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NATIONAL STANDARD OF THE

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YBB 60412012

Tests for Coefficient of Mean Linear Thermal Expansion

平均线热膨胀系数测定法

Tests for Coefficient of Mean Linear Thermal Expansion

This Method specifies a method for the determination of the mean linear thermal expansion coefficient of elastic solid glass, which is far below the transformation temperature.

This Method is applicable to the determination of the mean linear thermal expansion coefficient of pharmaceutical glass of various materials.

[Definition]

- (1) Mean linear thermal expansion coefficient $a(t_0; t)$

Within a certain temperature interval, the ratio of the length change of the test object to the temperature interval and the initial length of the test object is expressed in Formula (1):

$$a(t_0; t) = \frac{l}{L_0} \times \frac{L - L_0}{t - t_0} \quad (1)$$

Where,

t_0 ---the initial temperature or reference temperature, expressed in (°C);

t ---the actual temperature of the test object, expressed in (°C);

L_0 ---the length of the glass test object at the temperature t_0 during the test, expressed in (mm);

L ---the length of the test object at the temperature t , expressed in (mm).

This Method specifies that the nominal reference temperature t_0 is 20 °C, hence, the mean linear thermal expansion coefficient is expressed as $a(20\text{ °C}; t)$.

- (2) Transformation temperature t_g

This temperature represents the transformation of glass from a brittle state to a viscous state, which corresponds to the temperature at the intersection of the tangent lines of the high-temperature part and the low-temperature part of the thermal expansion curve.

[Instruments]

- (1) A device for measuring the length of the test object, with an accuracy of 0.1%.
- (2) Push rod-type dilatometer (horizontal or vertical), capable of measuring the length change of the test object $2 \times 10^{-5} L_0$ (i.e., 2 μm/100 mm)

The contact force of the length gauge shall not exceed 1.0 N. This force acts through the contact between the flat surface and the spherical surface. The radius of curvature of the spherical surface shall not be smaller than the diameter of the test object. In some special devices, parallel flat surfaces are required.

The device carrying the test object shall ensure that the test object is placed in a stable position. During the entire test process, the test object shall be on the same axis as the push rod shaft, so as to prevent any slight changes.

If the device carrying the test object is made of quartz glass, see the precautions given in Result Expression (2).

Standard materials shall be used for instrument performance testing. For the method, see Instrument Performance Testing.

(3) Heating furnace

The heating furnace shall be matched to the dilatometer device, and its upper temperature limit shall be about 50 °C higher than the expected transformation temperature. The working position of the heating furnace relative to the dilatometer shall have reproducibility within 0.5 mm in both the axial and radial directions.

Within the test temperature range (i.e., the upper temperature limit is 150 °C lower than the highest expected transformation temperature t_g , and is at least 300 °C), throughout the entire length of the test object, the furnace temperature shall be kept constant within ± 2 °C.

(4) The furnace temperature control device shall comply with the control requirement of a lifting rate of 5 °C $\pm$ 1 °C/min.

(5) Temperature measuring device

Within the temperature range of t_0 and t , the temperature of the test object can be accurately measured, and the error shall be less than ± 2 °C.

[Test Object]

(1) Shape and size

The test object is usually in the shape of a rod, and its shape depends on the type of the dilatometer used. The length L_0 shall be at least 5×10^4 times the length measurement resolution of the length measuring device of the dilatometer.

NOTE: for example, the test object can be a round rod with a diameter of 5 mm, or a square rod with a cross-section of 5 mm $\times$ 5 mm and a length of 25 ~ 100 mm. In some cases, a cross-section of 100 mm² is more convenient.

(2) Preparation

Before testing, the test object shall be annealed: heat the test object to approximately 30 °C higher than the transformation temperature, then, at a rate of 2 °C/min, cool the test object to approximately 150 °C below the transformation temperature; without ventilation, further cool the test object to room temperature.

(3) Quantity

For fast testing, test two test objects. See Result Expression (4).

[Determination Method]

(1) Selection of test range

The reference temperature (t_0) is 20 °C (18 ~ 28 °C is allowable), and the end temperature (t) is 300 °C (290 ~ 310 °C is allowable).

(2) Determination of reference length

At the reference temperature t_0 , measure the reference length L_0 of the annealed test object, with an accuracy of 0.1%, then, place the test object in the dilatometer and stabilize it for 5 min.

(3) Temperature-rise test

When the initial temperature is t_0 , determine the position of the dilatometer, and take this reading as the zero point of the uncorrected length change ΔL_{meas} to be measured. Then, set the furnace temperature control device to the required heating program to start heating. Record the temperature t and the corresponding length change ΔL_{meas} , until reaching the required end temperature. Unless it is otherwise specified, the heating rate shall not exceed 5 °C/min.

In terms of the recorded expansion reading ΔL_{meas} during the temperature rise period from t_0 to t , since there is a temperature difference between the hot junction of the thermocouple and the test object, a correction value shall be added to the apparent temperature of the test object.

NOTE: this correction value depends on the rate of temperature change and the rate of heat exchange between the heating furnace and the test object. Basically, the correction value is determined by comparison with the constant-temperature test.

(4) Constant-temperature test

When the initial temperature is t_0 , determine the position of the dilatometer, and take this reading as the zero point of the uncorrected length change ΔL_{meas} to be measured. Then, heat it, until the furnace temperature reaches the selected end temperature t , and maintain the furnace temperature constant within ± 2 °C. After 20 min, read the value of ΔL_{meas} from the dilatometer.

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