

4221 Determination of Water for Rubber Closures

This method applies to the determination of water in rubber closures.

Method I (Oven-drying method)

Take no less than 10 test samples, cut an appropriate amount from the crown of each sample, quickly cut into pieces of no more than 3 mm×3 mm ×3 mm in size, mix well, take 2 - 5 g, lay flat in a flat weighing bottle dried to constant weight, with a total thickness of no more than 5 mm, weigh accurately, and dry at 110 °C for 5 hours. Take out and transfer to a desiccator, cool, weigh accurately. Calculate the water content (%) in the sample based on the weight lost.

Method II (Karl Fischer drying furnace-Coulometric titration method)

1. Direct method

The test shall be performed under the temperature of 23°C±2°C and the relative humidity of 50%±5%. Take no less than 10 samples, cut at least one piece vertically from the crown of each sample, each piece of appropriately 4 - 7mm in length. Take all the pieces and accurately weigh an appropriate amount (with water content of about 0.5 - 5 mg) into a Karl Fischer drying furnace and determine at 140 °C ± 2 °C. Titrate to end point using the dead-stop titration (General Chapter 0701). Perform a blank test. Calculate or read directly from the instrument the water content of the sample, where each 1 mg of water corresponds to 10.72 Coulomb.

2. Extrapolation method

The test shall be performed under the temperature of 23°C±2°C and the relative humidity of 50%±5%. Take no less than 10 samples, cut at least one piece vertically from the crown of each sample, each piece of appropriately 4 - 7mm in length. Take all the pieces and accurately weigh an appropriate amount (with water content of about 0.5 - 5 mg) into a Karl Fischer drying furnace and determine at 140 °C ± 2 °C. Record the curve of water content increasing with time until the slope of the curve approach the constant. Take the data at five time points (such as 90, 85, 80, 75, and 70 minutes) from the constant slope of the curve, draw the water content curve with the test time as the X-axis and the water content as the Y-axis, and the intercept is the water content of the sample. Perform a blank test. Calculate using the following equation:

$$\text{Water in sample (\%)} = \frac{m_1 - m_0}{m \times 1000} \times 100\%$$

Where, m is the weight of the sample, in mg;

m_0 is the blank water content determined by extrapolation method, in μg ;

m_1 is the water content of the sample determined by extrapolation method, in μg ;

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