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Issues Concerning Gas Permeability Test Standards of Sensor Method

Abstract: based on ISO and national standards of various countries concerning gas permeability testing, this article expounds on the issues that appear in the application of those standards. It also analyzes the disadvantages of some standards.
Key words: standards, sensor methods, gas permeability, calibration and oxygen sensor

Dated from 1970s in America, Sensor method is the most widely used approach of equal-pressure method. It most frequently used to test oxygen permeability of materials through oxygen sensor. ASTM issued ASTM 3985 standards for relevant test. With the development of international trade, sensor method gradually becomes popularized all over the world, which further riches gas permeability test method that is based on differential-pressure principle. To further supplement gas permeability testing standards, standards institutions of some countries began to introduce sensor method into their standard systems. ISO issued the equal-pressure method standard ISO 15105-2 containing sensor method in 2003. However, during the application of sensor method, problems in terms of calibration method, sensor type, standard universality and the start-stop point of test period are brought about. Based on abundant empirical tests, Labthink proposes corresponding analysis and solutions to the above mentioned problems as below:

1. Improve Calibration Method

It is not easy for operators to discover the consumption of oxygen sensor with time. Moreover, when the consumption reaches certain extent, data system of instrument should be calibrated. Therefore, a stable and effective calibration method is of vital importance.

In some sensor method gas permeability testing standards, Reference Material calibration is adopted as the calibration method. In this method, a film specimen with known oxygen permeability is tested by the instrument. Then the tested result is compared with the known oxygen permeability, by which whether the instrument works properly or whether the sensor is depleted can be decided. However, this method encounters some difficulties during actual application. First, not all standards institutions in the world take referential film as standard specimen and many countries are unable to obtain referential film that can be accepted by their national standard institutions, for which some sensor method instruments cannot obtain referential film for calibration. Secondly, the stability of referential film has time limit. Even Some imported instrument can obtain referential film from manufacturers; the cost is increased since they will depend on manufacturers for the long term requirement of referential film. Therefore, it is necessary to find a more efficient and wildly applied calibration method.

Problems relating referential calibration can be better solved when instruments are calibrated by gases with known concentration. Being the best method for oxygen sensor calibration, standard reference gas calibration is wildly used in micro determination of oxygen testing. It calibrates instruments through several standard gases (oxygen of certain concentration, PPM grade). Since the most important component of sensor method oxygen permeability testers is oxygen sensor, we can calibrate sensor method instrument with this method and solve the previously mentioned problems. First, standard reference gas can be obtained in local standard gases plants, thus the inconvenience of calibration source is solved. Next, the production of standard reference gas has a long history and its process has become very mature. With the accuracy of oxygen concentration, the authority and reliability of standard medium is no longer a problem. Therefore, if sensor method gas permeability test

standards adopt both referential film calibration and standard reference gas calibration, the obstacles of popularizing the application of sensor method can be completely eliminated. Moreover, standard reference gas calibration has already been used in German standard DIN 53380-3.

2. Increase Types of Oxygen Sensor

As what some people take delight in taking about, the sensor method ASTM D 3985 do has specification relating to the type of oxygen sensor. But such specification limits standard popularization to some extent. This is because on the one hand the specification impedes the application of new technology and on the other hand it provides conditions for market monopolization.

At present, the majority of sensor method gas permeability test standards have no requirements on the type of oxygen sensor. Some standards, such as ASTM D 3985, requires coulometric sensor, a coulometric device based on Faraday law producing linear signal according to the concentration of oxygen. During actual application of sensor method, the electrochemical sensor adopted by some manufacturers also belongs to this type. Electrochemical sensors can also realize accurate quantitative determination of oxygen. Although some people believe that coulometric sensor can completely absorb the oxygen, it is not possible. Even deoxidizer is not able to realize 100% oxygen eliminating. At the same time, if the sensor adsorbs great quantity of oxygen, testing elements inside will be depleted rapidly. Correspondingly, lifespan of the sensor is greatly shortened. Therefore, the complete adsorption of oxygen and longer lifespan of oxygen sensor are two contradictory aspects. The specification to sensor type in some standards has become an impediment for standard popularization. One thing worth special mentioning here is that in some newly issued sensor method standards, the specification to sensor type has already been cancelled.

3． Improve Standard Universality

Defining medium source appears to be a restriction to standard universality. Standards with big universality always attached importance to the final result while cared little about the actual means of application, thus provides wider room for users to design standards and to make breakthrough in the application of new technology. In these aspects, ISO enjoys the superiority.

Using specimens with one same origin is favorable for data comparison in a wider range. However, this should not become the excuse for restricting specimen source. Restricting specimen source may help some manufacturers benefit from monopolized business, For example, it is specified in ASTM D 3985 that the only source of alumina for the nominated catalyzer is the chemistry department of Englehard Industries Division and that of referential film is MOCON, Inc. Therefore, international standards should avoid having such specification. It is believed to scientific that standards only specify the required effect while permitting using specimens from other sources.

4． The Start-stop Point of Test period

It is a common problem that sensor method gas permeability test standards do not have clear definition to start-stop point of test period as well as the conditions to decide the end of test, which affects test methods directly mainly in two aspects: first, it is impossible to calculate test efficiency. The fact that Sensor method is believed to be highly efficient by some people is because they only take the time of test process into account while neglecting the purging period that may last several hours or even more than ten hours. On the other hand, since there are no clear conditions to judge the end of test, there is much randomicity.

Test period of sensor method divides into purging period and transmission period. System purging period

refers to the time needed in guiding carrier gas into sensor and keeping purging until the oxygen content of inner system reaches a very low state. Purging process is a very critical procedure in sensor method and can facilitate obtaining a higher test precision. There are clear requirements on purging period in testing standards, which is usually believed to be several hours. In developing sensor method oxygen permeability instruments, Labthink made a study on this topic and gets the conclusion as follows: system purging time has noting to do with material type and test item. To secure the purging effect, a period of several or more than ten hours is generally needed.

Transmission period is the time from the beginning of transmission to the equilibrium of transmission. If test time calculates from the beginning of system purging, there must be clear conditions provided in order to judge the end of test. Empirical tests have proved that the longer the test period, the more stable the test data will be. Test result obtained shortly after the test begins usually differs to some extent with that obtained after the transmission equilibrium. If operators decide the transmission equilibrium only by rule of thumb, the accuracy of test result will be obviously affected. Therefore, it is very essential to add specifications on how to judge the end of test. We propose that based on the determination method of transmission equilibrium in differential pressure method, users record the variation of sensor output signal internally. When the variation of three succeeding records do not exceeds certain percentage (the small the percentage, the strict the evaluation criteria; the small the variation, the more stable the data would be), it can be considered that this signal is constant signal and oxygen transmission has reached an equilibrium state. Record the current value of that time. Then calculate test results in accordance with the formula given by the standards.