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Notice of Barrier Property Testing and Instrument Usage

Abstract: This paper presented a summary of problems arising in proficiency testing project “Barrier property test of plastic packaging material——Determination of oxygen and water vapor transmission rate”, then gives out solutions and suggestions for the development of instruments and practical experiences.

Key Words: oxygen transmission rate, water vapor transmission rate, experimental conditions, gravimetric method, permeate equilibrium

The laboratories proficiency testing project “Barrier property test of plastic packaging material——Determination of oxygen and water vapor transmission rate” has been fulfilled. Through proficiency testing we investigated the barrier property testing capabilities and gained tremendous achievement. However, a number of widespread problems revealed. Some laboratories did not realize them fully and testing data from them were considered to be “outlier” and “suspiciousness”. In this paper we presented a summary to these problems.

1． Experimental conditions of barrier property testing

Some experimenters, got used to the routine tests, considered that a 23℃ temperature is suitable to barrier property testing. In fact, we must follow the standards of testing method and adopt experimental conditions when testing oxygen transmission rate and water vapor transmission rate. But sometimes there are not only one standard experimental conditions in a standard, take water vapor transmission rate testing for example, there are 5 standard experimental conditions in ISO 2528: 25±1℃, 90±2％RH; 38±1℃, 90±2％RH; 25±1℃, 75±2％RH; 23±1℃, 85±2％RH; 20±1℃, 85±2％RH. 5 experimental conditions in the series of ISO 15106: 25±0.5℃, 90±2％RH; 38±0.5℃, 90±2％RH; 40±0.5℃, 90±2％RH; 23±0.5℃, 85±2％RH; 25±0.5℃, 75±2％RH. Standard experimental conditions of GB/T 1037 are: 38±0.6℃, 90±2％RH; 23±0.6℃, 90±2％RH. Other standards such as ASTM E96 are not consistent with conditions above. Only experimental conditions of 38℃, 90％RH satisfy the demands of water vapor transmission rate testing, excluding the differences in errors of temperature and humidity. It is the same condition to oxygen transmission rate testing. So, generally considered all testing standards, we adopted 23℃, 0％RH in oxygen transmission rate testing and 38℃, 90％RH in water vapor transmission rate testing, and they are not consistent with the experimental condition of 23℃, 50％RH in GB/T 2918.

The changes in temperature and humidity affect the material and greatly influence barrier property testing data at the same time, so it is fundamental to carry out proficiency testing project according to national standard experimental conditions. The oxygen transmission rate testing is under condition of 23℃, 0％RH and 38℃, 90％ for water vapor transmission rate testing. Some laboratories did not follow the demands of experimental conditions and the results from them are suspicious, this would be solved as soon as those laboratories changed experimental conditions under direction.

2． Error analysis of instrument in non-automatic gravimetric method

67 laboratories took part in the proficiency testing project, 66 laboratories offered effective data, only 10

laboratories did not use gravimetric method instrument, 6 of them used infrared sensor instrument, and 4 of them used electrolytic detection sensor instrument. 2 laboratories in all which employ gravimetric method are considered to be outliers. From statistical result of testing data in the first total testing, we saw that most laboratories using gravimetric method instrument were considered to be outliers and suspiciousness, but through further investigating and classifying, we learned that there was a big difference between laboratories using instruments of non-automatic gravimetric method and automatic gravimetric method.

None of the laboratories using non-automatic gravimetric method instrument is interlaboratory outlier, but cover 50％ of interlaboratory suspicious laboratories employ non-automatic gravimetric method instrument. And laboratory employ non-automatic gravimetric method instrument make up 87.5％ of within-laboratory outliers and 62.5％ of within-laboratory suspiciousness. However, to the laboratory use automatic gravimetric method instrument, no one is outlier, only 3 laboratories are within-laboratory suspiciousness including the effects of experimental conditions. Because no laboratory using instrument based on gravimetric method is outlier, the systematic error of gravimetric method instrument is within the range of national recognition. But the testing precision of instruments with non-automatic gravimetric method was greatly influence by testing error, laboratories using those instruments cover 87.5% of within-laboratory outliers.

Gravimetric method is the basic method of water vapor transmission rate testing, its testing data is the foundation of other test methods. Testing error of desiccant method is inevitable, so the development of water method and using automatic gravimetric method instruments is the only solution at present. The testing error of instrument with non-automatic gravimetric method are not so obvious in the past because the lack of effective testing methods. However with the popularization of other automatic testing instruments, the shortages of non-automatic gravimetric method instrument become more obvious. Data from laboratories using with non-automatic gravimetric method instrument are not satisfying, 52.2％ of them are considered to be suspicious and even outlying, popular attentions were paid to the condition of instrument usage due to the surprising data. And this also helped institutes and labs in choosing barrier property testing instrument.

3． Stability estimation of oxygen transmission rate testing and experimental condition

The oxygen transmission rate testing method includes differential-pressure method and equal-pressure method, the differential-pressure method is more widely used. In this project of proficiency testing, 69 laboratories participated in oxygen transmission rate testing, only 13 of them using equal pressure method instruments. Many factors depending on different testing methods, would affect the test data of oxygen transmission rate testing.

The problem gained special attentions is the vacuum limitation of system and the time spend in vacuum pumping. Experiment demonstrates that, too short times of vacuum pumping could affect experimental results. On the one hand, it is because the gas from vacuum pipes was mixed into the permeation gas, this led to slightly large and instable of testing data. On the other hand, gas and impurity would infiltrate from the surface of material through the specimen has been pretreated, time spend on vacuum pumping would affect the degree of gas and impurity infiltration. The more thoroughly the vacuum is, the better the effect of exclusion and more stable the testing data. The volume of testing cavity is relevant to the time spending on vacuum pumping. Less time spend on smaller volume, but it is hard for vacuum method instrument to keep pressures if time spends on vacuum pumping is too short. The degree of vacuum and time spend on vacuum pumping is definitely prescribed in gas transmission rate testing, such as GB/T 1038 standard required 3 hours of non interrupted pumping after arrived

at the specified degree of vacuum (27Pa). Laboratories is suspicious and outlying caused by don't spend enough time on vacuum pumping would get satisfying testing data by prolonging time spending on vacuum pumping.

Special attentions need to be paid on purging and permeate equilibrium of testing system when equal-pressure method is used. System purging is to put carrier gas into oxygen sensor to reach the very low of oxygen content. Purging affects the precise of testing and is a key step in equal-pressure method. The time spend on purging which commonly considered to be several hours, is specifically prescribed. Different from differential-pressure method, the estimate of permeate equilibrium in equal-pressure method depends on the observation of experimenter at present. According to experiences, the longer the time spending on testing after entering testing status, the more stable the testing data is. Experimental results received at the beginning of experiment often different from the real testing data after arriving at permeate equilibrium. If it only depend on the observation of operators to determine the arrival of permeate equilibrium, the accuracy of testing data would be affected seriously and lead to the status that data obtained from permeate equilibrium differ from person to person. So we suggest to prolonging the time spending on testing especially for high barrier specimen.

4. Summary

There are many obvious problems in this proficiency testing as illustrated above. Since barrier property testing is a microscopic testing, we must pay attention to many other problems in test operation, for example specimen clamping, the time spending on purging in equal-pressure method, cup operation, specimen preparing, the sensors protecting and so on. It is optimistic that many problems have been solved in the proficiency testing, other problems also aroused attentions from operators. It is a good beginning of regular operating steps, reducing artificial error and unification of testing data.