

4222 Determination of Silicone Oil on the Surface of Rubber Closures

Rubber closures need to be siliconized to increase lubricity, so as to reduce particles generated by friction during storage and transportation, and facilitate packing and cork pressing. Silicone oil is a widely used and excellent rubber closure lubricant, among which the most commonly used is dimethyl silicone oil. In this method, the principle of infrared spectroscopy is used for determination. The dimethyl silicone oil has obvious Si-CH₃ bending vibration characteristic absorption peak at the wave number of (1260±10) cm⁻¹. The absorption peak height of this characteristic wave number is used for determination.

This method applies to the determination of the surface silicone oil content of rubber closure.

Apparatus and device Fourier transform infrared spectrometer is used for determination. The apparatus shall meet the requirements of General Chapter 0402 and shall be equipped with liquid cell accessories.

Preparation of reference solution Take about 0.5 g of dimethyl silicone oil (recommended kinematic viscosity: 300 - 1000 mm²/s), accurately weighed, in a 25 ml volumetric flask, dissolve with cyclohexane and dilute to the scale, shake well, and use as the reference stock solution. Take the appropriate amount of the reference stock solution and dilute quantitatively with cyclohexane to produce solutions containing about 0.3 mg, 0.5 mg, 1.0 mg, 2.0 mg, 4.0 mg and 10.0 mg per ml respectively, as the reference solution (if necessary, the linear range can be adjusted according to the actual situation of the test sample).

Preparation of sample solution Take the test sample (with a surface area of no less than 200 cm²), add isopropyl alcohol in the ratio of surface area to volume of isopropyl alcohol as 2.5cm²/ml, soak for 5 minutes, shake, transfer the solution to a heart-shaped bottle. Wash the rubber closure with 20ml of isopropyl alcohol by swinging, merge the wash liquid into the heart-shaped bottle, dry up with rotation at 65 °C, add an appropriate amount of cyclohexane to dissolve, transfer to a 5ml volumetric flask, dilute with cyclohexane to scale, shake well, and use as the sample solution. Prepare the blank solution using the same manner.

Determination The transmission method (the Method I of General Chapter 4002) or attenuated total reflection method (the Method II of General Chapter 4002) is used for determination. Inject the reference solution and the sample solution into a liquid cell and seal. Use Cyclohexane as the background to scan the infrared spectrum of the reference solution and the sample solution at the wave number of 4000-700 cm⁻¹ with a resolution of 4 cm⁻¹. Calculate the absorbance (measured by the peak height) near the wave number of 1260 cm⁻¹, and draw the standard curve. Calculate the surface silicone oil content of the rubber closure according to the following formula.

$$X = \frac{c \times V}{n} \times 1000$$

Where, X is the silicone oil content of the sample, µg/piece;

c is the concentration of silicone oil in the sample solution obtained from the standard curve, mg/ml;

V is the re-dissolved volume of cyclohexane, ml;

n is the sample quantity of rubber closures, piece.

or

$$X = \frac{c \times V}{n \times S} \times 1000$$

Where, X is the silicone oil content of the sample, µg/cm²;

- 43 c is the concentration of silicone oil in the sample solution obtained from the standard
44 curve, mg/ml;
45 V is the re-dissolved volume of cyclohexane, ml;
46 n is the sample quantity of rubber closures, piece;
47 S is the surface area of rubber closures, cm²/piece.
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