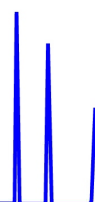




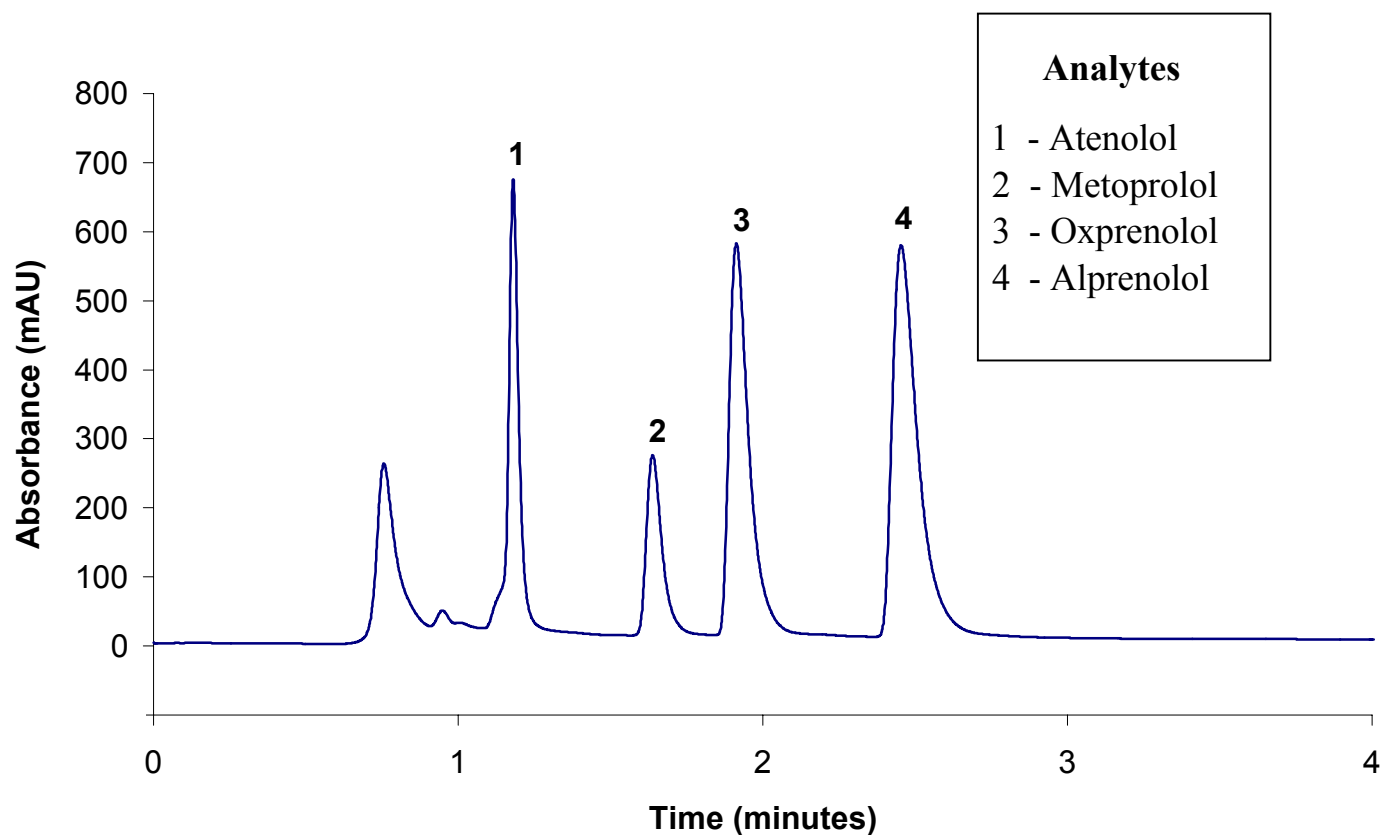
ZirChrom®

Technical
Bulletin #236



... For Peak Performance

Separation of Beta-Blockers



LC Conditions

Column: **DIAMOND BOND™18**, 100 × 4.6 mm

Mobile Phase: 20/20/60 A/B/C

A: ACN

B: THF

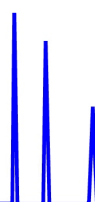
C: 20 mM Ammonium phosphate, pH 11.0

Flow rate: 1.0 mL/min.

Temperature: 75 °C

Injection volume: 5 µL

Detection: 254 nm





ZirChrom®

LC/MS Compatible Separation of Beta-Blockers on ZirChrom®-MS

Clayton McNeff, Ph.D., Bingwen Yan, Ph.D. and Steven Rupp
ZirChrom Separations, Inc.

Technical Bulletin # 298

The chromatography of basic pharmaceuticals (with amine functionalities) on C18-silica phases can be difficult due to secondary interactions that cause tailed peaks in the neutral pH range where most silica phases are stable¹. ZirChrom®-MS is a new zirconia-based reversed-phase column that has mixed mode retention characteristics which allow for LC/MS chromatography of highly basic amines with excellent peak shape and efficiency.

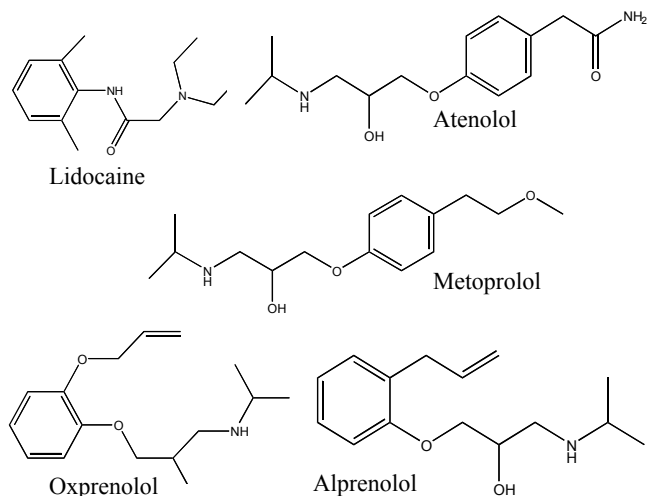


Figure 1: Structures for the Compounds of Interest.

Introduction

The chromatography of basic drugs on C18-silica phases has traditionally been problematic. The surface chemistry of zirconia-based phases is dominated by Lewis acid sites, rather than the Bronsted acid sites, which dominate the surface chemistry of silica phases. The mixed-mode retention character of ZirChrom®-MS (cation-exchange and reversed-phase) allows separations that were previously difficult using conventional silica C18 phases. This application note shows an impressive LC/MS compatible separation of beta-blockers in a highly organic, near neutral pH mobile phase.

Experimental

A mixture of five amine-containing compounds was separated using a ZirChrom®-MS column at 35 °C. The separation conditions were as follows:

Column: ZirChrom®-MS, 50 mm x 4.6 mm i.d.
(Part Number: MS01-0546)
Mobile Phase: Isocratic elution: 65/35 A/B
A: acetonitrile
B: 10mM ammonium acetate, pH 5.0
Temperature: 35 °C

Flow Rate: 1.0 ml/min.
Injection Vol.: 5 µl
Pressure Drop: 59 bar
Detection: UV at 254 nm

Five Beta-Blockers were separated using simple acetonitrile/water isocratic elution and an LC/MS friendly acetate buffer. The selectivity of all five compounds is excellent using only a short 5 cm long column.

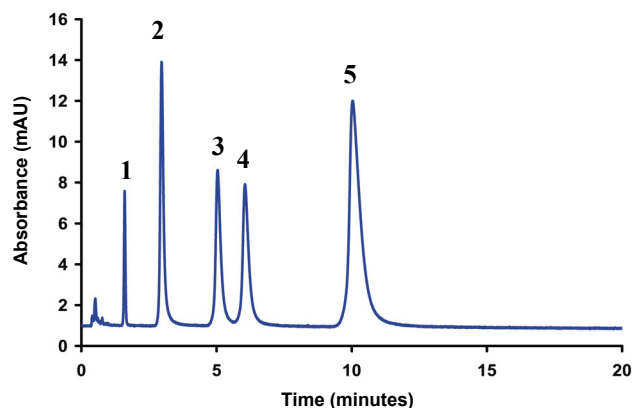


Figure 2: Separation of 1= Lidocaine, 2= Atenolol, 3= Metoprolol, 4= Oxprenolol, and 5= Alprenolol.

This method can be tailored to your specific application needs. ZirChrom method developers can help to optimize and transfer this method to your site. Please contact ZirChrom technical support at 1-866-STABLE-1 or support@zirchrom.com for details.

ZirChrom phases offer unique selectivity, high efficiency, and excellent chemical and thermal stability.

References

- 1) G.B. Cox, *J. Chromatography A*. **656**, 353, 1993.

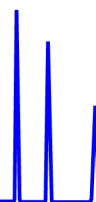
ZirChrom Separations, Inc.
617 Pierce Street, Anoka, MN 55303
1-866-STABLE-1
support@zirchrom.com

Visit www.zirchrom.com for more application notes using ultra-stable, high efficiency ZirChrom columns.



ZirChrom®

Technical
Bulletin #201

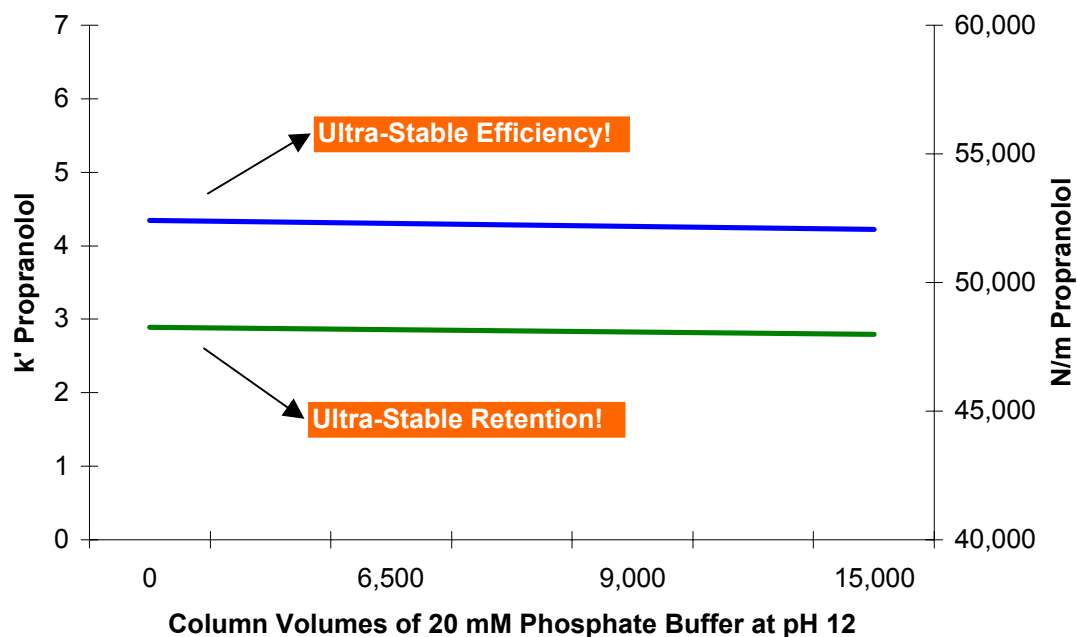


... For Peak Performance

ZirChrom®-PBD: Ultra-Stable for Basic Drugs at pH 12

Analytes

- 1 - Labetalol
- 2 - Atenolol
- 3 - Acebutolol
- 4 - Metoprolol
- 5 - Oxprenolol
- 6 - Lidocaine
- 7 - Quinidine
- 8 - Alprenolol
- 9 - Propranolol



LC Conditions

Column: ZirChrom®-PBD

Mobile Phase: 28/72 A/B

A: Acetonitrile

B: 20 mM potassium phosphate at pH=12.0

Flow rate: 1.0 mL/min.

Temperature: 30 °C

Detection: 254 nm

