

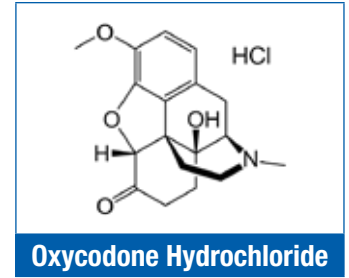
APPLICATION

Oxycodone Hydrochloride and Related Substances

Ph. Eur. monograph 1793

Overview

The Ph. Eur. Monograph 1793 outlines the separation of Oxycodone Hydrochloride from impurities. This method was studied and improvements were made to provide faster separations within allowable adjustments.



Ph. Eur. Monograph 1793 Details

Test Solution Dissolve 0.100 g of Oxycodone Hydrochloride CRS* in a 1 % V/V solution of dilute acetic acid R and dilute to 50.0 mL with the same solvent

Reference Solution p (a) Dissolve 20.0 mg of Oxycodone Impurity D CRS* in a 1.0 % V/V solution of dilute acetic acid R and dilute to 10.0 mL with the same solution
(b) To 1.0 mL of the test solution, add 1 mL of reference solution (a) and dilute to 100.0 mL with a 1 % V/V solution of dilute acetic acid R. Dilute 1.0 mL of the solution to 10.0 mL with a 1.0 % V/V solution of dilute acetic acid R.

Column

Size 150 x 4.6 mm
Stationary Phase Octadecylsilyl silica gel for chromatography R (5 µm)
Temperature 40 °C
Mobile Phase A: Mix 830 mL of a 1.1 g/L solution of sodium heptanesulfonate monohydrate R previously adjusted to pH 2.0 with a mixture of equal volumes of phosphoric acid R and water R, with 70 mL of acetonitrile R and 100 mL of methanol R
 B: Mix 600 mL of a 1.1 g/L solution of sodium heptanesulfonate monohydrate R previously adjusted to pH 2.0 with a mixture of equal volumes of phosphoric acid R and water R, with 150 mL of acetonitrile R and 250 mL of methanol R
Gradient **Time (min)** **%B**
 0 – 60 min 0 - 50
Flow Rate 1.5 mL/min
Detection Spectrophotometer @ 230 nm
Injection 20 µL

Relative Retention with Reference to Oxycodone (about 24 min)**

Impurity B about 0.7

System Suitability

Reference Solution (a) Minimum resolution of 3.0 between peaks due to Oxycodone and Impurity D

* Oxycodone Hydrochloride CRS (Y0000492) and Oxycodone Impurity D CRS (Y0000453) 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).

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

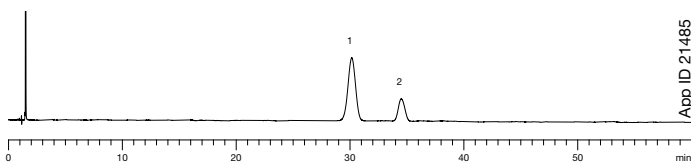
Method 1

Original Method as Described in the Monograph

Column: Luna[®] C18(2) 5µm Fully Porous
Dimensions: 150 x 4.6 mm
Part No.: 00F-4252-E0
Flow Rate: 1.5 mL/min
Sample: 1. Oxycodone
 2. Impurity B

Elution Time of Last Peak: 34.51 min

Rs Oxycodone and Impurity D: 3.79



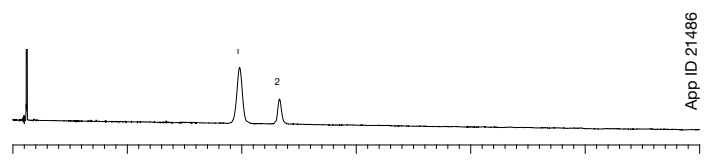
Method 2

Faster Method Utilizing Core-Shell Technology

Column: Kinetex[®] Core-Shell C18 5µm
Dimensions: 150 x 4.6 mm
Part No.: 00F-4601-E0
Flow Rate: 1.5 mL/min
Sample: 1. Oxycodone
 2. Impurity B

Elution Time of Last Peak: 23.30 min

Rs Oxycodone and Impurity D: 4.75



Adjustments for Meeting System Suitability

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

Method Parameter	Allowed Adjustments (gradient elution)	Method 1	Method 2
Mobile Phase pH	No adjustment permitted	2 (as specified)	As specified
Concentration of Salts in Buffer	No adjustment permitted	As specified in Monograph 1793 Details Table	As specified
Composition of the Mobile Phase	Minor adjustments of the composition of the mobile phase and the gradient are acceptable, if the system suitability requirements are met, the principle peak(s) elute(s) within $\pm 15\%$ of the indicated retention time(s) and the final elution power of the mobile phase is not weaker in elution power than the prescribed composition	As specified in Monograph 1793 Details Table	As specified
Wavelength of Detector	No deviations permitted	230 nm (as specified)	As specified
Injection Volume	May be decreased, provided detection and repeatability of the peak(s) to be determined are satisfactory.	20 μ L (as specified)	As specified
Column Temperature	$\pm 5^\circ\text{C}$	40 $^\circ\text{C}$ (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	May be decreased, $\pm 70\%$	150 mm (as specified)	As specified
Column Internal Diameter	$\pm 25\%$	4.6 mm (as specified)	As specified
Particle Size	No adjustment permitted	5 μ m (as specified)	As specified
Flow Rate	Acceptable when changing the column dimensions	1.5 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	AJO-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-YO	00D-4601-YO	00F-4601-YO	AJO-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	AJO-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	AJO-9278 for 9-16 mm ID

[†]SecurityGuard ULTRA Cartridges require holder, Part No.: AJO-9000

^{***}SemiPrep SecurityGuard Cartridges require holder, Part No.: AJO-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 AJO-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 AJO-4286	/10pk AJO-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 [‡]
C18(2)	00D-4252-E0	00F-4252-E0	00G-4252-E0	00G-4252-N0	/10pk AJO-4287	/3pk AJO-7221 for ID: 3.2-8.0 mm 9-16 mm

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

[‡]SemiPrep SecurityGuard Cartridges require holder, Part No.: AJO-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, similar phase and dimensions, return the product with comparative data within 45 days for a FULL REFUND.



APPLICATION

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