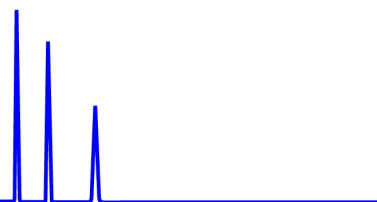




ZirChrom®

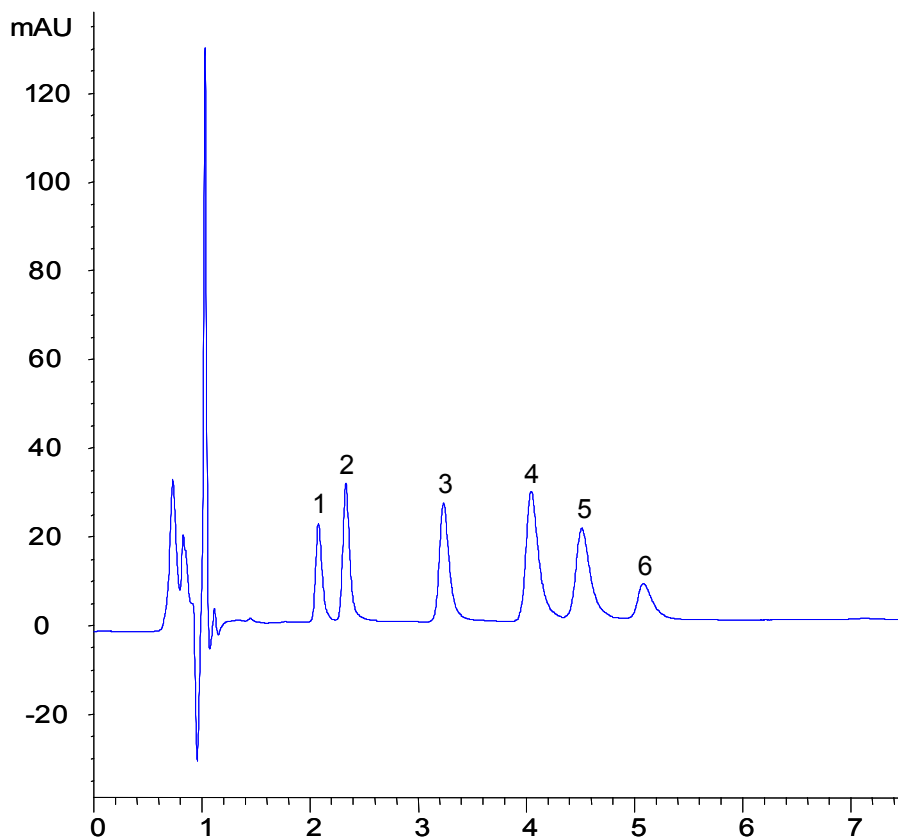
Technical
Bulletin #250



... For Peak Performance

Separation of Antihistamines

- Analytes**
- 1 - Pheniramine
 - 2 - Doxylamine
 - 3 - Chlorpheniramine
 - 4 - Methapyrilene
 - 5 - Tripeleennamine
 - 6 - Cyclizine



LC Conditions

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

Mobile Phase: 10/25/65 A/B/C

A: ACN

B: THF

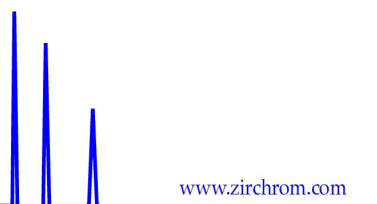
C: 50mM Tetramethylammonium hydroxide,
10mM Triethylamine, pH 12.2

Flow rate: 1.0 mL/min.

Temperature: 75 °C

Injection volume: 5 µL

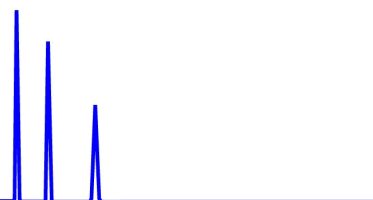
Detection: 254 nm





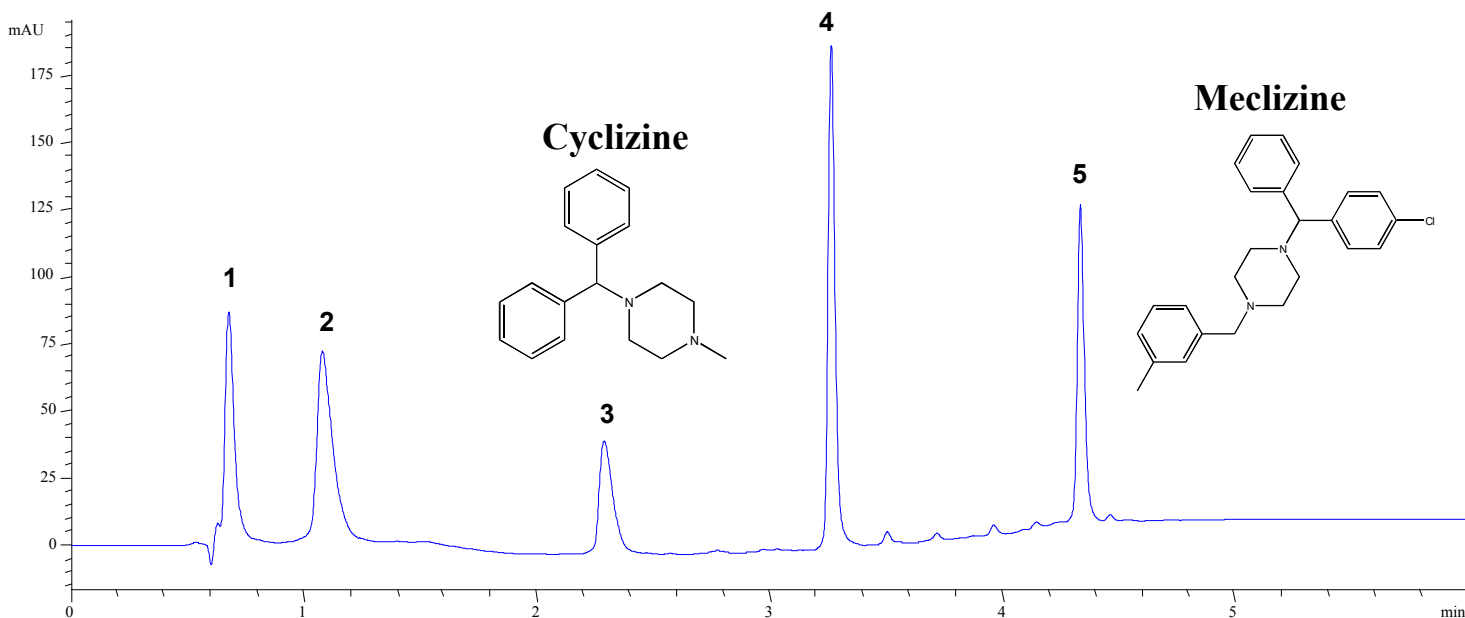
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Technical
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... For Peak Performance

Antihistamines on ZirChrom®-PS



Analytes

1 - Tripeleennamine 2 - Triprolidine 3 - Cyclizine 4 - Pyrrobutamine 5 - Meclizine

LC Conditions

Column: ZirChrom®-PS, 50 mm × 4.6 mm i.d.

Mobile Phase: Gradient Elution

Flow rate: 1.0 mL/min.

Temperature: 40 °C

Injection volume: 0.5 µL

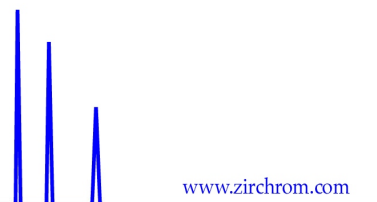
Detection: 254 nm

Back Pressure: 56 bar

Time (Minutes)	% A	%B
0	95	5
1	85	15
4	40	60

A: 25mM HCl, pH 1.9

B: ACN





Faster Analysis and Higher Efficiency with Thermally-Stable HPLC Columns

Technical Bulletin #269

Dwight Stoll, Peter W. Carr
ZirChrom Separations, Inc.

Chromatographers have long known that modest increases in operating temperature can dramatically improve both the efficiency and speed of an HPLC separation. Until now, this potential has been unrealized because of the short lifetimes of high efficiency stationary phases at elevated temperature. This note shows that fast analysis with high efficiency is now easy to achieve using ultra-stable zirconia columns.

The initial separation is shown in Figure 1. Then, the temperature was increased to 50 °C, and the eluent flow rate also increased to maintain the same system backpressure. The separation (not shown) was more than twice as fast, with the resolution of the two closely eluting compounds maintained.

Finally, the temperature was increased to 80 °C, again increasing the eluent flow rate to maintain system backpressure. Now the separation is 5 times faster, maintaining the same resolution of the closely eluting peaks (see Figure 2).

Figure 1: Separation of antihistamines at room temperature.
1=Doxylamine, 2=Methapyrilene, 3=Chlorpheniramine,
4=Triprolidine, 5=Meclizine

Introduction

In a recent article, David V. McCalley found large increases in the efficiency for basic compounds at elevated temperature¹. McCalley suggested both that basic compounds should be analyzed at high temperature and that columns should be developed that are stable at high temperature.

In addition to improved efficiency, high temperature operation allows for dramatic improvements in analysis speed. Raising the temperature decreases mobile phase viscosity, allowing for increased eluent flow rate (and faster analysis) without excessive backpressure.

Experimental

A mixture of antihistamines was separated at room temperature using a ZirChrom®-PBD column. The separation conditions were as follows:

Column: 4.6 mm x 100 mm ZirChrom-PBD
Mobile Phase: 29/71 ACN / 50mM Tetramethylammonium hydroxide, pH 12.2
Injection Vol.: 0.5 ul
Pressure Drop: 195 bar
Detection: UV at 254 nm

Figure 2: Separation of antihistamines at 80 °C. Analytes as in Figure 1.

Note that even temperature-sensitive compounds can benefit from modest increases in temperature, making faster analysis possible. ZirChrom's technical support group has extensive experience in this area, and would be happy to help you with your particular application.

ZirChrom columns combine the high efficiency usually associated with silica columns with complete chemical and thermal stability.

References

(1) David V. McCalley, J. Chrom. A 902, 311-321 (2000).

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