

Each mL of 0.005 mol/L silver nitrate VS
= 0.6345 mg of I

(3) Fluorine

Apply a small amount of water to the upper part of A, pull out C carefully, transfer the test solution and the blank solution to 50 mL volumetric flasks separately, wash C, B and the inner side of A with water, add the washings and water to make 50 mL, and use these solutions as the test solution and the correction solution. Pipet the test solution (V mL) equivalent to about 0.03 mg of fluorine, V mL of the correction solution and 5 mL of standard fluorine solution, transfer to 50-mL volumetric flasks separately, add 30 mL of a mixture of alizarin complexone TS, acetic acid-potassium acetate buffer solution, pH 4.3 and cerium (III) nitrate TS (1:1:1), add water to make 50 mL, and allow to stand for 1 hour. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using a blank prepared with 5 mL of water in the same manner. Determine the absorbances, A_T , A_C and A_S , of the subsequent solutions of the test solution, the correction solution and the standard solution at 600 nm.

Amount (mg) of fluorine (F) in the test solution
= amount (mg) of fluorine in 5 mL of

$$\text{the standard solution} \times \frac{A_T - A_C}{A_S} \times \frac{50}{V}$$

Standard Fluorine Solution: Dry sodium fluoride (standard reagent) in a platinum crucible between 500°C and 550°C for 1 hour, cool it in a desiccator (silica gel), weigh accurately about 66.3 mg of it, and dissolve in water to make exactly 500 mL. Pipet 10 mL of this solution, and dilute with sufficient water to make exactly 100 mL.

(4) Sulfur

Apply a small amount of water to the upper part of A, pull out C carefully, and wash C, B and the inner side of A with 15 mL of methanol. To this solution add 40 mL of methanol, then add exactly 25 mL of 0.005 mol/L barium perchlorate VS, allow to stand for 10 minutes, add 0.15 mL of arsenazo III TS with a measuring pipet, and titrate with 0.005 mol/L sulfuric acid VS. Perform the test with the blank solution in the same manner.

Each mL of 0.005 mol/L barium perchlorate VS
= 0.1603 mg of S

43. Paper Chromatography

Paper Chromatography is a method to separate each ingredient by developing a mixture in a mobile phase, using a sheet of filter paper, and is used for identification, purity test, etc. of substances.

Procedure

Unless otherwise specified, proceed by the following method.

Designate a line about 50 mm distant from the bottom of a sheet of filter paper, 20 to 30 mm wide and 400 mm long, as the starting line, spot the directed amount of the sample solution in the monograph with a micropipet or capillary tube on the center of the starting line, and air-dry. Then, suspend the paper in a container for developing of about 500

mm in height, which contains the developing solvent in its bottom section beforehand and the inside of which is already saturated by the vapor of the solvent, taking care to avoid contact with the walls. Immerse the paper in the solvent so that the lower edge of paper is covered with the solvent to about 10 mm from the bottom. Seal the container and allow the solvent to ascend on the paper at ordinary temperature.

When the solvent front has ascended from the starting line to the distance directed in the monograph, remove the paper from the container, make the solvent front immediately, and air-dry again. Observe the location, color, etc. of the spots by the method specified in the monograph. Calculate the R_f value by using the following equation:

$$R_f = \frac{\text{distance from the starting line to the center of the spot}}{\text{distance from the starting line to the solvent front}}$$

44. Particle Size Distribution Test for Preparations

Particle Size Distribution Test for Preparations is a method to determine the particle size distribution of the granules and powders described in General Rules for Preparations.

Procedure

(1) Granules

The test is performed employing No. 10 (1700 μm), No. 12 (1400 μm), and No. 42 (355 μm) sieves with the inside diameter of 75 mm.

Weigh accurately 20.0 g of granules to be tested, and place on the uppermost sieve which is placed on the other sieves described above and a close-fitting receiving pan and is covered with a lid. Shake the sieves in a horizontal direction for 3 minutes, and tap slightly at intervals. Weigh the amount remaining on each sieve and in the receiving pan.

(2) Powders

The test is performed employing No. 18 (850 μm), No. 30 (500 μm), and No. 200 (75 μm) sieves with the inside diameter of 75 mm.

Weigh accurately 10.0 g of powders to be tested, and place on the uppermost sieve which is placed on the other sieves described above and a close-fitting receiving pan and is covered with a lid. Shake the sieves in a horizontal direction for 3 minutes, and tap slightly at intervals. Weigh the amount remaining on each sieve and in the receiving pan.

45. pH Determination

pH is defined as the reciprocal of the common logarithm of hydrogen ion activity, which is the product of hydrogen ion concentration and the activity coefficient. Conventionally it is used as a scale of hydrogen ion concentration of a sample solution.

pH of a sample solution is expressed by the following equation in relation to the pH of a standard solution (pHs), and can be measured by a pH meter using a glass electrode.

$$\text{pH} = \text{pH}_s + \frac{E - E_s}{2.3026 RT/F}$$