# Attachment: 4019 Determination of Thermal Shock and Thermal Shock Endurance for Glass Containers

## 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 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.

Based 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 of 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 heaters for temperature regulation to keep the water temperature stable within the upper limit temperature $t_1$ ±1℃.

Cold water bath: The capacity shall be at least five times of 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 $t_2$ ±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 separated from each other, and be equipped with a device to fix the samples and prevent them from floating when immersed.

### Determination

(1)The samples, which shall be free from any glass defects, should be untested for other properties (such as mechanical, thermal properties, etc.), and be placed in the test environment for at least 30 minutes in advance to ensure that the temperature of the samples is consistent with the environment.

(2)Fill the two water baths (for cold and hot water bath, respectively) with sufficient water to submerge the top of the containers by at least 50 mm, then regulate the water temperature to $t_1$ and $t_2$, respectively. Generally, the water temperature of $t_2$ is 0℃-27℃, and the selected $t_1$ shall be able to obtain the desired thermal shock temperature difference $t_1$-$t_2$ (℃). The temperature difference between $t_1$ and $t_2$ shall not exceed ±1℃ of the specified value during the process where samples, placed in a net basket, are transferred from the hot water bath to the cold water bath

(3)First place the samples in a net basket, keep them upright and separated, then immerse them in the hot water bath with a temperature of $t_1$, fill the samples with water, and keep the top of the bottle mouth at least 50 mm below the water surface. Allow the samples to soak for at least 5 minutes to ensure that the temperature equilibrium is reached between the samples and the water.

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 with a temperature of $t_2$ quickly. The transfer process of the samples must be completed within 10 s ± 2 s.

(5)The samples immersed in the cold water bath for 30 seconds. Then take out the net basket, together with 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.

The qualified samples shall not be used in other tests.If the temperature of the hot water bath has reached to 95℃ during the test, 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 to 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, which can keep the temperature fluctuation within ±1℃ when the temperature is below or equal to 180℃, and can keep the temperature fluctuation within ±2℃when the temperature is at 180-300℃.

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

**Determination**

(1)The samples should be untested for other properties (such as mechanical, thermal properties, etc.) and be placed in an oven preheated to the upper temperature, $t_1$, for at least 30 minutes to ensure that the temperature of the samples reaches equilibrium with that of the oven.

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, or take out the net basket together with samples from the oven if two or more samples are tested at the same time. Submerge the samples (or together with the net basket) in the cold water tank to half of the height (for 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, $t_2$, at 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.

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.

The qualified samples shall not be used in other tests.

**Result Evaluation**

Thermal shock: After thermal shock test with specified temperature $t_1$ and $t_2$, if the number of broken samples is less than the specified number, it is adjudicated as qualified.

Thermal shock endurance: Expressed as the temperature difference at which 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 as qualified. The value of the temperature difference can be obtained from the curve drawn with the cumulative percentage of broken vs. the corresponding temperature difference.

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