Triclofos Sodium Syrup

Monosodium Trichloroethyl Phosphate Syrup

トリクロホススナトリウムシロップ

Triclofos Sodium Syrup contains not less than 90% and not more than 110% of the labeled amount of triclofos sodium (C₂₃H₂₂Cl₃NaO₂P: 251.37).

Method of preparation Prepare as directed under Syrups, with Triclofos Sodium.

Identification (1) Weigh a portion of Triclofos Sodium Syrup, equivalent to 0.25 g of Triclofos Sodium according to the labeled amount, add 40 mL of water, shake well, add 5 mL of diluted sulfuric acid (3 in 50), and extract with 25 mL of 3-methyl-1-butanol. Take 5 mL of the extract, evaporate on a water bath to dryness, and add 1 mL of diluted sulfuric acid (1 in 2) and 1 mL of a solution of potassium permanganate (1 in 20) to the residue. Heat in a water bath for 5 minutes, add 7 mL of water, and then add a solution of oxalic acid dihydrate (1 in 20) until the color of the solution disappears. To 1 mL of this solution add 1 mL of pyridine and 1 mL of a solution of sodium hydroxide (1 in 5), and heat in a water bath, while shaking, for 1 minute: a light red color develops in the pyridine layer.

(2) Take 10 mL of the extract obtained in (1), evaporate on a water bath to dryness, add 1 g of anhydrous sodium carbonate to the residue, and heat for 10 minutes. After cooling, dissolve the residue in 40 mL of water, filter if necessary, and render the filtrate acidic with dilute nitric acid: the solution responds to the Qualitative Tests (2) for chloride. The remainder of the filtrate responds to the Qualitative Tests (1) for chloride and to the Qualitative Tests for phosphate.

pH 6.0–6.5

Assay Weigh accurately a portion of Triclofos Sodium Syrup, equivalent to 0.13 g of Triclofos Sodium according to the labeled amount, add 15 mL of water, 1 mL of sodium hydroxide TS and 15 mL of diethyl ether, shake for 1 minute, and separate the water layer. Wash the diethyl ether layer with 1 mL of water, and combine the washing with above water layer. To this solution add 2.5 mL of diluted sulfuric acid (3 in 50), and extract with four 10-mL portions of 3-methyl-1-butanol. Combine the 3-methyl-1-butanol extracts, and add 3-methyl-1-butanol to make exactly 50 mL. Measure exactly 10 mL each of this solution, and dilute with potassium hydroxide-ethanol TS. Place in a glass ampule, fire-seal, mix, and heat at 120°C for 2 hours in an autoclave. After cooling, transfer the contents to a flask, add 20 mL of diluted nitric acid (63 in 500) and exactly 25 mL of 0.02 mol/L silver nitrate VS, shake well, and titrate the excess silver nitrate with 0.02 mol/L ammonium thiocyanate VS (indicator: 2 to 3 drops of ammonium iron (III) sulfate TS). Perform a blank determination.

Each mL of 0.02 mol/L silver nitrate VS = 1.676 mg of C₂₃H₂₂Cl₃NaO₂P

Containers and storage Containers—Tight containers. Storage—in a cold place.

Trihexyrophyridyld Hydrochloride

塩酸トリヘキシフェニルジル

C₂₉H₂₅NO.HCl: 337.93
(RS)-1-Cyclohexyl-1-phenyl-3-(piperidin-1-yl)propan-1-ol monohydrochloride [52-49-3]

Trihexyrophyridyld Hydrochloride, when dried, contains not less than 98.5% of C₂₉H₂₅NO.HCl.

Description Trihexyrophyridyld Hydrochloride occurs as a white, crystalline powder. It is odorless, and has a bitter taste.

It is soluble in ethanol (95), sparingly soluble in acetic acid (100), slightly soluble in water, very slightly soluble in acetic anhydride, and practically insoluble in diethyl ether.

Melting point: about 250°C (with decomposition).

Identification (1) Dissolve 1 g of Trihexyrophyridyld Hydrochloride in 100 mL of water by warming, and cool. Use this solution as the sample solution. To 5 mL of the sample solution add 1 mL of a solution of 2,4,6-trinitrophenol in chloroform (1 in 50), and shake vigorously: a yellow precipitate is formed.

(2) To 20 mL of the sample solution obtained in (1) add 2 mL of sodium hydroxide TS: a white precipitate is formed. Collect the precipitate, wash with a small amount of water, recrystallize from methanol, and dry in a desiccator (in vacuum, silica gel) for 2 hours: the crystals so obtained melt between 113°C and 117°C.

(3) The sample solution obtained in (1) responds to the Qualitative Tests (2) for chloride.

pH Dissolve 1.0 g of Trihexyrophyridyld Hydrochloride in 100 mL of water by warming, and cool: the pH of this solution is between 5.0 and 6.0.

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Trihexyrophyridyld Hydrochloride in 100 mL of water by warming: the solution is clear and colorless.

(2) Heavy metals—Dissolve 1.5 g of Trihexyrophyridyld Hydrochloride in 60 mL of water by warming on a water bath at 80°C, cool, and filter. To 40 mL of the filtrate add 2 mL of dilute acetic acid and water to make 50 mL, and 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 water to make 50 mL (not more than 20 ppm).

(3) Piperidylpropionophenone—Dissolve 0.10 g of Trihexyrophyridyld Hydrochloride in 40 mL of water and 1 mL of 1 mol/L hydrochloric acid VS by warming, cool, and add water to make 100 mL. Determine the absorbance of this solution at 247 nm as directed under the Ultraviolet-visible Spectrophotometry: the absorbance is not more than 0.50.

Loss on drying Not more than 0.5% (1 g, 105°C, 3 hours).