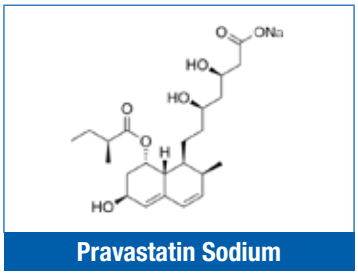
APPLICATION

Pravastatin Sodium
USP



Pravastatin Sodium

Overview
The related substances test of the USP monograph outlines the separation of all relevant impurities from Pravastatin Sodium. This method was studied and improvements were made to provide higher resolution (Rs) and a faster separation time within allowable adjustments

USP Monograph: Pravastatin Sodium Details

Diluent	Prepare a mixture of methanol and water (1:1)
Buffer pH 7.0	Prepare a 0.08 M phosphoric acid solution, adjust with triethylamine to pH 7.0, mix
Standard Solution*	Dissolve an accurately weighed quantity of USP Pravastatin 1,1,3,3-Tetramethylbutylamine RS in Diluent, and dilute quantitatively with Diluent to obtain a solution having a known concentration of about 1.25 µg of pravastatin 1,1,3,3-tetramethylbutylamine per mL
System Suitability Solution	Dissolve accurately weighed quantities of USP Pravastatin 1,1,3,3-Tetramethylbutylamine Rs and USP Pravastatin Related Compound A RS in Diluent to obtain a solution containing about 0.6 mg of USP Pravastatin 1,1,3,3 tetramethylbutylamine RS and 0.001 mg of USP Pravastatin Related Compound A RS per mL. (Note-USP Pravastatin Related Compound A RS is a sodium salt of 3α-hydroxisocompactin acid)
Test Solution*	Transfer about 50 mg of Pravastatin Sodium to a 100 mL volumetric flask, dissolve in and dilute with Diluent to volume, and mix

Column

Size	100 x 4.0 mm
Stationary Phase	3 µm, L1: Octadecyl silane chemically bonded to porous or non-porous silica or ceramic microparticles, 1.5 to 10 µm in diameter, or a monolithic rod
Mobile Phase	Use variable mixtures of Solution A and Solution B as directed for: **A.** Prepare a filtered and degassed mixture of water, Buffer pH 7.0, and acetonitrile (52:30:10) **B.** Prepare a filtered and degassed mixture of acetonitrile, Buffer pH 7.0, and water (60:30:10)

Gradient

Time	**%B**
0 – 3.0 min	0
3.0 – 26.5 min	0 → 100
26.5 – 26.6 min	100 → 0
26.6 – 30.0 min	0

Flow Rate	1.0 mL/min
Detection	Spectrophotometer @ 238 nm
Injection	10 µL

Relative Retention with Reference to Pravastatin**

Related Compound A	about 1.1

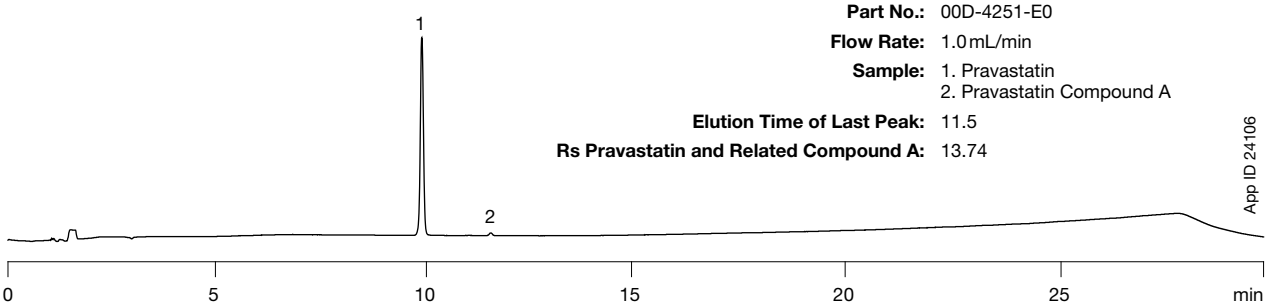
System Suitability
Minimum resolution of 2.0 between Pravastatin and Pravastatin Related Compound A

*The Standard solution and the Test solution are maintained at 15° C until injected into the chromatograph

** Retention times, relative retentions, and retardation factors are provided for information only and are not mandatory, no deviation allowance is defined.

Method 1
Original Method Outside Allowable Adjustments

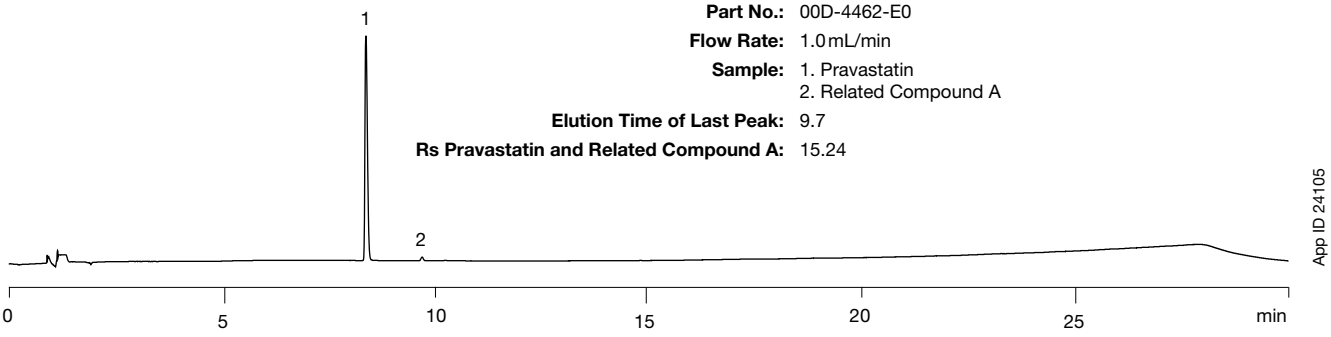
Column: Luna® C18(2) 3 µm Fully Porous
Dimensions: 100 x 4.6 mm
Part No.: 00D-4251-E0
Flow Rate: 1.0 mL/min
Sample: 1. Pravastatin
2. Pravastatin Compound A
Elution Time of Last Peak: 11.5
Rs Pravastatin and Related Compound A: 13.74



Method 2

Faster and Higher Resolution Outside Allowable Adjustments

Column: Kinetex® Core-Shell C18 2.6 µm
Dimensions: 100 x 4.6 mm
Part No.: 00D-4462-E0
Flow Rate: 1.0 mL/min
Sample: 1. Pravastatin
2. Related Compound A
Elution Time of Last Peak: 9.7
Rs Pravastatin and Related Compound A: 15.24



Adjustments for Meeting System Suitability

Method Parameter	Allowed Adjustments (gradient elution)	Method 1	Method 2
Mobile Phase pH	± 0.2 units	As specified	As specified
Concentration of Salts in Buffer	± 10 %	As specified	As specified
Composition of the Mobile Phase	Changes to gradient composition are not recommended	As specified in Monograph Details Table	As specified
Wavelength of Detector	No deviations permitted	238 nm (as specified)	As specified
Injection Volume	Can be adjusted as much as needed; must be consistent with linearity, precision, and detection requirements	10 µL (as specified)	As specified
Column Temperature	± 10 °C	Ambient (as specified)	As specified
Stationary Phase	No change of the identity of the substituent permitted (e.g. no replacement of C18 by C8)	L1 (as specified)	As specified
Column Length	No deviations permitted	100 mm (as specified)	As specified
Column Internal Diameter	No deviations permitted	4.6 mm (+15 %)	4.6 mm (+15)
Particle Size	No deviations permitted	3 µm (as specified)	2.6 µm (-13 %)
Flow Rate	No deviations permitted	1.0 mL/min (as specified)	As specified

*Alternatively (as for the application of particle size adjustment to superficially porous particles), other L/dp combinations can be used provided that the number of theoretical plates (N) is within -25 % to +50 %

Kinetex® Ordering Information

2.6 µm Minibore Columns (mm)						SecurityGuard™ ULTRA Cartridges‡
Phases	30 x 2.1	50 x 2.1	75 x 2.1	100 x 2.1	150 x 2.1	3/pk
C18	00A-4462-AN	00B-4462-AN	00C-4462-AN	00D-4462-AN	00F-4462-AN	AJ0-8782
						for 2.1 mm ID

2.6 µm MidBore™ Columns (mm)						SecurityGuard ULTRA Cartridges‡
Phases	30 x 3.0	50 x 3.0	75 x 3.0	100 x 3.0	150 x 3.0	3/pk
C18	00A-4462-Y0	00B-4462-Y0	00C-4462-Y0	00D-4462-Y0	00F-4462-Y0	AJ0-8775
						for 3.0 mm ID

2.6 µm Analytical Columns (mm)						SecurityGuard ULTRA Cartridges‡
Phases	30 x 4.6	50 x 4.6	75 x 4.6	100 x 4.6	150 x 4.6	3/pk
C18	00A-4462-E0	00B-4462-E0	00C-4462-E0	00D-4462-E0	00F-4462-E0	AJ0-8768
						for 4.6 mm ID

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

Luna® Ordering Information

3 µm Capillary Columns (mm)						Guard Columns (mm)	
Phases	50 x 0.30	150 x 0.30	50 x 0.50	150 x 0.50	250 x 0.50	20 x 0.30	20 x 0.50
3 µm C18(2)	00B-4251-AC	00F-4251-AC	00B-4251-AF	00F-4251-AF	—	03M-4251-AC	03M-4251-AF

3 µm Microbore and Minibore Columns (mm)							SecurityGuard Cartridges (mm)
Phases	50 x 1.0	150 x 1.0	30 x 2.0	50 x 2.0	100 x 2.0	150 x 2.0	4 x 2.0*
							/10pk
C18(2)	00B-4251-A0	00F-4251-A0	00A-4251-B0	00B-4251-B0	00D-4251-B0	00F-4251-B0	AJ0-4286
							for ID: 2.0-3.0 mm

3 µm MidBore™ and Analytical Columns (mm)									SecurityGuard™ Cartridges (mm)	
Phases	30 x 3.0	50 x 3.0	150 x 3.0	30 x 4.6	50 x 4.6	75 x 4.6	100 x 4.6	150 x 4.6	4 x 2.0*	4 x 3.0*
									/10pk	/10pk
C18(2)	00A-4251-Y0	00B-4251-Y0	00F-4251-Y0	00A-4251-E0	00B-4251-E0	00C-4251-E0	00D-4251-E0	00F-4251-E0	AJ0-4286	AJ0-4287
									for ID: 2.0-3.0 mm	3.2-8.0 mm

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

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

guarantee

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, similiar phase and dimensions, return the product with comparative data within 45 days for a FULL REFUND.

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