

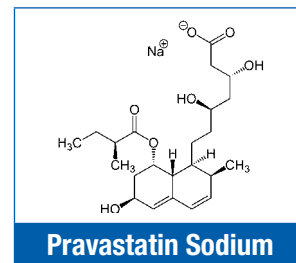
APPLICATION

Pravastatin Sodium and Related Substances

Ph. Eur. monograph 2059

Overview

The Ph. Eur. Monograph 2059 outlines the separation of Pravastatin from impurities. This method was studied and improvements were made to provide faster separations within allowable adjustments.



Ph. Eur. Monograph 2059 Details

Solvent Mixture	Methanol R, water R (9:11 V/V)
Test Solution	(a) Dissolve 0.1000g of Pravastatin 1,1,3,3-tetramethylbutylamine CRS* in the solvent mixture and dilute to 100.0 mL with the solvent mixture (b) Dilute 10.0 mL of the test solution (a) to 100.0 mL with the solvent mixture
Reference Solution (a)	Dissolve the contents of a vial of Pravastatin Impurity A CRS* in 1.0 mL of test solution (b)

Column

Size	150 x 4.6 mm
Stationary Phase	Octadecylsilyl silica gel for chromatography R (5 µm)
Temperature	25 °C
Mobile Phase	Glacial acetic acid R, trimethylamine R, methanol R, water R (1:1:450:550 V/V/V/V)
Flow Rate	1.3 mL/min
Detection	Spectrophotometer @ 238 nm
Injection	10 µL
Run Time	2.5 times the retention time of Pravastatin
Elution Order	1. Impurity A 2. Pravastatin

system Suitability

Minimum resolution of 7.0 between peaks due to Impurity A and Pravastatin

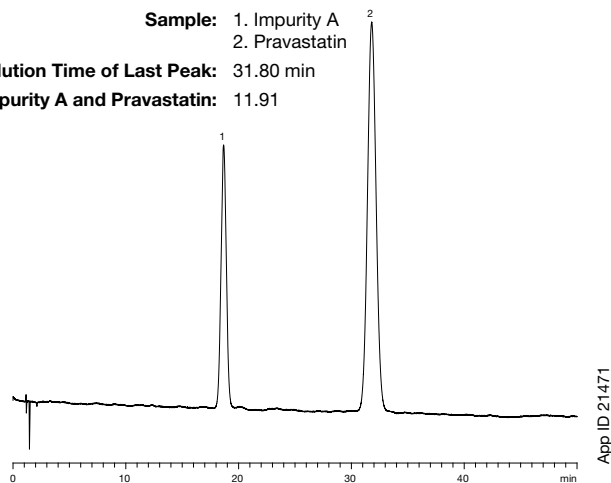
* Pravastatin 1,1,3,3-tetramethylbutylamine CRS (Y0000204) and Pravastatin Impurity A CRS (Y0000223) were purchased from European Directorate for the Quality of Medicines & HealthCare (EDQM) – Council of Europe; Postal address: 7 Allée Kastner CS 30026F - 67081 STRASBOURG (France).

Method 1

Original Method as Described in the Monograph

Column: Luna[®] C18(2) 5 µm Fully Porous
Dimensions: 150 x 4.6 mm
Part No.: 00F-4252-E0
Flow Rate: 1.3 mL/min
Sample: 1. Impurity A
2. Pravastatin

Elution Time of Last Peak: 31.80 min
Rs Impurity A and Pravastatin: 11.91

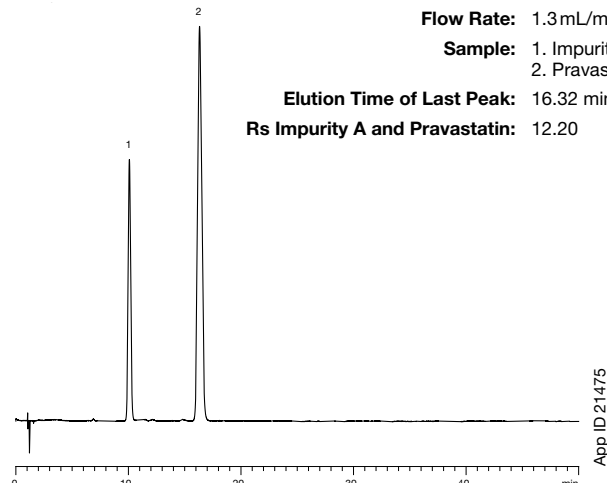


Method 2

Faster Method Utilizing Core-Shell Technology

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 A
2. Pravastatin

Elution Time of Last Peak: 16.32 min
Rs Impurity A and Pravastatin: 12.20



Adjustments for Meeting System Suitability

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

Method Parameter	Allowed Adjustments (isocratic elution)	Method 1	Method 2
Mobile Phase pH	± 0.2 units	As specified	As specified
Concentration of Salts in Buffer	± 10 %	As specified in Monograph 2059 Details Table	As specified
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 2059 Details Table	As specified
Wavelength of Detector	No deviations permitted	238 nm (as specified)	As specified
Injection Volume	May be decreased, provided detection and repeatability of the peak(s) to be determined are satisfactory.	10 µL (as specified)	As specified
Column Temperature	± 10 °C	25 °C (as specified)	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 (as specified)	As specified
Column Length	± 70 %	150 mm (as specified)	As specified
Column Internal Diameter	± 25 %	4.6 mm (as specified)	As specified
Particle Size	-50 %	5 µm (as specified)	As specified
Flow Rate	± 50 %	1.3 mL/min (as specified)	As specified

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

Luna® Ordering Information

5 µm Microbore and Minibore Columns (mm)								SecurityGuard™ Cartridges (mm)	
Phases	50 x 1.0	150 x 1.0	250 x 1.0	30 x 2.0	50 x 2.0	150 x 2.0	250 x 2.0	4 x 2.0*	
C18(2)	00B-4252-A0	00F-4252-A0	00G-4252-A0	00A-4252-B0	00B-4252-B0	00F-4252-B0	00G-4252-B0	/10pk	
								AJ0-4286	
								for ID: 2.0-3.0 mm	

5 µm MidBore and Analytical Columns (mm)								SecurityGuard™ Cartridges (mm)	
Phases	30 x 3.0	50 x 3.0	150 x 3.0	250 x 3.0	30 x 4.6	50 x 4.6	75 x 4.6	4 x 2.0*	4 x 3.0*
C18(2)	00A-4252-Y0	00B-4252-Y0	00F-4252-Y0	00G-4252-Y0	00A-4252-E0	00B-4252-E0	00C-4252-E0	/10pk	/10pk
								AJ0-4286	AJ0-4287
								for ID: 2.0-3.0 mm	3.2-8.0 mm

5 µm Analytical and Semi-Prep Columns (mm)					SecurityGuard™ Cartridges (mm)	
Phases	100 x 4.6	150 x 4.6	250 x 4.6	250 x 10	4 x 3.0*	10 x 10 ²
C18(2)	00D-4252-E0	00F-4252-E0	00G-4252-E0	00G-4252-N0	/10pk	/3pk
					AJ0-4287	AJ0-7221
					for ID: 3.2-8.0 mm	9-16 mm

*SecurityGuard™ Analytical Cartridges require holder, Part No.: KJ0-4282

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



If Phenomenex products in this technical note do not provide at least an equivalent separation as compared to a competing product of the same particle size, similar phase and dimensions, return the product with comparative data within 45 days for a FULL REFUND.



APPLICATION

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