Attachment: 4022 Determination of Coefficient of Mean Linear Thermal Expansion for Glass

4022 Determination of Coefficient of Mean Linear Thermal Expansion for Glass

The coefficient of mean linear thermal expansion is an important physical characteristic of glass. It refers to the elongation per unit length of the glass as the temperature rises by 1℃ in a certain temperature range. This method specified the determination of the coefficient of mean linear thermal expansion of elastic solid glass well below the transition temperature. This method applies to the determination of the coefficient of mean linear thermal expansion of glass for pharmaceutical use with various materials.

Determination Principle

In this method, the sample of a certain length is heated to a certain temperature with the specified heating procedure. The elongation of the sample is measured after the temperature increases, and the coefficient of mean linear thermal expansion of the sample is calculated. It can be expressed as:

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

Where, t_0 is the initial temperature or the reference temperature, in ℃;

t is the temperature of the sample after being heated, in ℃;

L_0 is the length of the sample at the temperature of t_0 during the test, in mm;

L is the length of the sample at the temperature of t, in mm.

The temperature range of pharmaceutical glass product for linear thermal expansion coefficient index is 20-300℃, then the nominal reference temperature, t_0, is 20℃, and the terminal temperature of the sample, t, is 300 ℃, which can also be expressed as α(20℃; 300℃).

Instruments

(1)Measuring device (for example, calipers), with an accuracy of 0.1%.

(2)Push rod dilatometer (horizontal or vertical type) can measure the length variation of the sample for 2×10^{-5} L_0 (i.e., 2 μm/100 mm). The contact force of the length-measuring device shall not exceed 1.0 N. This force acts through the contact between the plane and the sphere. The radius of curvature of the sphere shall not be smaller than the diameter of the sample.Paralleled planes are required in some special devices.

The device used to support the sample shall ensure the sample in a stable position, and the sample shall be on the same axis with the push rod shaft during the test to prevent any minor changes.

(3)furnace

The heating furnace should match with the dilatometer, and its upper limit temperature shall be at least 50℃ higher than the expected transition temperature. The working position of the heating furnace relative to the dilatometer shall be reproducible within 0.5 mm in the axial and radial directions.

The furnace temperature shall be constant within ±1℃ within the range of test temperatures (i.e., the upper limit temperature shall be 150℃ lower than the highest expected transition temperature, t_g, and shall be at least 300℃). The furnace temperature control device shall meet the control requirements that the ramp rate is 5℃/min±1℃/min. In the temperature range of t_0 and t, the temperature of the sample can be accurately determined, and the error shall be within ±1℃.

To check whether the whole test instrument is in normal operation, the reference material for mean linear thermal expansion coefficient of glass (national pharmaceutical reference material) should be used to calibrate the instrument.

Sample Preparation Select a sample without obvious defects in appearance and cut into the shape and size required by the instrument by mechanical cutting or other processing means. (For example, the sample can be made into a round rod with a diameter of 5-6 mm and a length of 18-100 mm, or in other shape and size meeting the requirements of the instrument). The length, L_0, shall be at least 5×10^4 times of the resolution of the length-measuring device of the dilatometer.

The sample shall be annealed before the test: heat the sample to a temperature approximately 30℃ higher than the transition temperature, then cool it to a temperature approximately 150℃ lower than the transition temperature at a rate of 2℃/min, and further cool it to room temperature without ventilation.

Determination

(1)Selection of test temperature range

Based on the practical reasons, the nominal reference temperature is generally $18℃\leq t_0\leq 28℃$, and the terminal temperature is generally $290℃\leq t\leq 310$ ℃. The accuracy of both temperature and temperature difference readings shall be ±1℃, although the actual measured temperature should be used in the actual calculation of the result expression, the identification of the test range shall be indicated by the nominal temperature. For a given coefficient a(20℃;300℃) that is expressed in nominal temperature, as long as the actual temperature selected is within the specified limits, the effect on the coefficient can be negligible.

(2)Determination of the reference length

Measure the reference length L_0 of the annealed sample under the reference temperature t_0, then put the sample in the dilatometer to stabilize for more than 5 min.

(3)Test method I: temperature rising test

Determine the position of the dilatometer under the initial temperature of t_0 and take this reading as the zero point of the uncorrected length variation to be measured, ΔL_{meas}, and heat up the heating furnace following the desired heating procedure. Record the temperature t and the corresponding variation in length, ΔL_{meas}, until the desired terminal temperature is reached. Unless otherwise specified, the heating rate shall not exceed 5℃/min.

(4)Test method II: thermostatic test

Determine the position of the dilatometer under an initial temperature of t_0 and take this reading as the zero point of the uncorrected length variation to be measured, ΔL_{meas}. Then heat the furnace up to the selected terminal temperature t and keep the furnace temperature constant within t±1 ℃. After 20 min, read the value of ΔL_{meas} from the dilatometer.

Although the temperature rising test allows the coefficient $a(t_0;t)$ of various temperature t to be determined during the test, if only one terminal temperature t is required, a thermostatic test shall be preferred, for it can provide better accuracy.

Result Calculation and Representation

$$a(t_0;t)=\frac{1}{L_0}\times\frac{\Delta L_{meas}}{t-t_0}$$

Where, $a(t_0;t)$ is the coefficient of mean linear thermal expansion of the sample;

t_0 is the initial temperature or reference temperature, in ℃;

t is the temperature of the sample after being heated, in ℃;

L_0 is the length of the sample at the temperature of t_0 during the test, in mm;

ΔL_{meas} is the corrected length variation of the sample under the temperature t, in mm.

Due to the corresponding thermal expansion of the device that supporting the sample in the process of temperature rise, there is a temperature difference between the measuring point for temperature measuring and the sample in the process of heating up, so the measuring system of the instrument shall be corrected according to the method provided by the instrument.

Calculate $a(t_0; t)$ for the two samples. Generally, t_0 is 20℃, t is 300℃, and a is expressed as (20℃;300℃). If a(20℃;300℃)<$10\times10^{-6}K^{-1}$, round it off to two significant digits. If a(20℃;300℃)≥$10\times10^{-6}K^{-1}$, round it off to three significant digits.

If the deviation between the determination results of the two samples is not more than $0.2\times10^{-6}K^{-1}$, average the two results. Otherwise, the test must be repeated with two additional samples.

Drafted by: National Institutes for Food and Drug Control Contact number: 010-67095110