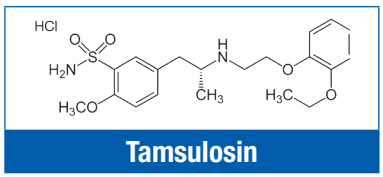
APPLICATIONS

Tamsulosin Hydrochloride and Related Substances

Ph. Eur. monograph 2131



Tamsulosin

Overview

The Ph. Eur. Monograph 2131 outlines the separation of Tamsulosin from impurities. This method was studied and improvements were made to provide faster separations within allowable adjustments.

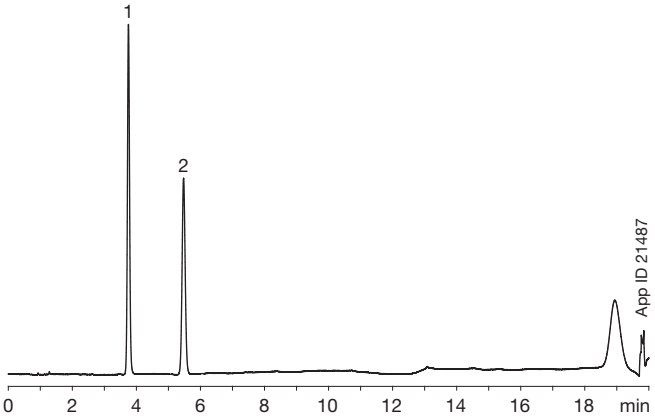
Ph. Eur. Monograph 2131 Details- Tamsulosin (A)	
Reference Solution	**(b)** Dissolve 4 mg of Tamsulosin Impurity D CRS* and 4 mg Tamsulosin Hydrochloride CRS* in the mobile phase and dilute to 20.0 mL with the mobile phase. Dilute 2.0 mL of this solution to 20.0 mL with the mobile phase. **(c)** Dissolve 4 mg of Tamsulosin Impurity H CRS* and 4 mg Tamsulosin Hydrochloride CRS* in the mobile phase and dilute to 20.0 mL with the mobile phase. Dilute 2.0 mL of this solution to 20.0 mL with the mobile phase.
Column	
Size	150 x 4.6 mm
Stationary Phase	Octadecylsilyl silica gel for chromatography R (5 µm).
Temperature	40 °C
Mobile Phase	Dissolve 3.0 g of sodium hydroxide R in a mixture of 8.7 mL of perchloric acid R and 1.9 L of water R; adjust to pH 2.0 with 0.5 M sodium hydroxide and dilute to 2 L with water R; to 1.4 L of this solution, add 600 mL of acetonitrile R.
Flow Rate	1.3 mL/min
Detection	Spectrophotometer @ 225 nm
Injection	10 µL
Run Time	1.5 times the retention of Tamsulosin (about 6 min)
System Suitability	
Reference Solution (b)	Minimum resolution of 6.0 between peaks due to Impurity D and Tamsulosin

* Tamsulosin impurity D CRS* (Y0000651), Tamsulosin Impurity H CRS (Y0000652) and Tamsulosin Hydrochloride CRS (Y0000650) were purchased from European Directorate for the Quality of Medicines & HealthCare (EDQM) – Council of Europe; Postal address: 7 Allee Kastner CS 30026F - 67081 STRASBOURG (France).

Method 1

Original Method as Described in the Monograph

Column: Kinetex® Core-Shell C18 5 µm
Dimensions: 150 x 4.6 mm
Part No.: 00F-4601-E0
Flow Rate: 1.3 mL/min
Sample: 1. Impurity B
2. Tamsulosin
Elution Time of Last Peak: 5.47 min
Rs Impurity D and Tamsulosin: 11.78



Adjustments for Meeting System Suitability

(European Pharmacopeia 9.0, Chapter 2.2.46. Chromatographic separation techniques)

Method Parameter	Allowed Adjustments (isocratic elution)	Method 1
Mobile Phase pH	± 0.2 units	2 (as specified)
Concentration of Salts in Buffer	± 10 %	As specified in Monograph 2131 Details Table
Composition of the Mobile Phase	± 30 % of the minor solvent component relative or 2 % absolute, whichever is the larger. No other component is altered by more than 10 % absolute.	As specified in Monograph 2131 Details Table
Wavelength of Detector	No deviations permitted	225 nm (as specified)
Injection Volume	May be decreased, provided detection and repeatability of the peak(s) to be determined are satisfactory.	10 µL (as specified)
Column Temperature	± 10 %	40 °C (as specified)
Stationary Phase	No change of the identity of the substituent permitted (e.g. no replacement of C8 by C18)	Octadecylsilyl silica gel for chromatography (as specified)
Column Length	± 70 %	150 mm (as specified)
Column Internal Diameter	± 25 %	4.6 mm (as specified)
Particle Size	-50 %	5 µm (as specified)
Flow Rate	± 50 %	1.3 mL/min (as specified)

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Ph. Eur. Monograph 2131 Details- Tamsulosin (B)

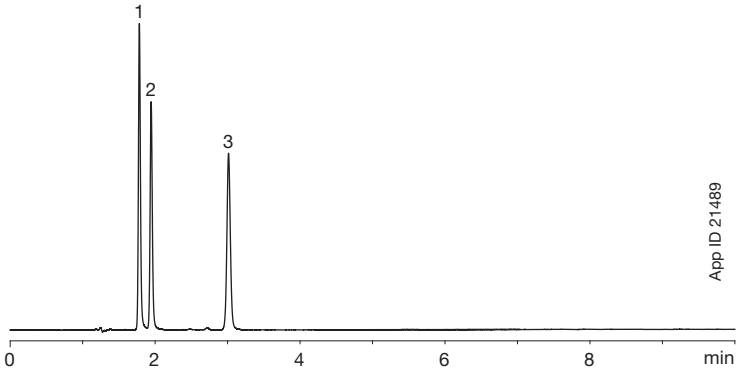
Reference Solution	**(c)** Dissolve 4 mg of Tamsulosin Impurity H CRS* and 4 mg Tamsulosin Hydrochloride CRS* in the mobile phase and dilute to 20.0 mL with the mobile phase. Dilute 2.0 mL of this solution to 20.0 mL with the mobile phase.
Column	
Size	150 x 4.6 mm
Stationary Phase	Octadecylsilyl silica gel for chromatography R (5 µm).
Temperature	40 °C
Mobile Phase	Dissolve 3.0 g of sodium hydroxide R in a mixture of 8.7 mL of perchloric acid R and 1.9 L of water R; adjust to pH 2.0 with 0.5 M sodium hydroxide and dilute to 2 L with water R; add 2 L of acetonitrile R.
Flow Rate	1.0 mL/min
Detection	Spectrophotometer @ 225 nm
Injection	10 µL
Run Time	5 times the retention of Tamsulosin (about 2.5 min)
System Suitability	
Reference Solution (c)	Minimum resolution of 2.0 between peaks due to Tamsulosin and Impurity H

* Tamsulosin Impurity D CRS* (Y0000651), Tamsulosin Impurity H CRS (Y0000652) and Tamsulosin Hydrochloride CRS (Y0000650) were purchased from European Directorate for the Quality of Medicines & HealthCare (EDQM) – Council of Europe; Postal address: 7 Allee Kastner CS 30026F - 67081 STRASBOURG (France).

Method 1

Original Method as Described in the Monograph

Column: Kinetex® Core-Shell C18 5 µm
Dimensions: 150 x 4.6 mm
Part No.: 00F-4601-E0
Flow Rate: 1.0 mL/min
Sample: 1. Impurity D
2. Tamsulosin
3. Impurity H
Elution Time of Last Peak: 3.01 min
Rs Tamsulosin and Impurity H: 15.37



Method 1Adjustments for Meeting System Suitability
(European Pharmacopeia 9.0, Chapter 2.2.46. Chromatographic separation techniques)

Method Parameter	Allowed Adjustments (isocratic elution)	Method 1
Mobile Phase pH	± 0.2 units	2 (as specified)
Concentration of Salts in Buffer	± 10 %	As specified in Monograph 2131 Details Table
Composition of the Mobile Phase	± 30 % of the minor solvent component relative or 2 % absolute, whichever is the larger. No other component is altered by more than 10 % absolute.	As specified in Monograph 2131 Details Table
Wavelength of Detector	No deviations permitted	225 nm (as specified)
Injection Volume	May be decreased, provided detection and repeatability of the peak(s) to be determined are satisfactory.	10 µL (as specified)
Column Temperature	± 10 °C	40 °C (as specified)
Stationary Phase	No change of the identity of the substituent permitted (e.g. no replacement of C18 by C8)	Octadecylsilyl silica gel for chromatography
Column Length	± 70 %	150 mm (as specified)
Column Internal Diameter	± 25 %	4.6 mm (as specified)
Particle Size	-50 %	5 µm (as specified)
Flow Rate	± 50 %	1.0 mL/min (as specified)

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Kinetex® Ordering Information

5 µm Minibore Columns (mm)					SecurityGuard™ ULTRA Cartridges‡
Phases	30 x 2.1	50 x 2.1	100 x 2.1	150 x 2.1	3/pk
C18	00A-4601-AN	00B-4601-AN	00D-4601-AN	00F-4601-AN	AJ0-8782
					for 2.1 mm ID

5 µm MidBore™ Columns (mm)				SecurityGuard ULTRA Cartridges‡
Phases	50 x 3.0	100 x 3.0	150 x 3.0	3/pk
C18	00B-4601-Y0	00D-4601-Y0	00F-4601-Y0	AJ0-8775
				for 3.0 mm ID

5 µm Analytical Columns (mm)					SecurityGuard ULTRA Cartridges‡
Phases	50 x 4.6	100 x 4.6	150 x 4.6	250 x 4.6	3/pk
C18	00B-4601-E0	00D-4601-E0	00F-4601-E0	00G-4601-E0	AJ0-8768
					for 4.6 mm ID

5 µm Semi-Preparative Columns (mm)			SecurityGuard SemiPrep Cartridges***
Phases	150 x 10	250 x 10	3/pk
C18	00F-4601-N0	00G-4601-N0	AJ0-9278
			for 9-16 mm ID

‡SecurityGuard ULTRA Cartridges require holder, Part No.: AJ0-9000

***SemiPrep SecurityGuard Cartridges require holder, Part No.: AJ0-9281

APPLICATIONS

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