

of vincristine is about 19 minutes.

Selection of column: Dissolve 0.010 g each of Vincristine Sulfate and vinblastine sulfate in 100 mL of water. Proceed with 20 μ L of this solution under the above operating conditions, and calculate the resolution. Use a column giving elution of vincristine and vinblastine in this order with the resolution between these peaks being not less than 4.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of vincristine from 20 μ L of the standard solution is between 5 mm and 15 mm.

Time span of measurement: About 3 times as long as the retention time of vincristine after the solvent peak.

Loss on drying Not more than 12.0% (0.05 g, in vacuum, 105°C, 2 hours).

Assay Weigh accurately about 0.01 g of Vincristine Sulfate, dissolve in acetic acid-sodium acetate buffer solution, pH 5.0, to make exactly 50 mL. Pipet 5 mL of this solution, add acetic acid-sodium acetate buffer solution, pH 5.0, to make exactly 50 mL. Determine the absorbance A of this solution at the maximum wavelength at about 296 nm as directed under the Ultraviolet-visible Spectrophotometry.

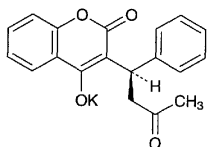
$$\begin{aligned} \text{Amount (mg) of } C_{46}H_{56}N_4O_{10} \cdot H_2SO_4 \\ = \frac{A}{177} \times 5000 \end{aligned}$$

Containers and storage Containers—Hermetic containers.

Storage—Light-resistant, and in a cold place.

Warfarin Potassium

ワルファリンカリウム



and enantiomer

$C_{19}H_{15}KO_4$: 346.42

Monopotassium (*RS*)-2-oxo-3-(3-oxo-1-phenylbutyl)-chromen-4-olate [2610-86-8]

Warfarin Potassium, when dried, contains not less than 98.0% and not more than 102.0% of $C_{19}H_{15}KO_4$.

Description Warfarin Potassium occurs as a white, crystalline powder. It is odorless, and has a slightly bitter taste.

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

It is affected by light.

Identification (1) Dissolve 0.1 g of Warfarin Potassium in 25 mL of water, and add 3 drops of dilute hydrochloric acid. Collect the precipitate produced, wash with four 5-mL portions of water, and dry at 105°C for 1 hour: the residue melts between 157°C and 167°C.

(2) Determine the absorption spectrum of a solution of Warfarin Potassium in 0.02 mol/L potassium hydroxide TS (1 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Refer-

ence Spectrum 1: both spectra exhibit similar intensities of absorption at the same wavelengths. Separately, determine the absorption spectrum of a solution of Warfarin Potassium in 0.02 mol/L hydrochloric acid TS (1 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum 2: both spectra exhibit similar intensities of absorption at the same wavelengths.

(3) The filtrate obtained in (1) responds to the Qualitative Tests for potassium salts.

pH Dissolve 1.0 g of Warfarin Potassium in 100 mL of water: the pH of the solution is between 7.2 and 8.3.

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Warfarin Potassium in 10 mL of water: the solution is clear and colorless.

(2) Alkaline colored substances—Dissolve 1.0 g of Warfarin Potassium in a solution of sodium hydroxide (1 in 20) to make exactly 10 mL, and determine the absorbance at 385 nm within 15 minutes as directed under the Ultraviolet-visible Spectrophotometry, using a solution of sodium hydroxide (1 in 20) as a blank: it does not exceed 0.20.

(3) Heavy metals—Dissolve 2.0 g of Warfarin Potassium in 30 mL of ethanol (95), add 2 mL of dilute acetic acid and ethanol (95) to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 2.0 mL of Standard Lead Solution, 2 mL of dilute acetic acid and ethanol (95) to make 50 mL (not more than 10 ppm).

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

Loss on drying Not more than 10.0% (1 g, 105°C, 3 hours).

Residue on ignition 24.3–25.7% (after drying, 0.4 g, 700°C).

Assay Weigh accurately about 0.1 g of Warfarin Potassium, previously dried, and add 0.02 mol/L potassium hydroxide TS to make exactly 100 mL. Pipet 10 mL of this solution, and add 0.02 mol/L potassium hydroxide TS to make exactly 1000 mL. Determine the absorbance A of this solution at the maximum wavelength at about 308 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\text{Amount (mg) of } C_{19}H_{15}KO_4 = \frac{A}{405} \times 100,000$$

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Warfarin Potassium Tablets

ワルファリンカリウム錠

Warfarin Potassium Tablets contain not less than 95% and not more than 105% of the labeled amount of warfarin potassium ($C_{19}H_{15}KO_4$: 346.42).

Method of preparation Prepare as directed under Tablets, with Warfarin Potassium.

Identification (1) Determine the absorption spectrum of the solution T₂ obtained in the Assay, using 0.02 mol/L potassium hydroxide TS as the blank, as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 306 nm and 310 nm, and a minimum between 258 nm and 262 nm. Separately, determine the absorption spectrum of the solution T₁ obtained in the Assay, using 0.02 mol/L hydrochloric acid TS as the blank, as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 281 nm and 285 nm and between 303 nm and 307 nm, and a minimum between 243 nm and 247 nm.

(2) Weigh a quantity of Warfarin Potassium Tablets, equivalent to 0.01 g of Warfarin Potassium according to the labeled amount, add 10 mL of acetone, shake, and filter. Heat the filtrate on a water bath to evaporate the acetone. To the residue add 10 mL of diethyl ether and 2 mL of dilute hydrochloric acid, and shake: the aqueous layer responds to the Qualitative Tests (1) for potassium salt.

Content uniformity test Powder 1 tablet of Warfarin Potassium Tablets, add 40 mL of water, and shake vigorously for 30 minutes. Add water to make exactly *V* mL of this solution containing about 20 μg of warfarin potassium (C₁₉H₁₅KO₄) per mL. Filter this solution, discard the first 5 mL of the filtrate, and use the subsequent filtrate as the sample solution. Separately, weigh accurately about 0.04 g of warfarin potassium for assay, previously dried at 105°C for 3 hours, and dissolve in water to make exactly 100 mL. Pipet 5 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Pipet 20 mL each of the sample solution and the standard solution, add 0.05 mol/L hydrochloric acid TS to make exactly 25 mL, and use these solutions as the solution T₁ and the solution S₁, respectively. Separately, pipet 20 mL each of the sample solution and the standard solution, add 0.05 mol/L potassium hydroxide TS to make exactly 25 mL, and use these solutions as the solution T₂ and the solution S₂, respectively. Determine the absorbances, A_T and A_S, of the solution T₁ and the solution S₁ at 272 nm as directed under the Ultraviolet-visible Spectrophotometry, using the solution T₂ and the solution S₂ as the blank, respectively.

$$\begin{aligned} &\text{Amount (mg) of warfarin potassium (C}_{19}\text{H}_{15}\text{KO}_4) \\ &= \text{amount (mg) of warfarin potassium for assay} \\ &\quad \times \frac{A_T}{A_S} \times \frac{V}{2000} \end{aligned}$$

Assay Weigh accurately and powder not less than 20 Warfarin Potassium Tablets. Weigh accurately a portion of the powder, equivalent to about 4 mg of warfarin potassium (C₁₉H₁₅KO₄), add 80 mL of water, shake vigorously for 15 minutes, and add water to make exactly 100 mL. Filter this solution, discard the first 10 mL of the filtrate, and use the subsequent filtrate as the sample solution. Separately, weigh accurately about 0.08 g of warfarin potassium for assay, previously dried at 105°C for 3 hours, and dissolve in water to make exactly 100 mL. Pipet 5 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Pipet 10 mL each of the sample solution and the standard solution, add 0.02 mol/L hydrochloric acid TS to make exactly 20 mL, and use these solutions as the solution T₁ and the solution S₁, respectively. Separately, pipet 10 mL each of the sample solution and the standard solution, add 0.02 mol/L potassium hydroxide TS to make exactly 20 mL, and use these solutions as the solution T₂ and

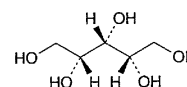
the solution S₂, respectively. Determine the absorbances, A_T and A_S, of the solution T₁ and the solution S₁ at 272 nm as directed under the Ultraviolet-visible Spectrophotometry, using the solution T₂ and the solution S₂ as the blank, respectively.

$$\begin{aligned} &\text{Amount (mg) of warfarin potassium (C}_{19}\text{H}_{15}\text{KO}_4) \\ &= \text{amount (mg) of warfarin potassium for assay} \\ &\quad \times \frac{A_T}{A_S} \times \frac{1}{20} \end{aligned}$$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Xylitol

キシリトール



C₅H₁₂O₅: 152.15
meso-Xylitol [87-99-0]

Xylitol, when dried, contains not less than 98.0% of C₅H₁₂O₅.

Description Xylitol occurs as white crystals or powder. It is odorless and has a sweet taste.

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

It is hygroscopic.

Identification (1) To 1 mL of a solution of Xylitol (1 in 2) add 2 mL of iron (II) sulfate TS and 1 mL of a solution of sodium hydroxide (1 in 5): blue-green color is produced without turbidity.

(2) Determine the infrared absorption spectrum of Xylitol, 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.

pH Dissolve 5.0 g of Xylitol in 10 mL of freshly boiled and cooled water: the pH of this solution is between 5.0 and 7.0.

Melting point 93.0 – 95.0°C

Purity (1) Clarity and color of solution—Dissolve 5 g of Xylitol in 10 mL of water: the solution is clear and colorless.

(2) Chloride—Perform the test with 2.0 g of Xylitol. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.005%).

(3) Sulfate—Perform the test with 4.0 g of Xylitol. Prepare the control solution with 0.50 mL of 0.005 mol/L sulfuric acid VS (not more than 0.006%).

(4) Heavy metals—Proceed with 4.0 g of Xylitol 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).

(5) Nickel—Dissolve 0.5 g of Xylitol in 5 mL of water, add 3 drops of dimethylglyoxime TS and 3 drops of ammo-