

(3) Determine the infrared absorption spectrum of Potassium Canrenoate, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(4) The solution of Potassium Canrenoate (1 in 10) responds to the Qualitative Tests (1) for potassium salt.

**Optical rotation**  $[\alpha]_D^{20}$ :  $-71 - -76^\circ$  (after drying, 0.2 g, methanol, 20 mL, 100 mm).

**pH** Dissolve 1.0 g of Potassium Canrenoate in 20 mL of water: the pH of this solution is between 8.4 and 9.4.

**Purity (1)** Clarity and color of solution—Dissolve 0.5 g of Potassium Canrenoate in 5 mL of water: the solution is clear, and shows a pale yellow to light yellow color.

(2) Heavy metals—Proceed with 2.0 g of Potassium Canrenoate according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(3) Arsenic—Prepare the test solution with 1.0 g of Potassium Canrenoate according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(4) Canrenone—Place 0.40 g of Potassium Canrenoate in a glass-stoppered centrifuge tube, cool in ice-water to a temperature not higher than  $5^\circ\text{C}$ , add 6 mL of boric acid-potassium chloride-sodium hydroxide buffer solution, pH 10.0, being cooled to a temperature not higher than  $5^\circ\text{C}$  to dissolve, and add 8 mL of water being cooled to a temperature not higher than  $5^\circ\text{C}$ . Add exactly 10 mL of chloroform, allow to stand for 3 minutes at a temperature not higher than  $5^\circ\text{C}$ , shake vigorously for 2 minutes, and centrifuge. Drain off the water layer, collect 5 mL of the chloroform layer, transfer to a glass-stoppered centrifuge tube containing 3 mL of boric acid-potassium chloride-sodium hydroxide buffer solution, pH 10.0, cooled to a temperature not higher than  $5^\circ\text{C}$ , and 4 mL of water cooled to a temperature not higher than  $5^\circ\text{C}$ , shake for 1 minute, and centrifuge. Drain off the water layer, pipet 2 mL of the chloroform layer, and add chloroform to make exactly 10 mL. Determine the absorbance of this solution at 283 nm as directed under the Ultraviolet-visible Spectrophotometry: it is not more than 0.67.

**Loss on drying** Not more than 0.5% (1 g,  $105^\circ\text{C}$ , 4 hours).

**Assay** Weigh accurately about 0.2 g of Potassium Canrenoate, previously dried, dissolve in 75 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Use a solution of saturated potassium chloride-acetic acid (100) as the internal liquid. Perform a blank determination, and make any necessary correction.

$$\begin{aligned} \text{Each mL of 0.1 mol/L perchloric acid VS} \\ = 39.657 \text{ mg of } \text{C}_{22}\text{H}_{29}\text{KO}_4 \end{aligned}$$

**Containers and storage** Containers—Tight containers.

## Potassium Chloride

塩化カリウム

KCl: 74.55

Potassium Chloride, when dried, contains not less than 99% of KCl.

**Description** Potassium Chloride occurs as colorless or white crystals or crystalline powder. It is odorless, and has a saline taste.

It is freely soluble in water, and practically insoluble in ethanol (95) and in diethyl ether.

A solution of Potassium Chloride (1 in 10) is neutral.

**Identification** A solution of Potassium Chloride (1 in 50) responds to the Qualitative Tests for potassium salt and for chloride.

**Purity (1)** Clarity and color of solution—Dissolve 1.0 g of Potassium Chloride in 5 mL of water: the solution is clear and colorless.

(2) Acid and alkali—Dissolve 5.0 g of Potassium Chloride in 50 mL of freshly boiled and cooled water, and add 3 drops of phenolphthalein TS: no red color develops. Then add 0.50 mL of 0.01 mol/L sodium hydroxide VS: a red color develops.

(3) Bromide—Dissolve 1.0 g of Potassium Chloride in water to make 100 mL. To 5 mL of the solution add 3 drops of dilute hydrochloric acid and 1 mL of chloroform, and add 3 drops of sodium toluenesulfonchloramide TS dropwise while shaking: no yellow to yellow-red color develops in the chloroform layer.

(4) Iodide—Dissolve 0.5 g of Potassium Chloride in 10 mL of water, add 3 drops of iron (III) chloride TS and 1 mL of chloroform, shake, allow to stand for 30 minutes, and shake again: no red-purple to purple color develops in the chloroform layer.

(5) Heavy metals—Proceed with 4.0 g of Potassium Chloride according to Method 1, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 5 ppm).

(6) Calcium and magnesium—Dissolve 0.20 g of Potassium Chloride in 20 mL of water, add 2 mL of ammonia TS, 2 mL of ammonium oxalate TS and 2 mL of disodium hydrogenphosphate TS, and then allow to stand for 5 minutes: no turbidity is produced.

(7) Sodium—Dissolve 1.0 g of Potassium Chloride in 20 mL of water, and perform the Flame Coloration Test (1): no persistent, yellow color develops.

(8) Arsenic—Prepare the test solution with 1.0 g of Potassium Chloride according to Method 1, and perform the test using Apparatus B (not more than 2 ppm).

**Loss on drying** Not more than 0.5% (1 g,  $130^\circ\text{C}$ , 2 hours).

**Assay** Weigh accurately about 0.2 g of Potassium Chloride, previously dried, dissolve in 50 mL of water, and titrate with 0.1 mol/L silver nitrate VS while shaking vigorously (indicator: 1 mL of potassium chromate TS).

Each mL of 0.1 mol/L silver nitrate VS = 7.455 mg of KCl

**Containers and storage** Containers—Tight containers.