

## 4223 Determination of Specific Residues for Silicone Rubber Closures

This method applies to the determination of specific residues derived from formula and process in silicone rubber closures.

**Phenylated compounds** Take 2.0 g, accurately weighed, add 100 ml of n-hexane, and weigh. Boil under a reflux condenser for 4h, cool to room temperature, and make up for weight loss with n-hexane. After rapid filtration with a G3 or G4 sintered funnel, take the subsequent filtrate as the test solution. Prepare the blank solution using the same manner. Take the above two solutions, determine the maximum absorbance at a wavelength of 250 -340 nm according to the Ultraviolet-Visible spectrophotometry method (General Chapter 0401).

**Nonvolatile substance in n-hexane** Take 25.0 ml of test solution and blank solution respectively obtained in the test for **phenylated compounds** in an evaporator with constant weight, evaporate to dryness on a water bath or by electrical heating, dry at 105 °C for 1 hour and then weigh, calculate the difference in weight between the two.

$$X = m_1 - m_0$$

Where,  $m_0$  is the weight of non-volatile substance in blank solution, mg;

$m_1$  is the weight of non-volatile substance in test solution, mg.

**Volatile substance** Weigh 5.0g of the test sample previously stored for 48 h in a desiccator over anhydrous calcium chloride R. place in a weighing bottle with constant weight, dry at 200 °C for 4 hours, weigh accurately, and calculate the weight loss.

$$X (\%) = \frac{(m_0 + m_b) - m_1}{m_0} \times 100\%$$

Where, X is the percentage of weight loss, %;

$m_0$  is the initial weight of the specimen, g;

$m_b$  is the weight of the weighing bottle with constant weight, g;

$m_1$  is the total weight of the specimen and weighing bottle after heating drying, g;

**Mineral oil** Take 2.0 g in a conical flask with a stopper, add 30 ml of a mixture of ammonia-pyridine (5:95), shake out for 2 hours, filter, take the subsequent filtrate in a Nessler tube, and observe whether it shows fluorescence under a 365 nm ultraviolet lamp. If it shows fluorescence, compare the fluorescence with that of 0.005 mol/L sulfuric acid solution containing 10 µg quinine sulfate per ml, the fluorescence shall not be more significant.

**Peroxide (applies to silicone rubber closures with peroxide as catalyst)** Take 5.0 g, add 150 ml of dichloromethane, seal and mechanically stir for 16 hours. Quickly filter and collect the filtrate in an iodine flask. Fill the flask with nitrogen, add 1 ml of 20% sodium iodide glacial acetic acid solution (prepare the solution just before use), plug to seal, fully shake out, and leave for 30 minutes in the dark. Add 50 ml of water, use 0.25 ml of starch solution as an indicator, and immediately titrate with sodium thiosulfate titration solution (0.01 mol/L) until the color of the water layer fades away. Prepare the blank solution using the same manner and calculate the difference between the titration solution consumption of the sample and in the blank test.

---

Drafted by: Sichuan Institute for Drug Control (Sichuan Medical Device Testing Center) Contact number: 028-64020264

Participants: Shanghai Food and Drug Packaging Materials Testing Institute, Jiangsu Best New Medical Material Co., Ltd., Shandong Institute of Medical Device and Drug Packaging Inspection, China National Pharmaceutical Packaging Association