**4019 Determination of Thermal Shock and Thermal Shock Endurance for Glass Containers**

Thermal shock (also known as thermal-shock resistance) refers to the difference between the temperature at which the sample is heated (upper limit temperature *t*1) and the temperature of the cold water bath in which the sample is placed (lower limit temperature *t*2).

Thermal shock endurance refers to the temperature difference when 50% of the glass container samples are broken in thermal shock test.

This method applies to the determination of thermal shock and thermal shock endurance of glass containers for pharmaceutical use.

Depending on the temperature difference, the determination methods can be divided into cold and hot water baths method and oven method.

**Method I. Cold and hot water baths method**

This method applies to all kinds of glass containers for pharmaceutical use, whose test temperature difference is less than 100℃.

**Instruments:** Hot water bath: The capacity shall be at least twice the total volume of the samples in one test and shall not be less than 5 L. The water bath shall contain water circulators, temperature control components, and temperature regulating control heaters to keep the water temperature stable within the upper limit temperature *t1*±1℃.

Cold water bath: The capacity shall be at least five times the total volume of the samples in one test. The water bath shall contain water circulators, temperature control components, and thermostatic controllers to ensure that the water temperature is stable within the lower limit temperature *t2*±1℃.

Net basket: The material of the net basket (coated if necessary) shall not scratch or rub the samples during the test. The net basket shall be able to hold the glass samples upright and apart from each other, and be equipped with a device to hold the samples to prevent them from floating when immersed.

**Determination**

(1)The samples shall be the products that have not been subjected to tests for other properties (such as mechanical, thermal, etc.) (The samples shall be free of any glass defects) and be settled in the test environment for at least 30 minutes before the test to ensure that the temperature of the samples is consistent with the environment.

(2)Fill the two water baths (for cold and hot water, respectively) with water so that there is sufficient water to submerge the top of the containers by at least 50 mm, and then regulate the water temperature to t1 and t2, respectively. Generally, the water temperature t2 is 0℃-27℃, and the selected t1 shall be able to obtain the required thermal shock temperature difference t1-t2 (℃). The temperature difference between t1 and t2 shall not exceed ±1℃ of the specified value during the time when the samples placed in the net basket are transferred from the hot water bath to the cold water bath

(3)First place the samples in a net basket, make them stand upright and separate separated, then immerse them in a hot water bath at temperature t1, fill the samples with water, and keep the top of the bottle mouth at least 50 mm below the water surface, and allow to soak for at least 5 minutes to ensure that the temperature equilibrium between the samples and the water is reached.

Note: Experience has shown that the time required to reach temperature equilibrium depends on the maximum thickness of the samples. To ensure that both sides of the sample wall are heated, it takes at least 30 seconds per mm of wall thickness to reach temperature equilibrium.

(4)Transfer the samples filled with water in the net basket from the hot water bath to the cold water bath at the temperature of *t2* quickly. The transfer process of the samples must be completed within 10 s ± 2 s.

(5)The samples must be completely immersed in the water bath for 30 seconds. Then take out the net basket containing the samples from the cold water bath. The samples taken from the cold water bath shall be inspected immediately. Only those samples without breakage, crack or damage can be adjudicated as qualified products.

Notes: (1) The samples that have been confirmed to have no damage in the inspection shall not be used in other tests.

(2) If the temperature of the hot water bath has risen to 95℃, but the test is not finished, the test can be continued by lowering the temperature of the cold water bath.

**Method II. Oven method**

This method applies to all kinds of glass containers for pharmaceutical use whose test temperature difference is 80℃ or higher.

**Instruments:** Oven: the temperature can reach at least 300℃, and equipped with air stirrers or circulators, to ensure that the temperature variation does not exceed ±5℃. The oven must be equipped with an automatic temperature regulator. It can keep the temperature fluctuation within ±1℃ up to 180℃ and can keep the temperature fluctuation within ±2℃ at 180-300℃.

Cold water bath: With the same requirements for cold water bath as Method I. Net basket: With the same requirements for net basket as Method I. Clamps: the heads are wrapped with insulation materials, keep dry when in use.

**Determination**

(1)The samples shall be the products that have not been subjected to tests for other properties (such as mechanical, thermal, etc.). Before the test, the samples shall be put in an oven preheated to the upper temperature *t1*, and then the samples shall be kept at this temperature for at least 30 minutes to ensure that the temperature of the samples reaches equilibrium with that of the oven.

Note: Experience has shown that the time required to reach temperature equilibrium depends on the maximum thickness of the samples. To ensure that both sides of the sample wall are heated, it takes at least 6 minutes per mm of wall thickness to reach temperature equilibrium.

(2)Use the clamps with insulated heads to take out the samples from the oven. If two or more samples are tested at one time, remove the net basket containing the samples from the oven and completely submerge the samples (or together with the net basket) in a cold water tank to half of the height (in the case of bottles with necks, half of the total height excluding the necks) and hold for 30 s. The cold water bath shall be close to the oven and kept at the lower temperature *t2* of 0-27℃. The difference between this value of temperature difference and the temperature difference required when the samples are placed into the cold water bath shall not be greater than ±3℃. The transfer process of each sample must be completed within 5 s ± 1 s.

Note: The transfer process refers to the period from when the oven is being opened until the samples are immersed in cold water.

(3)The samples taken from the cold water bath shall be inspected immediately. The samples without breakage, crack or damage can be adjudicated as qualified.

Note: The samples that have not been damaged in the inspection shall not be used in other tests.

**Result Evaluation**

Thermal shock: After thermal shock test with specified temperature difference *t*1 and *t*2, if the number of broken samples is less than the specified number, it is adjudicated to be qualified.

Thermal shock endurance: Expressed as the temperature difference when 50% of the samples are broken by repeating the test with a temperature increment of 5-10℃ each time following the above test steps. If the temperature difference meets the specified requirements, it is adjudicated to be qualified. The value of temperature difference can be obtained from the curve drawn with the cumulative percentage of broken vs. the corresponding temperature difference.

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