method, electrolytic sensor method features rapid test speed, high resolution and less influencing factors. Therefore, this method is suitable for rapid determination of water vapor transmission rate through high barrier film, sheet and plastic-containing multi-layer structure materials.

However, due to sensor depletion or test accuracy, this instrument should be calibrated using gravimetric method test data periodically, which has been introduced in relating test standards. The calibration adopts reference film and the data of reference film is obtained according to gravimetric method. For example, In ASTM E 398-03, there is such description: “Standard Films, which have been calibrated by gravimetric means……A standard, calibration film whose WVTR has been gravimetrically determined…….”.

Because electrolytic sensor is of consumptive type, electrolytic sensor itself cannot maintain stable test data and therefore needs calibration periodically. For this reason, calibration is an important item for such test method. During standard enactment this time, there is detail explanation about the calibration of electrolytic sensor method. The experts think that to make test data of electrolytic sensor method comparable with that obtained from other method, this standard should specify requirement on traceability of test data. Therefore, in the ‘instrument’ chapter of this standard, it is stated that “Standard Films are used to calibrate instrument. Standard film is film with known WVT or the WVT of which is determined using gravimetric means’, which is consistent with the tractability requirements of ISO 15106-1:2003、ISO 15106-2:2003on test data. Such calibration can ensure that Water vapor permeability data obtained with electrolytic sensor method is consistent with that of gravimetric method.

Presently, according to the requirements of international water vapor testing standards, test data sensor methods should be calibrated by test data obtained with gravimetric means so as to secure the consistency. At the same time, since gravimetric method is the basic method for water vapor permeability testing, this can guarantee that tests performed under the same test environment can obtain the same test data. In this way, field test data of electrolytic sensor method can maintain consistency with that of international water vapor permeability test data.

3． Advantage of This Standard

Comparing with ISO 15106-3:2003, electrolytic sensor method national standard being made this time possesses the following advantages:

1. Humidity realizing method is added: in ISO 15106-3:2003, specified relative humidity is realized using sulfuric acid solution. However, the safety of using sulfuric acid solution in actual operation requires attention. Therefore, based on the content of original standard while referring to ASTM E 104-02 Standard Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions and DIN 53122.2（1982）Testing of Plastics Films, Elastomer Films, Paper, Board and Other Sheet Materials Determination of Water Vapor Transmission Rate electrolytic Method, this standard adds the method of using saturated saline solution to realize relative humidity.
2. Reduce the using of some restrictive conditions: for example, the electrolytic sensor method standard being made this time does not regulate operating voltage of electrolytic sensor and has relaxed restrictions on flow range and transmission area. At the same time, to make this standard more applicable, not only no particular specifications relating structure and components of test instruments is mentioned in this standard, categories of materials also increased. Such measures are beneficial to the application of new type materials and creative design of testing instruments, which paved the way for this standard to adapt to technological development.
3. Increased judging criteria of transmission equilibrium. As we all know, transmission of water vapor goes through a non-equilibrium state before reaching its equilibrium state. However, water vapor permeability

parameters must be measured under equilibrium state. Otherwise, error test data will be obtained. To specific film, the time required to reach equilibrium state from non-equilibrium state varies. For electrolytic sensor method, as long as the quantity of water vapor transmitting into sensor in per unit time does not exceed upper measuring limit of sensor, equilibrium process can be prolonged indefinitely until the sensor is depleted. Therefore, the end of test cannot be determined by length of test duration. If transmission equilibrium is estimated by human observation, accuracy of test data is lowered and repeatability of test data becomes poor. From the above statement we can see that it is necessary to set terms of transmission equilibrium like that of gravimetric method. Therefore, there is such statement relating the determination of transmission equilibrium in this standard: “ measure electrolytic current variation in fixed interval. When amplitude of three consecutive current fluctuations is not bigger than 5%, the current can be considered as stable and water vapor transmission enters equilibrium state. Actual application by Labthink has proved that using this method to determine transmission equilibrium and provide test data can greatly improve repeatability of test data and stability of instrument, thus avoiding error in judgment.

4. Introducing the definition of reference film: this standard increases requirements on traceability of test data. In addition to its reference to calibration methods of ISO and ASTM during standard enactment, the definition of reference is introduced for the first time. It is required that ‘calibrate instrument using reference film’. The using of reference film makes its calibration method more clear and simple. At the same time, this can ensure that test data of electrolytic sensor method are comparable to test data obtained with other methods.

4． Conclusion

Electrolytic sensor method is applicable to rapid determination of water vapor transmission rate through plastic film, sheet and plastic-containing multi-layer materials. The enactment of electrolytic sensor method standard not only enriches test methods of barrier property testing for domestic packaging materials, it can also better satisfy test requirements of high barrier packaging materials. Electrolytic sensor method strandard is formulated on the basis of ISO 15106-3:2003 with reference to the rich experience of barrier property tesing. It is believed that this stabdard can better adapt to actuall apllication requirment of our country.