is c_T (mOsm), can be calculated by means of the following equation,

$$\text{Osmolar ratio} = \frac{c_T}{c_S}$$

c_S: 286 mOsm

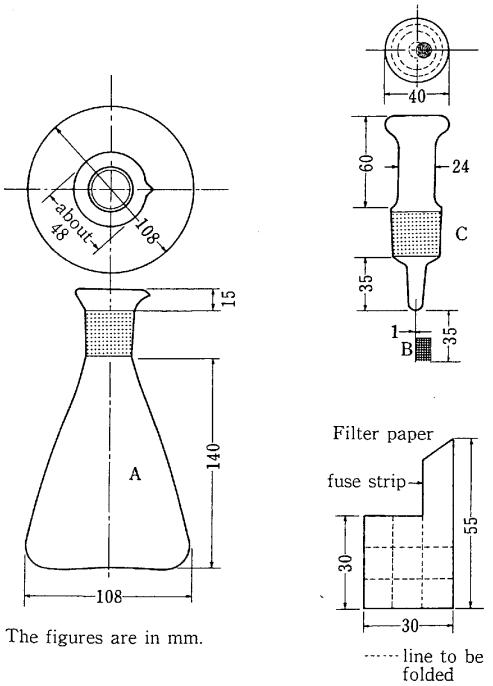
When the measurement is done by the dilution method, because the sample has an osmolarity over 1000 mOsm, the apparent osmolarity of the sample solution c_T can be calculated as $n \cdot c'_T = c_T$, in which n is the dilution number and c'_T is the measured osmolarity for the diluted solution. In this calculation, a linear relation between osmolarity and solute concentration is assumed. Thus when the dilution measurement is performed, the dilution number must be stated as (1 in n).

42. Oxygen Flask Combustion Method

The Oxygen Flask Combustion Method is a method for the identification or the determination of halogens or sulfur produced by combusting organic compounds, which contain chlorine, bromine, iodine, fluorine or sulfur, in a flask filled with oxygen.

Apparatus

Use the apparatus shown in the figure.



The figures are in mm.

A: Colorless, thick-walled (about 2 m), 500-mL hard glass flask, the upper part of which is made like a saucer. A flask made of quartz should be used for the determination of fluorine.

B: Platinum basket or cylinder made of platinum woven gauge. (It is hung at the end of the stopper C with platinum wire.)

C: Ground stopper made of hard glass. A stopper made of quartz should be used for the detemination of fluorine.

Preparation of test solution and blank solution

Unless otherwise specified, prepare them by the following method.

(1) Preparation of sample

(i) For solid samples: Place the quantity of the sample specified in the monograph on the center of the filter illustrated in the figure, weigh accurately, wrap the sample carefully along the dotted line without scattering, and place the parcel in a platinum basket or cylinder B, leaving its fuse-strip on the outside.

(ii) For liquid samples: Roll a suitable amount of absorbent cotton with filter paper, 50 mm in length and 5 mm in width, so that the end part of the paper is left to a length of about 20 mm as a fuse-strip, and place the parcel in a platinum basket or cylinder B. Place the sample in a suitable glass tube, weigh accurately, and moisten the cotton with the quantity of the sample specified in the monograph, bringing the edge of the sample in contact with the cotton.

(2) Method of combustion

Place the absorbing liquid specified in the monograph in flask A, fill it with oxygen, moisten the ground part of the stopper C with water, then ignite the fuse-strip, immediatey transfer it to the flask, and keep the flask airtight until the combustion is completed. Shake the flask occasionally until the white smoke in A vanishes completely, allow to stand for 15 to 30 minutes, and designate the resulting solution as the test solution. Prepare the blank solution in the same manner, without sample.

Procedure of determination

Unless otherwise specified in the monograph, perform the test as follows.

(1) Chlorine and bromine

Apply a small amount of water to the upper part of A, pull out C carefully, and transfer the test solution to a beaker. Wash C, B and the inner side of A with 15 mL of 2-propanol, and combine the washings with the test solution. To this solution add 1 drop of bromophenol blue TS, add dilute nitric acid dropwise until a yellow color develops, then add 25 mL of 2-propanol, and titrate with 0.005 mol/L silver nitrate VS according to the potentiometric titration under the Electrometric titration. Perform the test with the blank solution in the same manner, and make any necessary correction.

Each mL of 0.005 mol/L silver nitrate VS
= 0.17727 mg of Cl
Each mL of 0.005 mol/L silver nitrate VS
= 0.39952 mg of Br

(2) Iodine

Apply a small amount of water to the upper part of A, pull out C carefully, add 2 drops of hydrazine hydrate to the test solution, put C on A, and decolorize the solution by vigorous shaking. Transfer the content of A to a beaker, wash C, B and the inner side of A with 25 mL of 2-propanol, and transfer the washings to the above beaker. To this solution add 1 drop of bromophenol blue TS, then add dilute nitric acid dropwise until a yellow color develops, and titrate with 0.005 mol/L silver nitrate VS according to the Potentiometric tiration under the Electrometric Titration. Perform the test with the blank solution in the same manner, and make any necessary correction.

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 accuraly 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. Perfrom 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 stpots by the method specified in the monograph. Calculate the Rf value by using the following equation:

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

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.

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