



ZirChrom®

Polymer Coated Titania for Analytical and Preparative Reversed-Phase Chromatography

EAS 2005

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ZirChrom

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Outline

- Properties of Titania
- RP Phase Stationary Phase
- Chromatographic Data
 - Selectivity Comparison of Sachtopore-RP, Silica C18, ZirChrom-CARB, and ZirChrom-PBD
 - Chemical and Thermal Stability Testing
 - Effect of Lewis Base Mobile Phase Additive on Elution of Basic Compounds

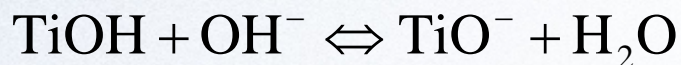
Conclusion - The Sachtopore[®]-RP shows very similar selectivity to ZirChrom[®]-PBD and has excellent stability from pH 1-12 and up to 100 °C.



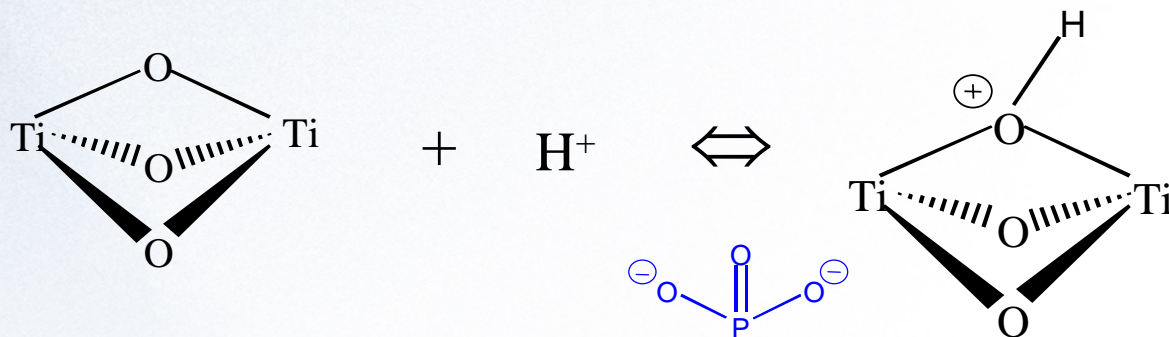
Surface Chemistry of Titania-Based Supports for HPLC



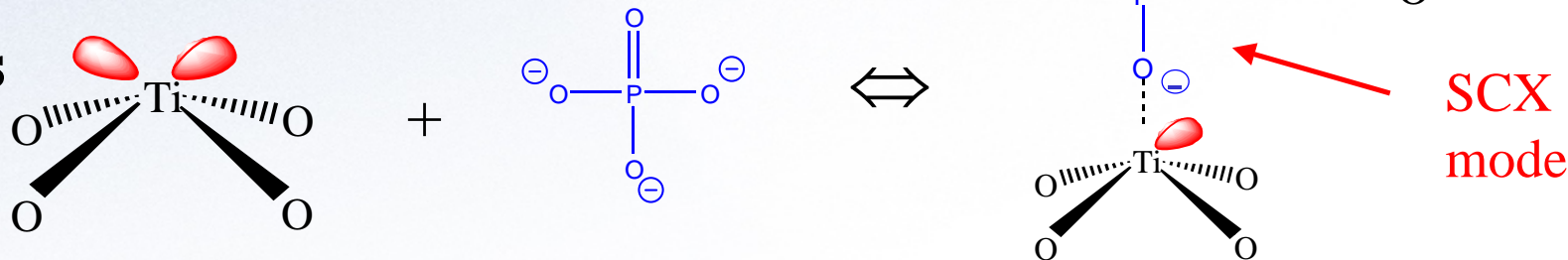
Weak Brönsted Acid:



Weak Brönsted Base:

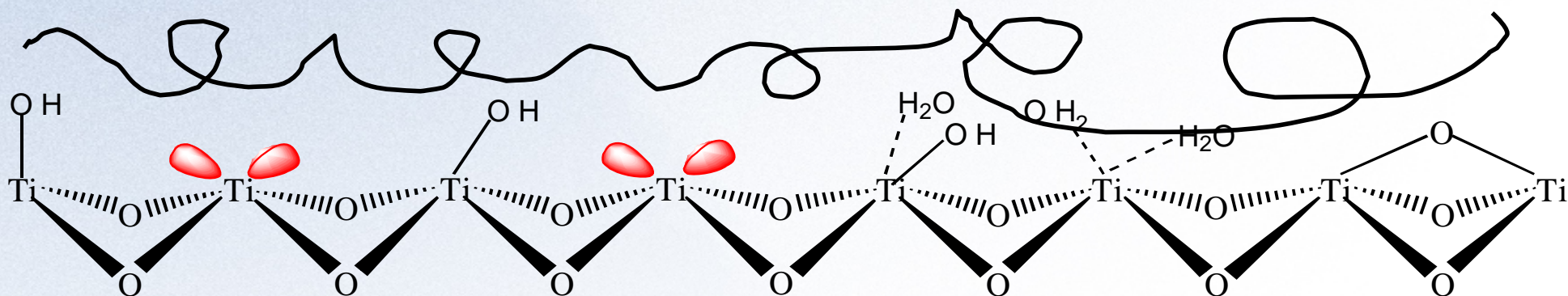
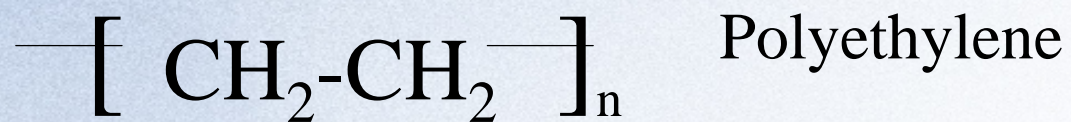


Strong Lewis Acid:





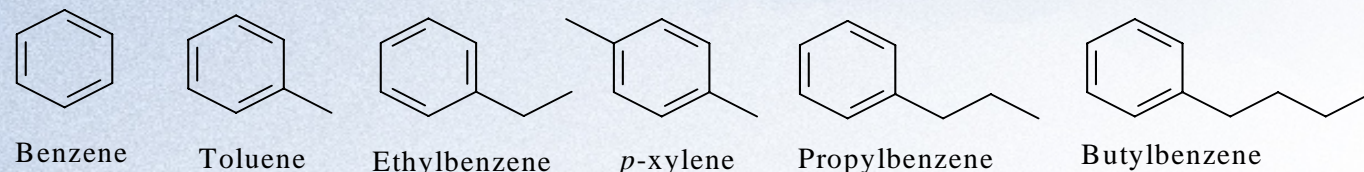
Polyethylene Coated Titania



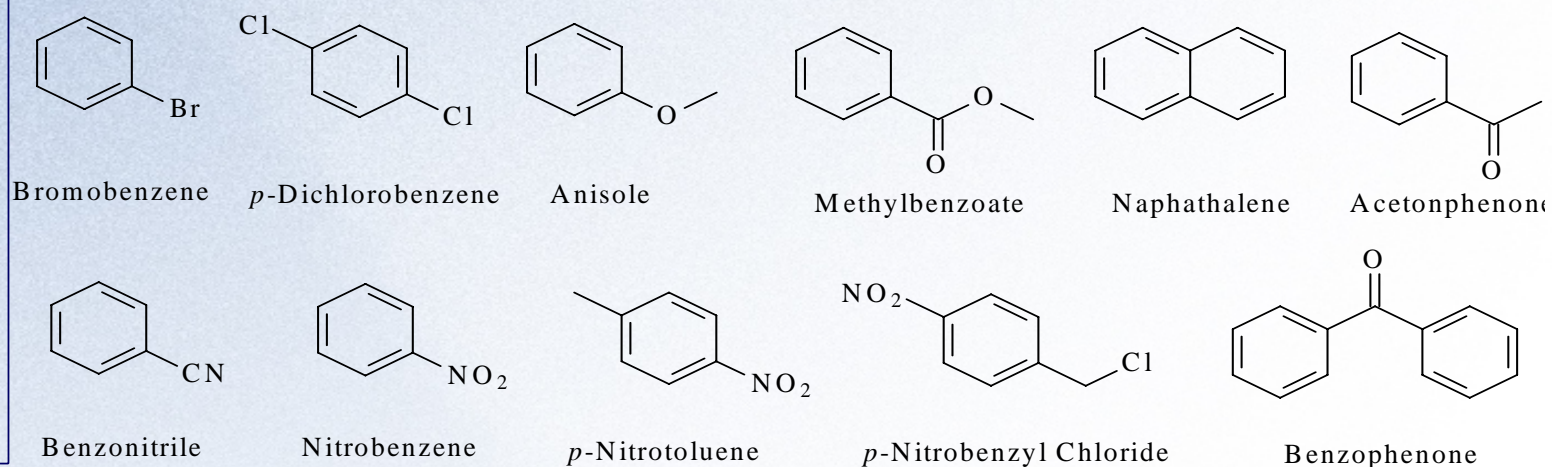


22 Non-electrolyte Solutes

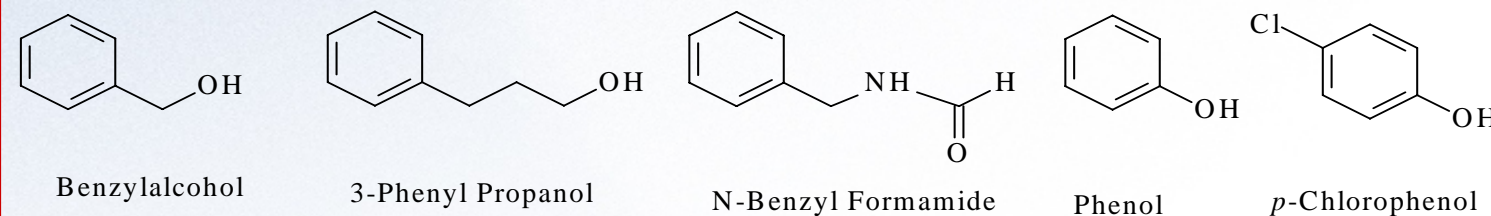
Nonpolar



Polar

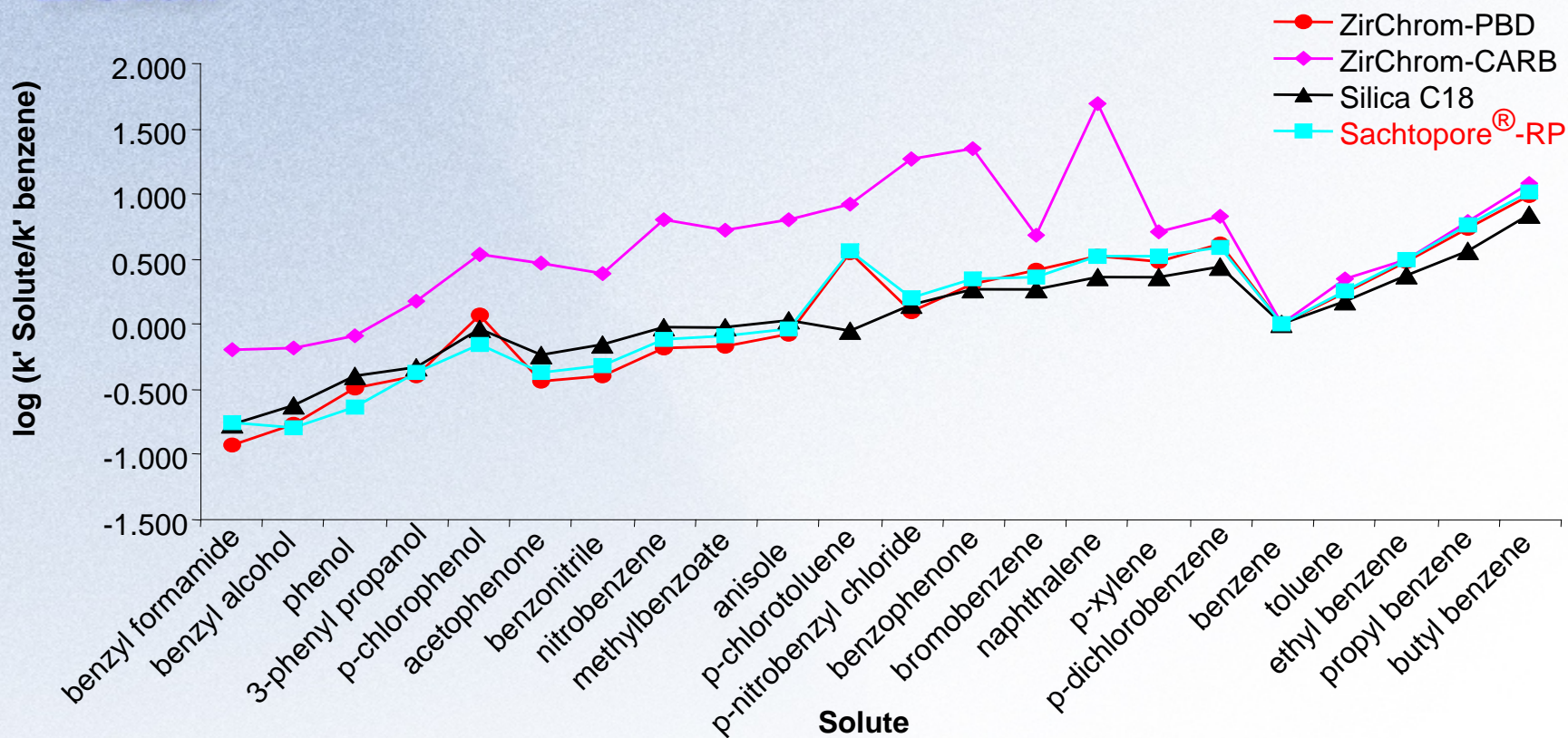


HB Donor





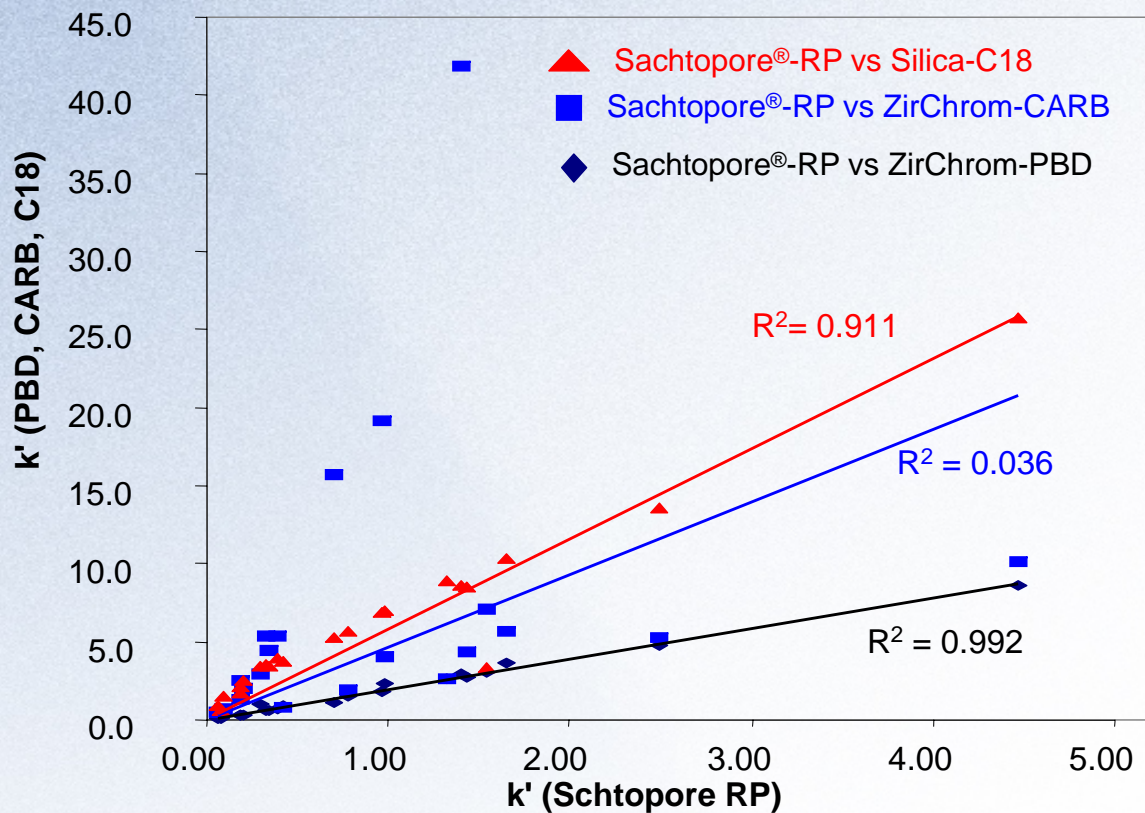
Selectivity Comparison



LC Conditions: Mobile phase, 40/60 ACN/Water; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5ul; Detection at 254nm.



K-K Comparison



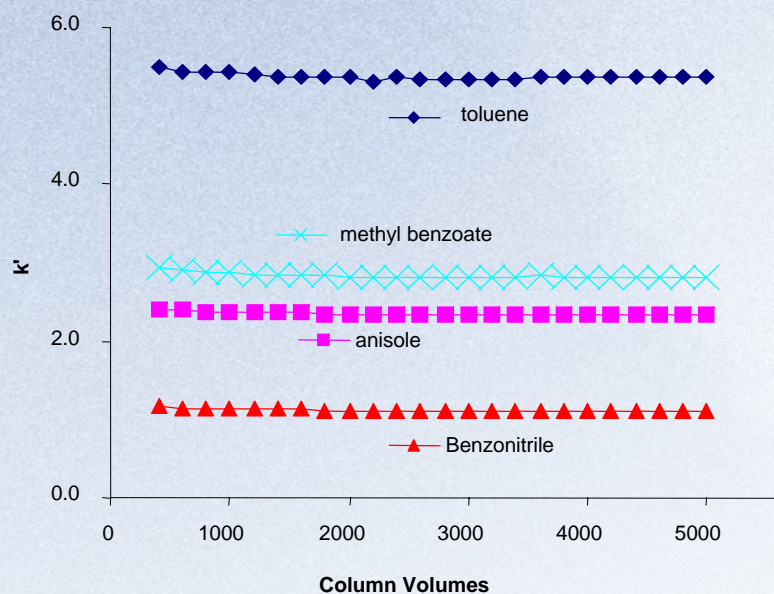
LC Conditions: Mobile phase, 40/60 ACN/Water; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5ul; Detection at 254nm.

Reference: Melander, W.; Stoveken, J.; Horvath, C. *J. Chromatogr.* **1980**, *199*, 35-56.

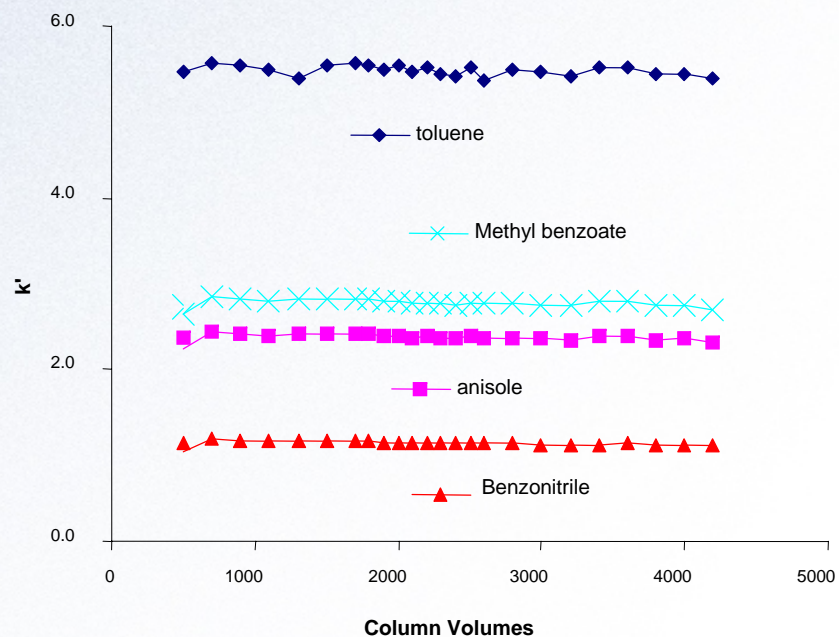


Chemical Stability

pH 1.0



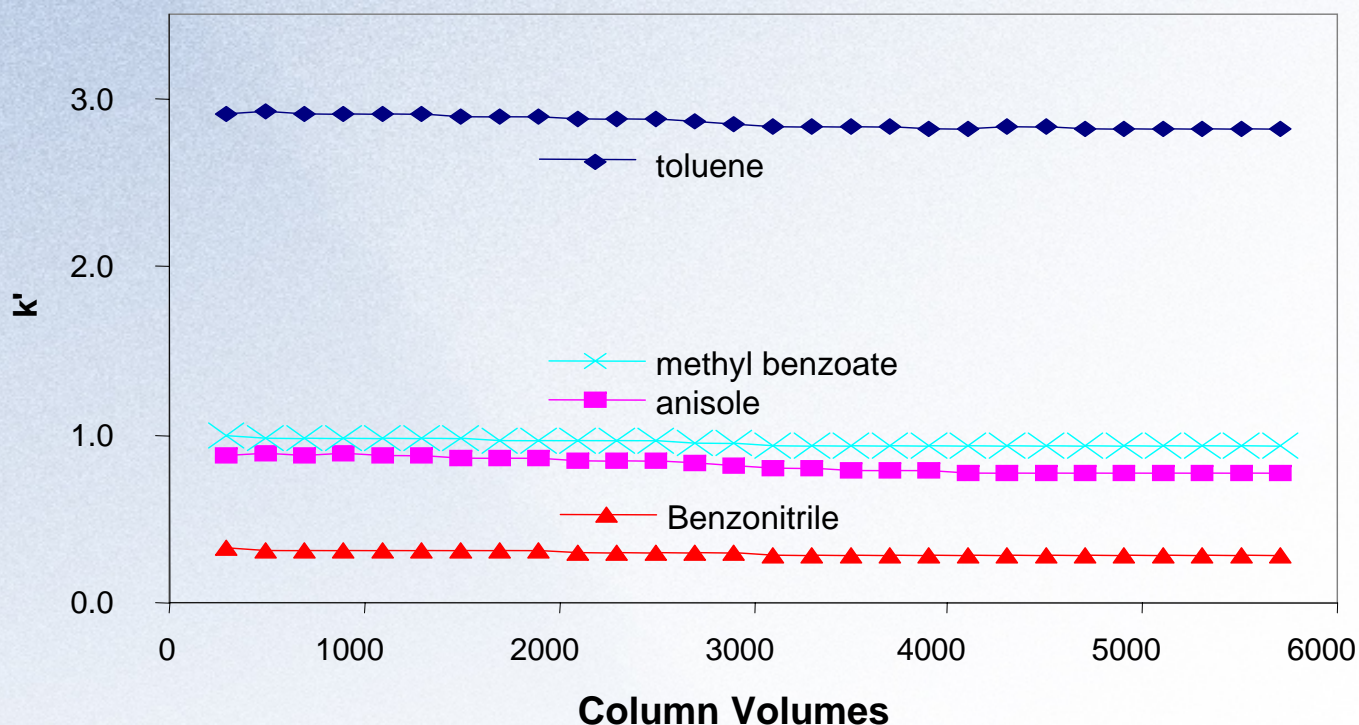
pH 12.0



Exposure and Evaluation Conditions: Mobile phase, 15/85 ACN/0.1M Nitric acid, pH 1.0, or 0.01M Tetramethylammoniumhydroxide, pH 12.0; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5 μ l; Detection at 254 nm; Column, 50 mm x 4.6 mm i.d., Sachtapore[®]-RP (part# TI01-0546).



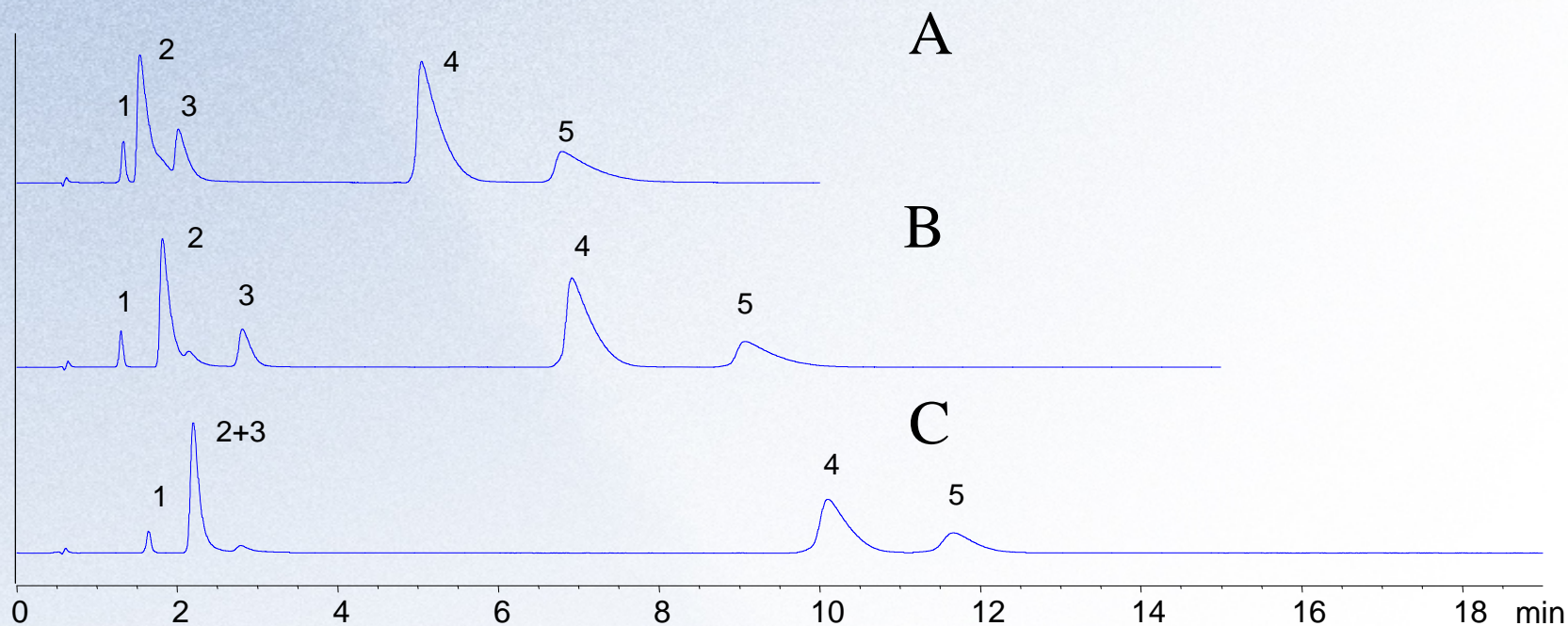
Temperature Stability at 100 °C



Exposure and Evaluation Conditions: Mobile phase, 15/85 ACN/water; Flow rate, 1.0 ml/min.; Temperature, 100 °C with Metalox heater; Injection volume, 5 ml; Detection at 254 nm; Column, 50 mm x 4.6 mm i.d., Sachtopore[®]-RP (part# TI01-0546).



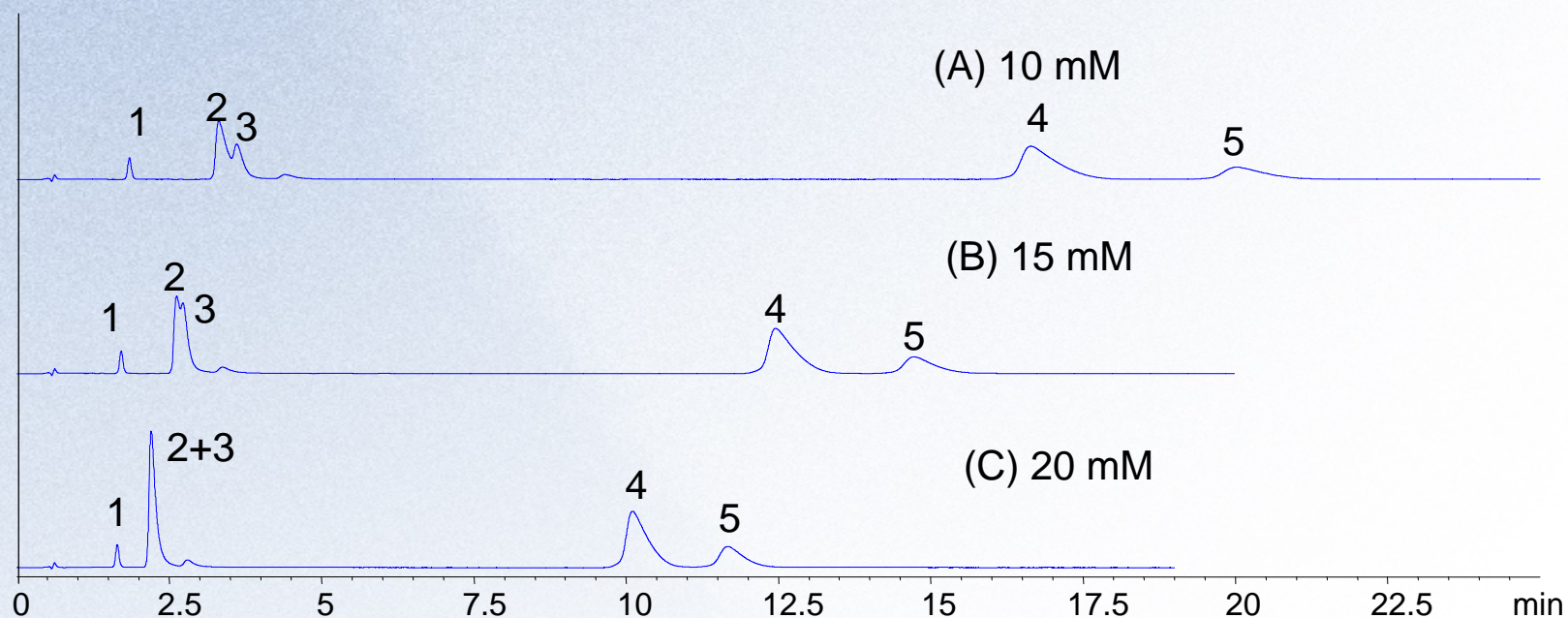
Effect of Lewis Base Additive on Separation of Basic Drugs



LC Conditions: Mobile phase: 30/70 ACN/20 mM buffer (pH=7). (A) ammonium acetate, (B) ammonium fluoride, (C) ammonium phosphate. flow rate: 1ml/min, temperature: 40 °C. Wavelength: 254 nm. Column, 50 mm x 4.6 mm i.d., Sachtapore®-RP (part# TI01-0546). Solutes: (1) lidocaine, (2) quinidine, (3) tryptamine, (4) amitriptyline, and (5) nortriptyline.



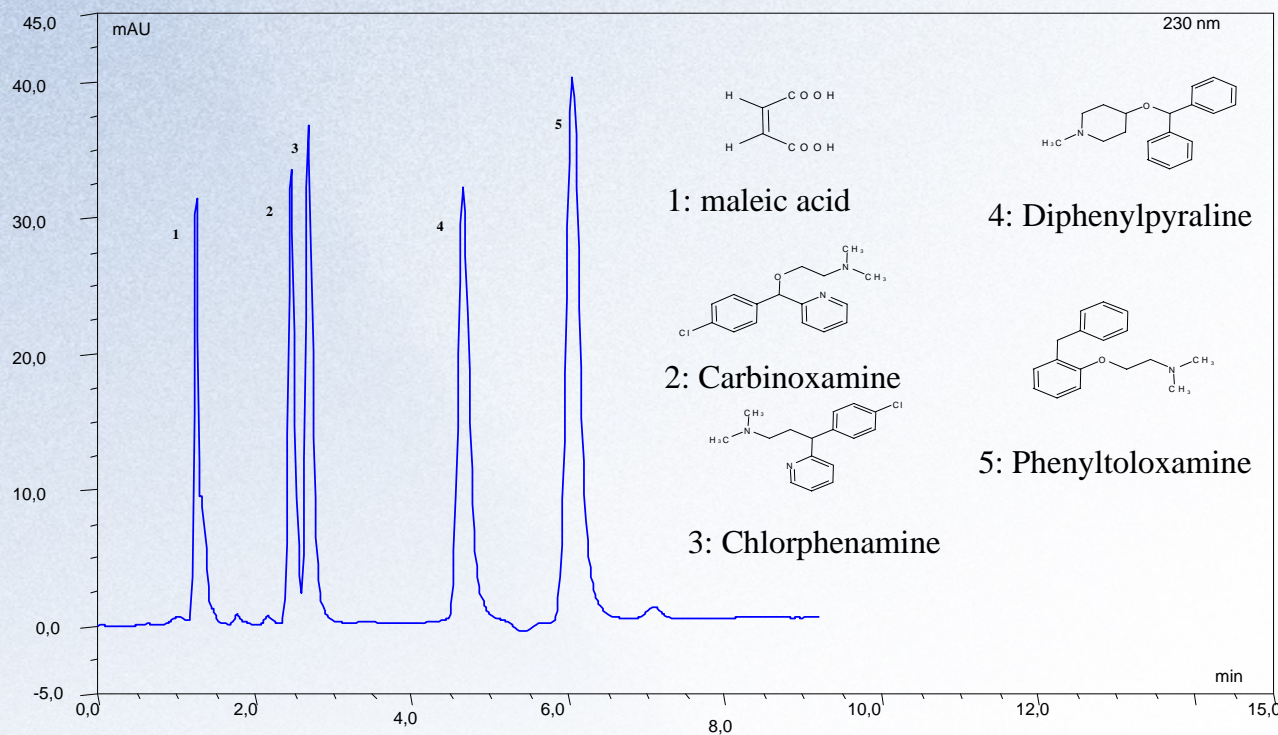
Effect of Ionic Strength on Separation of Basic Drugs



LC Conditions: Mobile phase: 30/70 ACN/phosphate buffer (pH=7). (A) 10 mM, (B) 15 mM, (C) 15 mM. flow rate: 1ml/min, temperature: 40 °C. Wavelength: 254 nm. Column, 50 mm x 4.6 mm i.d., Sachtapore[®]-RP (part# TI01-0546). Solutes: (1) lidocaine, (2) quinidine, (3) tryptamine, (4) amitriptyline, and (5) nortriptyline.

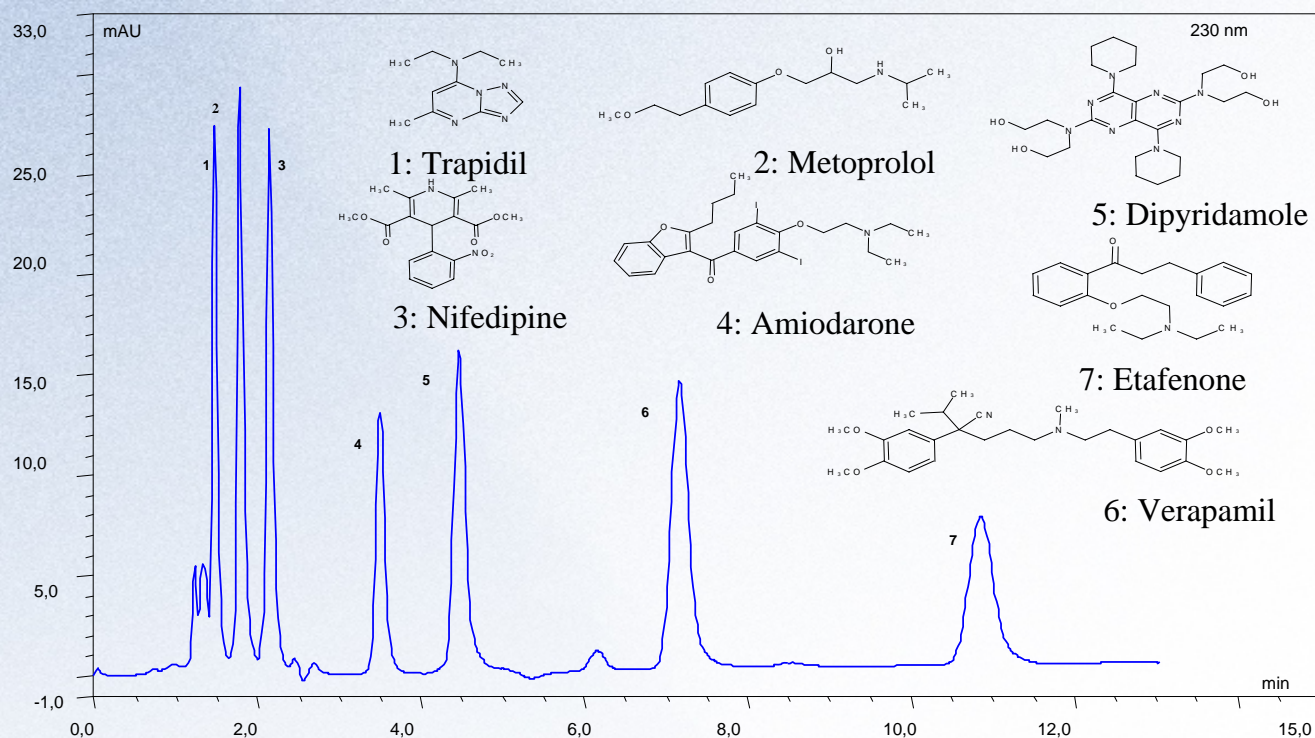


Antihistamines Separation at pH 10



LC Conditions: Mobile phase, 74% (50 mM H₃PO₄ + 5 mM KH₂PO₄), 26% ACN, pH 10; Flow rate, 1.0 ml/min.; Temperature, Ambient; Injection volume, 20 µl; Detection at 220 nm; Column, Sachtapore®-RP (300 Å, 3 µm, 150x4 mm).

Separation of Basic Cardiac Drugs at pH 10



LC Conditions: Mobile phase, 70% (10 mM Borax + 10 mM Soda), 30% ACN, pH 10; Flow rate, 1.0 ml/min.; Temperature, Ambient; Injection volume, 20 μ l; Detection at 230 nm; Column, Sachtapore[®]-RP (300 Å, 3 μ m, 150x4 mm).



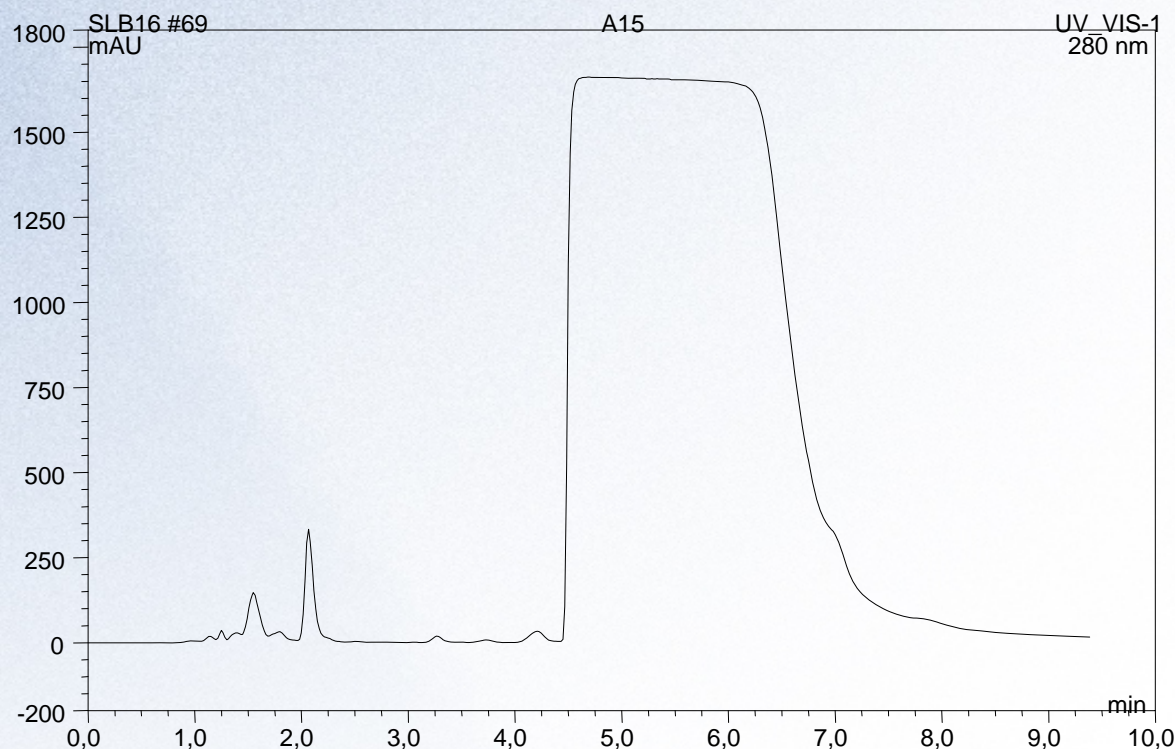
Semi-Prep Separation of Pentifylline (vasodilator)

Particle Sizes:

3,5,10,20,40,80
100 micron
1 mm

Pores sizes:

60, 100, 300,
500, 1000,
2000 Angstroms



LC Conditions: Mobile phase, (+ 10 mM $\text{Na}_2\text{B}_4\text{O}_7$ + 1 mM H_3BO_3), pH 8.8; Flow rate, 1.0 ml/min.; Temperature, Ambient; Injection volume, 20 μl ; Detection at 254 nm; Column, Sachtapore[®]-RP (300 Å, 3 μm , 150 x 4 mm).



Conclusions

- The Sachtopore[®]-RP shows *similar selectivity* to ZirChrom[®]-PBD (ODS-like for neutrals).
- The Sachtopore[®]-RP has *excellent stability* from pH 1-12 and up to 100°C.
- The type of Lewis base buffer has a profound effect on *selectivity* for Sachtopore[®]-RP.
- Basic (amine) analytes generally undergo *RP/CEX mixed-mode retention mechanism* on Sachtopore[®]-RP.



For More Information, Visit Us at Booth 220

A white rectangular banner containing the ZirChrom logo on the left, the text 'ZirChrom®' in the center, and a blue chromatogram on the right. The chromatogram shows a baseline with three distinct peaks of varying heights. To the right of the peaks, the text '1-866-STABLE-1' and 'www.zirchrom.com' is displayed. Below the chromatogram, the tagline '... For Peak Performance' is written in a smaller blue font.

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