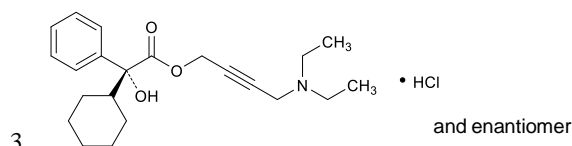# Oxybutynin Hydrochloride

オキシブチニン塩酸塩

and enantiomer

$C_{22}H_{31}NO_3 \cdot HCl$: 393.95

4-(Diethylamino)but-2-yn-1-yl (2*RS*)-2-cyclohexyl-2-hydroxy-
2-phenylacetate monohydrochloride

[*1508-65-2*]

Oxybutynin Hydrochloride, when dried, contains not less than 98.0% and not more than 101.0% of oxybutynin hydrochloride ($C_{22}H_{31}NO_3 \cdot HCl$).

Description Oxybutynin Hydrochloride occurs as a white crystalline powder.

It is freely soluble in water and in ethanol (99.5).

A solution of Oxybutynin Hydrochloride (1 in 50) shows no optical rotation.

Identification **(1)** Determine the absorption spectrum of a solution of Oxybutynin Hydrochloride (3 in 100,000) as directed under Ultraviolet-visible Spectrophotometry *<2.24>*, 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 Oxybutynin Hydrochloride, previously dried, as directed in the potassium chloride disk method under Infrared Spectrophotometry *<2.25>*, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(3) A solution of Oxybutynin Hydrochloride (1 in 50) responds to Qualitative Tests *<1.09>* for chloride.

Melting point *<2.60>* 124 – 129°C

Purity **(1)** Heavy metals *<1.07>*－Proceed with 2.0 g of Oxybutynin Hydrochloride according to Method 1, and perform the test. Prepare the control solution with 3.0 mL of Standard Lead Solution (not more than 15 ppm).

(2) Related substances－Dissolve 50 mg of Oxybutynin Hydrochloride in 10 mL of the mobile phase, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add the mobile phase to make exactly 200 mL, and use this solution as the standard solution. Perform the test with exactly 10 μL each of the sample solution and standard solution as directed under Liquid Chromatography *<2.01>* according to the following conditions, and determine each peak area by the automatic integration method: the peak area of the related substance A, having the relative retention time of about 1.6 to oxybutynin, obtained from the sample solution is not larger than 3 times the peak area of oxybutynin from the standard solution, and the area of the peak other than oxybutynin and the peak mentioned above from the sample solution is not larger than 1/5 times the peak area of oxybutynin from the standard solution. Furthermore, the total area of the peaks other than oxybutynin and the related substance A from the sample solution is not larger than the peak area of oxybutynin from the standard solution. For the peak area of the related substance A, multiply the relative response factor, 2.3.

Operating conditions －

Detector: An ultraviolet absorption photometer (wavelength: 210 nm).

Column: A stainless steel column 3.9 mm in inside diameter and 15 cm in length, packed with octylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 25°C.

Mobile phase: Dissolve 3.4 g of potassium dihydrogen phosphate and 4.36 g of dipotassium hydrogen phosphate in water to make 1000 mL. To 490 mL of this solution add 510 mL of acetonitrile for liquid chromatography.

Flow rate: Adjust so that the retention time of oxybutynin is about 15 minutes.

Time span of measurement: About 2 times as long as the retention time of oxybutynin.

System suitability －

Test for required detectability: Pipet 2 mL of the standard solution, and add the mobile phase to make exactly 20 mL. Confirm that the peak area of oxybutynin obtained with 10 μL of this solution is equivalent to 7 to 13% of that with 10 μL of the standard solution.

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, the number of theoretical plates and the symmetry factor of the peak of oxybutynin are not less than 5000 and not more than 1.5, respectively.

System repeatability: When the test is repeated 6 times with 10 μL of the standard solution under the above operating conditions, the relative standard deviation of the peak area of oxybutynin is not more than 2.0%.

Loss on drying *<2.41>* Not more than 3.0% (0.5 g, 105°C, 4 hours).

Residue on ignition *<2.44>* Not more than 0.1% (1 g).

Assay Weigh accurately about 0.5 g of Oxybutynin Hydrochloride, previously dried, dissolve in 70 mL of a mixture of acetic anhydride and acetic acid (100) (7:3), and titrate

94 *<2.50>* with 0.1 mol/L perchloric acid VS (potentiometric ti-
95 tration). Perform a blank determination in the same manner,
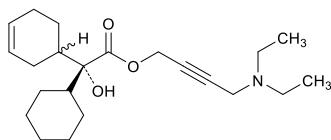96 and make any necessary correction.

97   Each mL of 0.1 mol/L perchloric acid VS
98              $= 39.40$ mg of $C_{22}H_{31}NO_3 \cdot HCl$

99 **Containers and storage**   Containers－Tight containers.
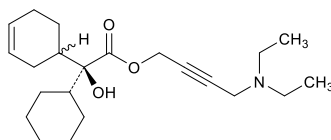100   Storage－Light-resistant.

101 **Others**
102 Related substance A:
103 4-(Diethylamino)but-2-yn-1-yl (2*R*)-2-(cyclohex-3-en-1-yl)-
104 2-cyclohexyl-2-hydroxyacetate

105

106 4-(Diethylamino)but-2-yn-1-yl (2*S*)-2-(cyclohex-3-en-1-yl)-
107 2-cyclohexyl-2-hydroxyacetate

108