4223 Determination of Specific Residues for Silicone Rubber Closures

This method applies to the test 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, dry at 105 ℃ for 1 hour and then weigh, calculate the difference in weight between the test solution and blank solution.

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 ℃ 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, add 0.25 ml of starch indicator solution, 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. Calculate the difference between the sodium thiosulfate titration solution (0.01 mol/L) consumption of the sample and in the blank test.

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