

$C_{17}H_{20}N_2O_2$ : 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- $\gamma$ -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).

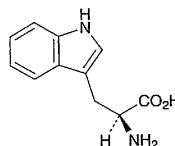
**Assay** Weigh accurately about 0.5 g of Tropicamide, previously dried, dissolve in 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (indicator: 3 drops of crystal violet TS). Perform a blank determination, and make any necessary correction.

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

control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(6) Arsenic—Dissolve 1.0 g of L-Tryptophan in 3 mL of 1 mol/L hydrochloric acid TS and 2 mL of water by heating, and perform the test with this solution as the test solution using Apparatus B (not more than 2 ppm).

(7) Other amino acids—Dissolve 0.30 g of L-Tryptophan in 1 mL of 1 mol/L hydrochloric acid TS, add water to make 50 mL, and use this solution as the sample solution. Pipet 1 mL of the sample solution, and add water to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 20 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5  $\mu$ L 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 1-butanol, water and acetic acid (100) (3:1:1) to a distance of about 10 cm, and dry the plate at 80°C for 30 minutes. Spray evenly a solution of ninhydrin in acetone (1 in 50) on the plate, and heat at 80°C for 5 minutes: the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

**Loss on drying** Not more than 0.30% (1 g, 105°C, 3 hours).

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

**Assay** Weigh accurately about 0.2 g of L-Tryptophan, previously dried, dissolve in 3 mL of formic acid, add 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

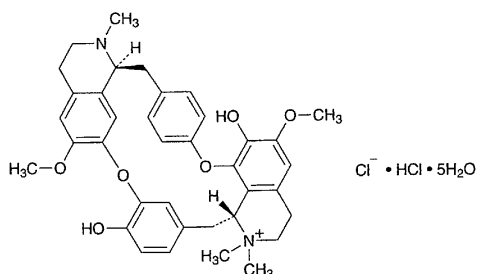
Each mL of 0.1 mol/L perchloric acid VS  
= 20.423 mg of  $C_{37}H_{41}ClN_2O_6 \cdot 5H_2O$

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

## Tubocurarine Chloride

### Tubocurarine Hydrochloride

塩化ツボクラリン



$C_{37}H_{41}ClN_2O_6 \cdot HCl \cdot 5H_2O$ : 771.72  
7',12'-Dihydroxy-6,6'-dimethoxy-2,2',2'-trimethyltubocuraranium chloride monohydrochloride pentahydrate [41354-45-4]

Tubocurarine Chloride contains not less than

98.0% of  $C_{37}H_{41}ClN_2O_6 \cdot HCl$  (mol. wt.: 681.65), calculated on the dried basis.

**Description** Tubocurarine Chloride occurs as white crystals or crystalline powder. It is odorless.

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

The pH of a solution of Tubocurarine chloride (1 in 100) is between 4.0 and 6.0.

Melting point: about 270°C (with decomposition).

**Identification (1)** To 20 mL of a solution of Tubocurarine Chloride (1 in 2000) add 0.2 mL of sulfuric acid and 2 mL of a solution of potassium iodate (1 in 100), shake, and heat on a water bath for 30 minutes: a yellow color is produced.

(2) To 1 mL of a solution of Tubocurarine Chloride (1 in 100) add 1 mL of a solution of Reinecke salt (1 in 25): a red precipitate is formed.

(3) Determine the absorption spectrum of a solution of Tubocurarine Chloride (3 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of a solution of Tubocurarine Chloride Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.

(4) A solution of Tubocurarine Chloride (1 in 50) responds to the Qualitative Tests (2) for chloride.

**Optical rotation**  $[\alpha]_D^{20}$ : +210 – +220° (0.1 g, calculated on the dried basis, water, 10 mL, after allowing to stand for 3 hours, 100 mm).

**Purity (1)** Clarity and color of solution—Dissolve 0.10 g of Tubocurarine Chloride in 10 mL of ethanol (95): the solution is clear and colorless.

(2) Chloroform-soluble substances—Weigh accurately about 0.2 g of Tubocurarine Chloride, calculated on the dried basis, add 200 mL of water and 1 mL of a saturated solution of sodium hydrogen carbonate, and extract with three 20-mL portions of chloroform. Combine the chloroform extracts, wash with 10 mL of water, filter the chloroform solution through absorbent cotton into a tared beaker, wash the absorbent cotton with two 5-mL portions of chloroform, and combine the filtrate and the washings. Evaporate the chloroform on a water bath, and dry the residue at 105°C for 1 hour: the mass of the residue is not more than 2.0% of the mass of Tubocurarine Chloride taken. Add 10 mL of water to the residue: the residue does not dissolve. Add 1 mL of hydrochloric acid, and stir: the residue dissolves.

**Loss on drying** 9 – 12% (0.5 g, in vacuum, phosphorus (V) oxide, 105°C, 4 hours).

**Residue on ignition** Not more than 0.25% (0.5 g).

**Assay** Weigh accurately about 0.5 g of Tubocurarine Chloride, add 20 mL of acetic acid (100), and dissolve by warming on a water bath. After cooling, add 60 mL of acetic anhydride, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.