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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Tests for Resistance to Attack of Glass by Boiling Hydrochloric Acid

玻璃耐沸腾盐酸浸蚀性测定法

Tests for Resistance to Attack of Glass by Boiling Hydrochloric Acid

This method is suitable for the determination of resistance to attack of various types of pharmaceutical glass containers and pipes by boiling hydrochloric acid.

According to the different principles and methods of observation, it is divided into gravimetric method and flame emission or atomic absorption spectrometry.

Method One: gravimetric method

This method is to etch a glass sample of approximately 100 cm^2 in boiling $6 \text{ mol/L} \pm 0.2 \text{ mol/L}$ hydrochloric acid solution for 6 hours. Determine the mass lost per unit surface area.

Preparation of test sample

Cut this product into glass tubes or pieces with regular geometric shapes and a total surface area of $100 \text{ cm}^2 \pm 10 \text{ cm}^2$. The cross section is finely ground. Do not use fire for polishing. The newly cut surface area shall not exceed approximately 10% of the total surface area. Wash with water. Then rinse with absolute ethanol or acetone. Dry in oven at 150°C for 45 minutes. Transfer to desiccator and cool for 45 minutes. Weigh accurately to ± 0.1 mg. Record the mass (m_1) . Prepare 2 test samples in the same way.

When measuring the acid resistance of glass materials, the influence of surface structure shall be considered. The test sample must be pre-treated with mixed acid. Its operation is as follows: Place a test sample in a plastic beaker. Add hydrofluoric acid (40%) - 2 mol/L hydrochloric acid (1:9) mixed solution to completely submerge it. Stir with a magnetic stirrer for 10 minutes. Use tweezers (the head is wrapped with plastic or platinum; treat it with dilute hydrochloric acid before use; then wash it with water) to take out the test sample. Wash, dry, cool and weigh as described above.

Determination method

In a wide-mouthed Erlenmeyer flask, add 800 ml of 6 mol/L \pm 0.2 mol/L hydrochloric acid solution. Heat to boil. Take the test sample and suspend it in the boiling acid with platinum wire (the test sample shall be completely submerged and suspended in the center). Install a condenser above the bottle mouth. After boiling evenly for 6 hours (see Figure 1), take out the test sample. Use water to rinse it clean. Dry it for 45 in a 150°C oven. Transfer to desiccator and cool for 45 minutes. Accurately weigh it to 0.1 mg. Record the mass (m₂).

Calculate the average of the results obtained from the two test samples. If the error between the two results and the average value is greater than 10%, two more test samples must be taken and re-measured.

Grading is based on half of the mass loss in milligrams per square decimeter of the test product. See Table 1:

Method Two: flame emission or atomic absorption spectrometry

This method is to etch a $30\sim40~\rm cm^2$ test sample in a $100\rm ^{\circ}C$ $6\rm mol/L \pm 0.2\rm mol/L$ hydrochloric acid solution for 3 hours. Determine the amount of alkaline oxide precipitated per unit surface area.

Preparation of test sample

Cut this product into glass tubes or sheets with regular geometric shapes and a total surface area of 30~40 cm². Finely grind the cross-section. Wash with water. Finally rinse with absolute ethanol or acetone. Dry in an oven at 115°C for 30 minutes. Prepare three test samples in the same way.

When measuring the acid resistance of glass materials, the influence of surface structure shall be considered. The test sample must be pretreated with mixed acid. The operation is as follows: Put the test sample in a plastic beaker. Add hydrochloric acid (40%) - 2mol/L hydrochloric acid (1:9) mixed solution to completely submerge it. Stir with a magnetic stirrer for 10 minutes. Use tweezers (the head is wrapped with plastic or platinum; treat it with dilute hydrochloric acid before use; then wash it with water) to take out the test sample. Wash and dry as described above.

Instruments

Flame photometer or atomic absorption spectrometer

Analytical balance: resolution is 0.1 mg

Determination method

Use tweezers to place the three washed test specimens into three polyethylene-covered dishes. Cover and place in oven at 115°C. At the same time, put in three polyvinyl

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