Each mL of 0.1 mol/L perchloric acid VS
= 34.083 mg of $C_{37}H_{41}ClN_2O_6.HCl$

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

# Tubocurarine Chloride Injection

## Tubocurarine Hydrochloride Injection

塩化ツボクラリン注射液

Tubocurarine Chloride Injection is an aqueous solution for injection. It contains not less than 93% and not more than 107% of the labeled amount of tubocurarine chloride ($C_{37}H_{41}ClN_2O_6.HCl.5H_2O$: 771.72).

**Method of preparation** Prepare as directed under Injections, with Tubocurarine Chloride.

**Description** Tubocurarine Chloride Injection is a clear, colorless liquid.

**Identification** **(1)** To a volume of Tubocurarine Chloride Injection, equivalent to 0.01 g of Tubocurarine Chloride according to the labeled amount, add water to make 20 mL, and proceed as directed in the Identification (1) under Tubocurarine Chloride.

**(2)** Proceed with a volume of Tubocurarine Chloride Injection, equivalent to 3 mg of Tubocurarine Chloride according to the labeled amount, as directed in the Identification (2) under Tubocurarine Chloride.

**(3)** To a volume of Tubocurarine Chloride Injection, equivalent to 3 mg of Tubocurarine Chloride according to the labeled amount, add water to make 100 mL, and determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 279 nm and 281 nm, and a minimum between 253 nm and 257 nm.

**Optical rotation** $\alpha_D^{20}$: +0.35 - +0.42° (200 mm), calculated with reference to the value of solution containing 1 mg of Tubocurarine Chloride per ml, according to the labeled amount of Tubocurarine Chloride Injection.

**pH** 3.0 - 6.0

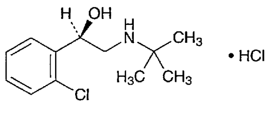
**Assay** Measure exactly a volume of Tubocurarine Chloride Injection, equivalent to about 0.015 g of tubocurarine chloride ($C_{37}H_{41}ClN_2O_6.HCl.5H_2O$), add water to make exactly 500 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.015 g of Tubocurarine Chloride Reference Standard (previously dry in the same manner as directed under Tubocurarine Chloride, and weigh to determine the loss on drying), dissolve in water to make exactly 500 mL, and use this solution as the standard solution. Determine the absorbances, $A_T$ and $A_S$, of the sample solution and the standard solution at 280 nm as directed under the Ultraviolet-visible Spectrophotometry, respectively.

Amount (mg) of tubocurarine chloride
($C_{37}H_{41}ClN_2O_6.HCl.5H_2O$)
= amount (mg) of Tubocurarine Chloride Reference Standard, calculated on the dried basis
$\times \frac{A_T}{A_S} \times 1.1321$

**Containers and storage** Containers—Hermetic containers.
Storage—Light-resistant, and under Nitrogen atmosphere.

# Tulobuterol Hydrochloride

塩酸ツロブテロール



and enantiomer

$C_{12}H_{18}ClNO.HCl$: 264.19
(*RS*)-2-*tert*-Butylamino-1-(2-chlorophenyl)ethanol monohydrochloride [*56776-01-3*]

Tulobuterol Hydrochloride, when dried, contains not less than 98.5% of $C_{12}H_{18}ClNO.HCl$.

**Description** Tulobuterol Hydrochloride occurs as white crystals or crystalline powder.

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

A solution of Tulobuterol Hydrochloride (1 in 20) shows no optical rotation.

Melting point: about 163°C

**Identification** **(1)** Determine the absorption spectrum of a solution of Tulobuterol Hydrochloride (1 in 2500) 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.

**(2)** Determine the infrared absorption spectrum of Tulobuterol Hydrochloride, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spctra exhibit similar intensities of absorption at the same wave numbers.

**(3)** A solution of Tulobuterol Hydrochloride (1 in 20) responds to the Qualitative Tests for chloride.

**Purity** **(1)** Clarity and color of solution—Dissolve 1.0 g of Tulobuterol Hydrochloride in 10 mL of water: the solution is clear and colorless.

**(2)** Heavy metals—Proceed with 2.0 g of Tulobuterol Hydrochloride according to Method 1, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

**(3)** Related substances—Dissolve 0.30 g of Tulobuterol Hydrochloride in 5 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Use a plate previously developed with the upper-layer of a mixture of ethyl acetate and ammonia solution (28) (200:9) to the top of the plate and air-dried. Spot 5 $\mu$L each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with the upper layer of a mixture of ethyl acetate and ammonia solution (28) (200:9) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot and the spot of starting point from the sample solution are not more intense than the spot from the standard solution.

**Loss on drying** Not more than 0.5% (0.5 g, in vacuum, 60°C, 4 hours).

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

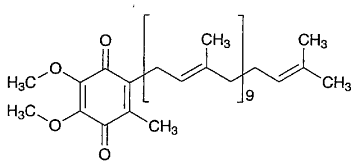
**Assay** Weigh accurately about 0.5 g of Tulobuterol Hydrochloride, previously dried, dissolve in 80 mL of a mixture of acetic anhydride and acetic acid (100) (7:3), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 26.419 mg of $C_{12}H_{18}ClNO.HCl$

**Containers and storage** Containers—Tight containers.

# Ubidecarenone

ユビデカレノン



$C_{59}H_{90}O_4$: 863.34
(2*E*,6*E*,10*E*,14*E*,18*E*,22*E*,26*E*,30*E*,34*E*,38*E*)-2-(3,7,11,15,19,23,27,31,35,39-Decamethyltetraconta-2,6,10,14,18,22,26,30,34,38-decaen-1-yl)-5,6-dimethoxy-3-methyl-1,4-benzoquinone [*303-98-0*]

Ubidecarenone contains not less than 98.0% of $C_{59}H_{90}O_4$, calculated on the anhydrous basis.

**Description** Ubidecarenone occurs as a yellow to orange crystalline powder.

It is odorless and has no taste.

It is soluble in diethyl ether, very slightly soluble in ethanol (99.5), and practically insoluble in water.

It is gradually decomposed and colored by light.

Melting point: about 48°C

**Identification (1)** Dissolve 0.05 g of Ubidecarenone in 1 mL of diethyl ether, and add 10 mL of ethanol (99.5). To 2 mL of this solution add 3 mL of ethanol (99.5) and 2 mL of dimethyl malonate, then add dropwise 1 mL of a solution of potassium hydroxide (1 in 5), and mix: a blue color appears.

**(2)** Determine the infrared absorption spectrum of Ubidecarenone as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of Ubidecarenone Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers.

**Purity (1)** Heavy metals—Proceed with 1.0 g of Ubidecarenone 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).

**(2)** Related substances—Dissolve 0.05 g of Ubidecarenone in 50 mL of ethanol (99.5) by warming at about 50°C for 2 minutes, and after cooling use this solution as the sample solution. To exactly 1 mL of the sample solution add ethanol (99.5) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 5 $\mu$L each of the sample solution and the standard solution 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 the peak of ubidecarenone from the sample solution is not larger than the peak area of ubidecarenone from the standard solution.

*Operating conditions—*

Detector, column, column temperature, mobile phase, flow rate, and selection of column: Proceed as directed in the operating conditions in the Assay.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of ubidecarenone obtained from 5 $\mu$L of the standard solution is between 20 mm and 40 mm.

Time span of measurement: About 2 times of the retention time of ubidecarenone after the solvent peak.

**Water** Not more than 0.20% (1 g, direct titration).

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

**Assay** Weigh accurately about 0.05 g each of Ubidecarenone and Ubidecarenone Reference Standard (separately determined the water content) dissolve each in 40 mL of ethanol (99.5) by warming at about 50°C for 2 minutes, and after cooling add ethanol (99.5) to make exactly 50 mL each, and use these solutions as the sample solution and the standard solution. Perform the test with exact 5 $\mu$L each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine peak areas, $A_T$ and $A_S$, of ubidecarenone of these solution.

$$\text{Amount (mg) of } C_{59}H_{90}O_4 = \text{amount (mg) of Ubidecarenone Reference Standard, calculated on the anhydrous basis} \times \frac{A_T}{A_S}$$

*Operating conditions—*

Detector: An ultraviolet absorption photometer (wavelength: 275 nm).

Column: A stainless steel column about 5 mm in inside diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 $\mu$m in particle diameter).

Column temperature: A constant temperature of about 35°C.

Mobile phase: A mixture of methanol and ethanol (99.5) (13:7).

Flow rate: Adjust the flow rate so that the retention time of ubidecarenone is about 10 minutes.