

[18559-59-6, anhydride]

Trimetoquinol Hydrochloride contains not less than 98.0% of $C_{19}H_{23}NO_5 \cdot HCl$ (mol. wt.: 381.86), calculated on the dried basis.

Description Trimetoquinol Hydrochloride occurs as white crystals or crystalline powder. It is odorless.

It is sparingly soluble in water and in ethanol (99.5), slightly soluble in acetonitrile and in acetic acid (100), and practically insoluble in acetone, in dehydrated diethyl ether and in chloroform.

Melting point: about 151°C (with decomposition, after drying).

Identification (1) Dissolve 0.01 g of Trimetoquinol Hydrochloride in 5 mL of water, add 1 mL of dilute iron (III) chloride TS: a deep green color develops. Add 4 drops of diluted ammonia TS (1 in 10): a red-purple color is produced.

(2) Dissolve 0.03 g of Trimetoquinol Hydrochloride in 5 mL of water, and add 3 drops of Reinecke salt TS: a light red precipitate is formed.

(3) Dissolve 0.01 g of Trimetoquinol Hydrochloride in 200 mL of 0.01 mol/L hydrochloric acid TS. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(4) Determine the infrared absorption spectrum of Trimetoquinol Hydrochloride as directed in the potassium chloride 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.

(5) A solution of Trimetoquinol Hydrochloride (1 in 50) responds to the Qualitative Tests (1) for chloride.

Optical rotation $[\alpha]_D^{20}$: -17 – -20° (0.25 g, calculated on the dried basis, water, after warming and cooling, 25 mL, 100 mm).

pH Dissolve 1.0 g of Trimetoquinol Hydrochloride in 100 mL of water by warming, and cool: the pH of this solution is between 4.5 and 5.5.

Purity (1) Clarity and color of solution—Dissolve 0.10 g of Trimetoquinol Hydrochloride in 10 mL of water by warming: the solution is clear and colorless.

(2) Sulfate—Perform the test with 0.5 g of Trimetoquinol Hydrochloride. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.038%).

(3) Heavy metals—Proceed with 1.0 g of Trimetoquinol Hydrochloride according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

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

(5) Related substances—Dissolve 0.050 g of Trimetoquinol Hydrochloride in 50 mL of the mobile phase, and use this solution as the sample solution. Pipet 1 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μ L each of the sample solution and the standard solu-

tion, as directed under the Liquid Chromatography according to the following conditions. Determine each peak area of both solutions by the automatic integration method: the total area of the peaks other than that of trimetoquinol from the sample solution is not larger than the peak area of trimetoquinol from the standard solution.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 283 nm).

Column: A stainless steel column about 4 mm in inside diameter and 15 to 30 cm in length, packed with octadecylsilylated silica gel for liquid chromatography (5 to 10 μ m in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: Dissolve 2 g of potassium dihydrogenphosphate and 2 g of sodium 1-pentane sulfonate in 1000 mL of water. Adjust with phosphoric acid to a pH between 2.8 and 3.2, and filter through a membrane filter (0.4 μ m). Add 200 mL of acetonitrile to 800 mL of the filtrate.

Flow rate: Adjust the flow rate so that the retention time of trimetoquinol is about 7 minutes.

Selection of column: Dissolve 5 mg of Trimetoquinol Hydrochloride and 1 mg of procaine hydrochloride in 50 mL of the mobile phase. Proceed with 20 μ L of this solution under the above operating conditions, and calculate the resolution. Use a column giving elution of procaine and trimetoquinol 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 trimetoquinol from 20 μ L of the standard solution is between 2 mm and 6 mm.

Time span of measurement: About twice as long as the retention time of trimetoquinol after the solvent peak.

Loss on drying 3.5 – 5.5% (1 g, in vacuum, 105°C, 4 hours).

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

Assay Weigh accurately about 0.5 g of Trimetoquinol Hydrochloride, dissolve in 2 mL of 0.1 mol/L hydrochloric acid VS and 70 mL of ethanol (99.5) with thorough shaking, and titrate with 0.1 mol/L potassium hydroxide-ethanol VS (potentiometric titration). Calculate the consumed volume of 0.1 mol/L potassium hydroxide-ethanol VS between the first inflection point and of the second inflection point.

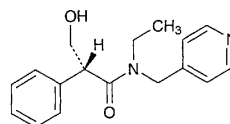
Each mL of 0.1 mol/L potassium hydroxide-ethanol VS = 38.186 mg of $C_{19}H_{23}NO_5 \cdot HCl$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Tropicamide

トロピカミド



and enantiomer

$C_{17}H_{20}N_2O_2$: 284.35

(*RS*)-*N*-Ethyl-3-hydroxy-2-phenyl-*N*-(pyridin-4-ylmethyl)-propanamide [1508-75-4]

Tropicamide, when dried, contains not less than 98.5% of $C_{17}H_{20}N_2O_2$.

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

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

It dissolves in dilute hydrochloric acid.

The pH of a solution of Tropicamide (1 in 500) is between 6.5 and 8.0.

Identification (1) To 5 mg of Tropicamide add 0.5 mL of a solution of ammonium vanadate (V) in sulfuric acid, (1 in 200), and heat: a blue-purple color develops.

(2) Dissolve 5 mg of Tropicamide in 1 mL of ethanol (95) and 1 mL of water, add 0.1 g of 1-chloro-2,4-dinitrobenzene, and heat on a water bath for 5 minutes. Cool, and add 2 to 3 drops of a solution of sodium hydroxide (1 in 10) and 3 mL of ethanol (95): a red-purple color develops.

Absorbance $E_{1\text{cm}}^{1\%}$ (255 nm): 166 – 180 (after drying, 5 mg, 2 mol/L hydrochloric acid TS, 200 mL).

Melting point 96 – 99°C

Purity (1) Chloride—Dissolve 1.0 g of Tropicamide in 30 mL of ethanol (95), add 6 mL of dilute nitric acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution with 0.45 mL of 0.01 mol/L hydrochloric acid VS, 30 mL of ethanol (95), 6 mL of dilute nitric acid, and add water to make 50 mL (not more than 0.016%).

(2) Heavy metals—Dissolve 1.0 g of Tropicamide in 30 mL of ethanol (95), 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, 30 mL of ethanol (95), 2 mL of dilute acetic acid and water to make 50 mL (not more than 20 ppm).

(3) *N*-Ethyl- γ -picolylamine—Dissolve 0.10 g of Tropicamide in 5 mL of water by heating, add 1 mL of a solution of acetaldehyde (1 in 20), and shake well. Add 1 to 2 drops of sodium pentacyanonitrosylferrate (III) TS and 1 to 2 drops of sodium hydrogen carbonate TS, and shake: no blue color develops.

(4) Tropic acid—To 0.010 g of Tropicamide add 5 mg of sodium borate and 7 drops of 4-dimethylaminobenzaldehyde TS, and heat in a water bath for 3 minutes. Cool in ice water, and add 5 mL of acetic anhydride: no red-purple color develops.

Loss on drying Not more than 0.30% (1 g, in vacuum, silica gel, 24 hours).

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

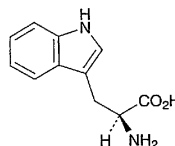
Assay Weigh accurately about 0.5 g of Tropicamide, previously dried, dissolve in 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (indicator: 3 drops of crystal violet TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 28.436 mg of $C_{17}H_{20}N_2O_2$

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

L-Tryptophan

L-トリプトファン



$C_{11}H_{12}N_2O_2$: 204.23

(*2S*)-2-Amino-3-(indol-3-yl)propanoic acid [73-22-3]

L-Tryptophan, when dried, contains not less than 98.5% of $C_{11}H_{12}N_2O_2$.

Description L-Tryptophan occurs as white to yellowish white crystals or crystalline powder. It is odorless, and has a slightly bitter taste.

It is freely soluble in formic acid, slightly soluble in water, and very slightly soluble in ethanol (95).

It dissolves in dilute hydrochloric acid.

Identification Determine the infrared absorption spectrum of L-Tryptophan, 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.

Optical rotation $[\alpha]_D^{20}$: –30.0 – –33.0° Weigh accurately about 0.25 g of L-Tryptophan, previously dried, and dissolve in 20 mL of water by warming. After cooling, add water to make exactly 25 mL, and determine the optical rotation of the solution in a 100-mm cell.

pH Dissolve 1.0 g in 100 mL of water by warming, and cool: the pH of this solution is between 5.4 and 6.4.

Purity (1) Clarity of solution—Dissolve 0.20 g of L-Tryptophan in 10 mL of 2 mol/L hydrochloric acid TS: the solution is clear.

(2) Chloride—Dissolve 0.5 g of L-Tryptophan in 6 mL of dilute nitric acid, and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.021%).

(3) Sulfate—Dissolve 0.6 g of L-Tryptophan in 40 mL of water and 1 mL of dilute hydrochloric acid, and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid VS (not more than 0.028%).

(4) Ammonium—Perform the test with 0.25 g of L-Tryptophan. Prepare the control solution with 5.0 mL of Standard Ammonium Solution (not more than 0.02%).

(5) Heavy metals—Proceed with 1.0 g of L-Tryptophan according to Method 4, and perform the test. Prepare the