

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

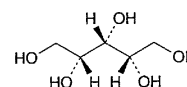
the solution S₂, respectively. Determine the absorbances, A_T and A_S, of the solution T₁ and the solution S₁ at 272 nm as directed under the Ultraviolet-visible Spectrophotometry, using the solution T₂ and the solution S₂ as the blank, respectively.

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

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

Xylitol

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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-

nia TS, and allow to stand for 5 minutes: no red color is produced.

(6) **Arsenic**—Prepare the test solution with 1.5 g of Xylitol according to Method 1, and perform the test using Apparatus B (not more than 1.3 ppm).

(7) **Sugars**—Dissolve 5.0 g of Xylitol in 15 mL of water, add 4.0 mL of dilute hydrochloric acid, and heat in a water bath for 3 hours under a reflux condenser. After cooling, neutralize with sodium hydroxide TS (indicator: 2 drops of methyl orange TS). Then add water to make 50 mL, transfer 10 mL of this solution to a flask, add 10 mL of water and 40 mL of Fehling's TS, boil gently for 3 minutes, and allow to stand to precipitate copper (I) oxide. Remove the supernatant liquid through a glass filter (G4), and wash the precipitate with warm water until the last washing does not show alkalinity. Filter these washings through the glass filter mentioned above. Dissolve the precipitate in the flask in 20 mL of iron (III) sulfate TS, filter the solution through the glass filter mentioned above, wash with water, combine the washings with the filtrate, heat at 80°C, and titrate with 0.02 mol/L potassium permanganate VS: not more than 1.0 mL of 0.02 mol/L potassium permanganate VS is consumed.

Loss on drying Not more than 1.0% (1 g, in vacuum, phosphorus (V) oxide, 24 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.2 g of Xylitol, previously dried, dissolve in water to make exactly 100 mL. Pipet 10 mL of this solution into an iodine flask, add 50 mL of potassium periodate TS exactly, and heat in a water bath for 15 minutes. After cooling, add 2.5 g of potassium iodide, stopper, shake well, allow to stand for 5 minutes in a dark place, and titrate with 0.1 mol/L sodium thiosulfate VS (indicator: 3 mL of starch TS). Perform a blank determination.

Each mL of 0.1 mol/L sodium thiosulfate VS
= 1.9018 mg of C₅H₁₂O₅

Containers and storage Containers—Tight containers.

Xylitol Injection

キシリトール注射液

Xylitol Injection is an aqueous solution for injection. It contains not less than 95% and not more than 105% of the labeled amount of xylitol (C₅H₁₂O₅; 152.15).

Method of preparation Prepare as directed under Injections, with Xylitol. No preservative may be added.

Description Xylitol Injection is a clear, colorless liquid. It has a sweet taste.

Identification Measure a volume of Xylitol Injection, equivalent to 0.1 g of Xylitol according to the labeled amount, add water to make 10 mL, and use this solution as the sample solution. Separately, dissolve 0.1 g of xylitol in 10 mL of water, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 2 μL each of the sample solution and the standard solution on a plate of silica gel

for thin-layer chromatography. Develop the plate with a mixture of ethanol (95), ammonia solution (28) and water (25:4:3) to a distance of about 10 cm, and air-dry the plate. Spray evenly silver nitrate-ammonia TS, and dry at 105°C for 15 minutes: the spots from the sample solution and the standard solution show a blackish brown color and the same R_f value.

pH 4.5 – 7.5

Bacterial endotoxins Less than 0.50 EU/mL.

Assay Measure exactly a volume of Xylitol Injection, equivalent to about 5.0 g of xylitol (C₅H₁₂O₅) according to the labeled amount, and add water to make exactly 250 mL. Measure exactly 10 mL of this solution, and add water to make exactly 100 mL. Then, pipet 10 mL of this solution into an iodine flask, and proceed as directed in the Assay under Xylitol.

Each mL of 0.1 mol/L sodium thiosulfate VS
= 1.9018 mg of C₅H₁₂O₅

Containers and storage Containers—Hermetic containers.

Zinc Oxide

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ZnO: 81.39

Zinc Oxide, when ignited, contains not less than 99.0% of ZnO.

Description Zinc Oxide occurs as a white, amorphous powder. It is odorless and tasteless.

It is practically insoluble in water, in ethanol (95), in acetic acid (100) and in diethyl ether.

It dissolves in dilute hydrochloric acid and in sodium hydroxide TS.

It gradually absorbs carbon dioxide from air.

Identification (1) Heat Zinc Oxide strongly: a yellow color develops on strong heating, and disappears on cooling.

(2) A solution of Zinc Oxide in dilute hydrochloric acid (1 in 10) responds to the Qualitative Tests for zinc salt.

Purity (1) Carbonate, and clarity and color of solution—Mix 2.0 g of Zinc Oxide with 10 mL of water, add 30 mL of dilute sulfuric acid, and heat on a water bath with stirring: no effervescence occurs, and the solution obtained is clear and colorless.

(2) **Alkali**—To 1.0 g of Zinc Oxide add 10 mL of water, and boil for 2 minutes. Cool, filter through a glass filter (G3), and to the filtrate add 2 drops of phenolphthalein TS and 0.20 mL of 0.1 mol/L hydrochloric acid VS: no color develops.

(3) **Sulfate**—Shake 0.5 g of Zinc Oxide with 40 mL of water, and filter. Take 20 mL of the filtrate, add 1 mL of dilute hydrochloric acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution with 0.50 mL of 0.005 mol/L sulfuric acid VS (not more than 0.096%).