

Solution to the Calibration Method of Equal-pressure Method Oxygen Permeability Tester

Abstract: This article elaborates on the calibration method of reference material calibration used by current equal-pressure method gas permeability testing, analyzes practical application situations of reference material calibration. At the same time, this article recommends another calibration method: standard gas calibration. It is pointed out that both reference material calibration and standard gas calibration should be adopted in equal-pressure method gas permeability testing, which can not only pave the way for popularizing equal-pressure method, but also reduce the use-cost.

Key Words: equal-pressure method, gas permeability, calibration, referential-film, standard gas

As we all know, oxygen sensor adopted in equal-pressure method will be depleted with time in terms of response time and response efficiency. During actual application, test data displays a reduce tendency with time. When the sensor is depleted, data system of instrument should be calibrated with standard materials. That is why the stability and convenience of calibration method is of significant importance.

Based on the application situations of current calibration method, this article discusses the problems of such calibration, introducing another method with better applicability.

1 . Present Status of Equal-pressure Method Calibration

At present, the commonly used calibration method adopted by equal-pressure method test standards is reference material calibration. This method uses a film with known oxygen permeability to calibrate instrument. Users can determine whether the instrument works properly or whether the sensor is depleted through comparing tested results with standard value of reference material. Detailed calibration procedures are as follows:

1. Ask instrument manufacturer for reference material. Service life of Reference material is usually within one year. Moreover, when being used repeatedly, reference material requires certain storage condition. The key point of reference material calibration is to obtain valid reference material.
2. Before testing, process reference material according to test requirements.
3. Place the specimen. Determine purging period according to the provided standard value and operation instruction of instrument. Then begin test.
4. When oxygen transmission reaches equilibrium, obtain test result.
5. Compare the tested result with standard value of reference material. Calculate calibration coefficient with

the following formula:

$$k = \frac{P_0}{P}$$

Where: k is the calibration coefficient. P_0 refers to standard value of oxygen permeability. P represents the tested value of film oxygen permeability. The calculation formula of calibration coefficient in some standards is the ratio of sensor output signal, which is the same with this formula in principle. Therefore, users can carry out equivalent conversion between formulas.

6. Apply the calibration coefficient to result calculation through program. Test results hereafter will contain calibration coefficient. In some standards, there may be such situation where calibration coefficient is not calculated but its ration form is directly applied to the calculation of test results.

Reference material calibration encounters some difficulties during actual application. First, not all institutions of standard materials in the world take reference material as standard specimen and many countries are unable to obtain reference material that is accepted by their national standard institutions, for which some sensor method instruments cannot obtain reference material for calibration. Secondly, the stability of reference material has time limit. Even if some imported instrument can obtain reference material from manufacturers, the cost is increased since they will depend on manufacturers for their long term requirement of reference material. Thirdly, at present, films that can obtain traceability of standard material institution are rather rare. Reference material calibration in actual application is carried out through single data calibration. Moreover, the adopted reference material belongs to medium barrier range, which may result in calibration error of high barrier and low barrier materials. Therefore, it is of great necessity to find a more efficient and wildly applied calibration method for current market in terms of convenience, accuracy and etc.

2 . Standard Gas Calibration

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 of reference material calibration.

In standard gas calibration, standard gases with different oxygen percentage are required. Generally, the gases are of 50ppm、100ppm、and 500ppm, which can be obtained in local standard gases plants and are relatively cheaper than reference material. Moreover, standard gases are valid in a long term. Detailed calibration procedures are as follows:

1. Fill system with high purity nitrogen gas (99.999%) and keep purging upper and lower chambers for about two hours.
2. Fill system with 50 ppm standard gas and purge upper and lower chambers simultaneously with the flow rates being 10ml/min. When output signal of the sensor becomes stable, select low side calibration. Observe whether output ppm value is consistent with standard value. If they are not consistent, adjust ppm value to the concentration value of filled standard gas. Then confirm calibration.
3. Fill system with 500 ppm standard gas and purge upper and lower chambers simultaneously with the flow rates being 10ml/min. When output signal of the sensor becomes stable, select high side calibration. Observe whether output ppm value is consistent with standard value. If they are not consistent, adjust ppm value to the concentration value of filled standard gas. Then confirm calibration.
4. Determine calibration coefficient with the formula below:

$$k = \frac{C_{ZERO}}{C_Z} = \frac{C_{END}}{C_E}$$

Where: k is calibration coefficient. C_{ZERO} refers to the concentration of standard gas at zero calibration. C_Z represents the output concentration of sensor at zero point calibration. C_{END} represents the output concentration of sensor at end point calibration.

5. The procedure after low side calibration and high side calibration is calibration verification. Fill system with high purity nitrogen gas and purge upper and lower chambers simultaneously for about two hours. Then fill system with 100 ppm standard gas and purge upper and lower chambers with a flow rate of 10ml/min. If sensor output (calibration finishes) is within 100 ± 3 ppm, the calibration is successful. Otherwise, find the reason of problem and recalibrate.
6. During calibration, standard gas with various oxygen concentrations can be adopted to increase the accuracy of calibration. If calibration data appear to be ideal each time, the newest calibration parameter will be used into test.

Advantages of standard gas calibration:

First, standard gas can be obtained in local standard gases plants, thus the inconvenience of calibration source is solved. Next, oxygen concentration of standard gas is indicated by ppm. ppm is a (10^{-6}) universal unit, thus solves the problem of unidentified units. Thirdly, the production of standard gas has a long history and the process has become very mature. With the accuracy and precision of oxygen concentration, the authority and reliability of standard medium is no longer a problem. **In addition, the calibration efficiency is rather high. Reference film calibration usually tests one specimen or one batch of specimens more than three times before providing medium value of tested data. According to test procedures of equal-pressure method, a period of about three days is required. Otherwise, the accuracy of test value cannot be secured. In spite of this, the calibration is only single data calibration of reference material. Generally, standard gas calibration (including calibration verification) only needs about ten hours, which can help reducing the influence of test environment during calibration process, thus greatly increases calibration precision. At present, standard reference gas calibration has gradually been used by equal pressure method gas permeability testing instruments. In German standard DIN 53380-3, there is already similar application.**

3 . Conclusion

In conclusion, both reference material calibration and standard reference gas calibration should be adopted by equal-pressure method gas permeability test, which can not only eliminate the obstacles of popularizing the application of equal-pressure method, but also obviously reduce the use-cost of equal-pressure method instruments.