Titania Based Reversed Stationary Phases for RP-HPLC and Preparative Liquid Chromatography

Pittcon 2007

DAN NOWLAN[1], BINGWEN YAN[1], CLAYTON V. MCNEFF[1], JOCHEN WINKLER[2]

[1]ZirChrom Separations, Inc. 617 Pierce St., Anoka, MN 55303,

[2]Sachtleben, Duisburg, Germany

Specialists in High Efficiency, Ultra-Stable Phases for HPLC

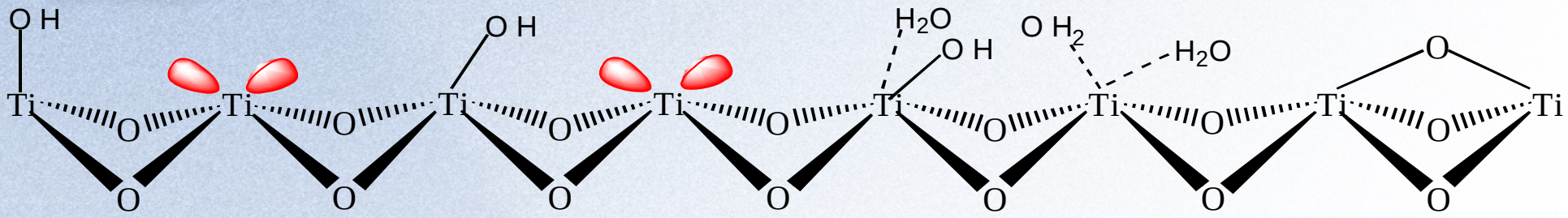
Outline

- Surface Chemistry of Titania
- 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
- Applications

Conclusion - The titania phases show similar selectivity to their zirconia counterparts, and have excellent stability from pH 1-12 and up to 100 ºC.

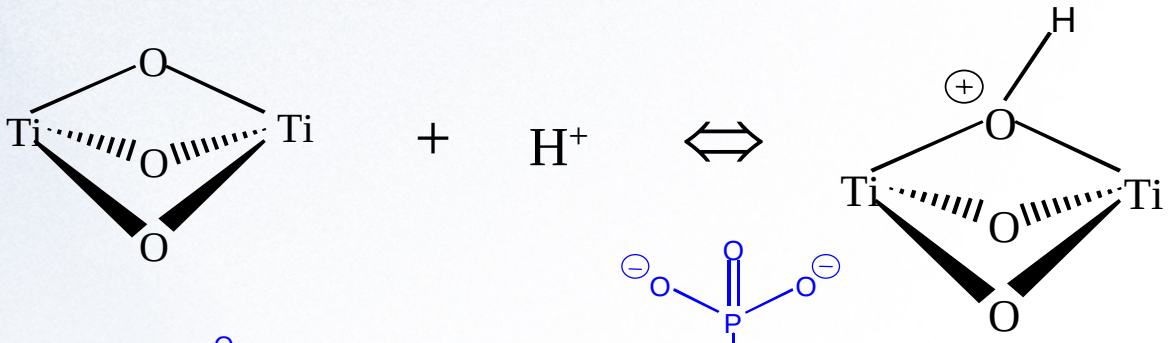
Surface Chemistry of Titania-Based Supports for HPLC

ZirChrom®

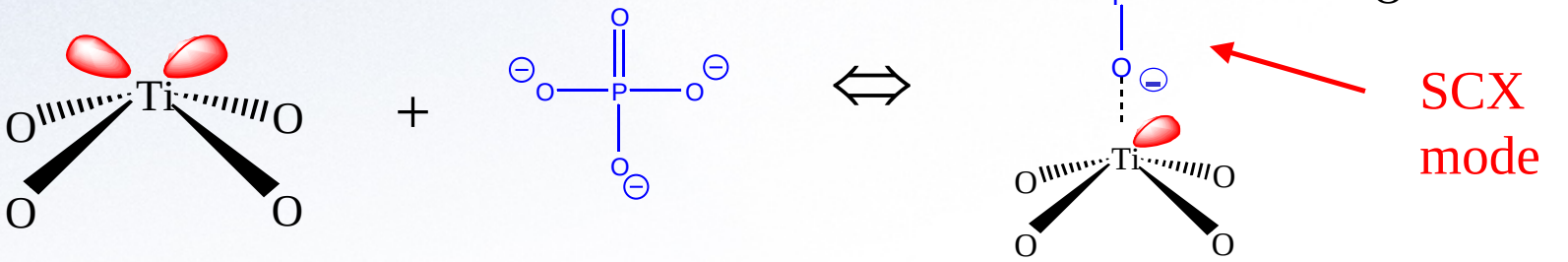


Weak Brönsted Acid: $TiOH + OH^- \Leftrightarrow TiO^- + H_2O$

Weak Brönsted Base:

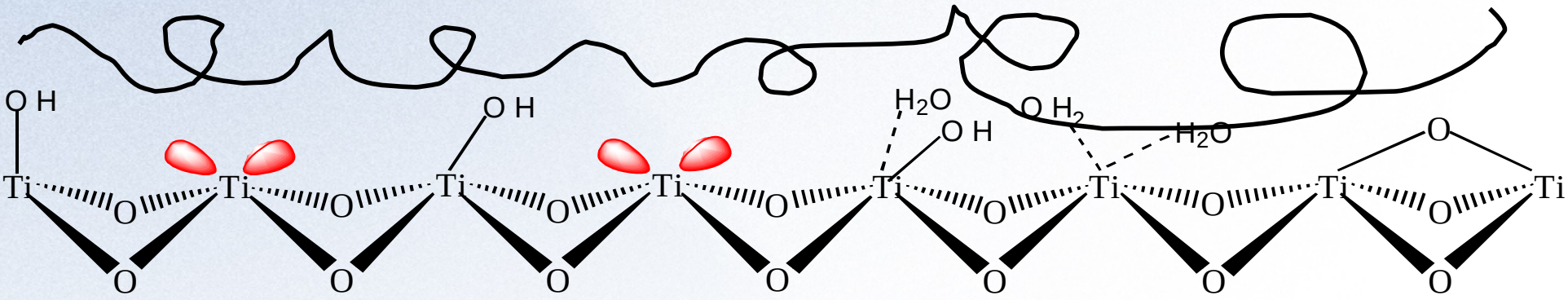


Strong Lewis Acid:



Polyethylene Coated Titania

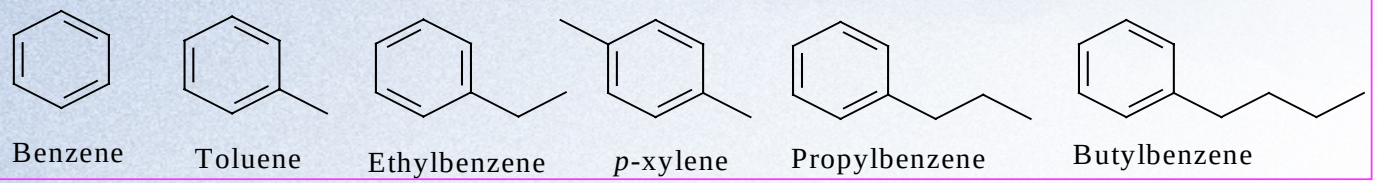
$$-\left[\ CH_2\text{-}CH_2\ \right]_n-$$ Polyethylene



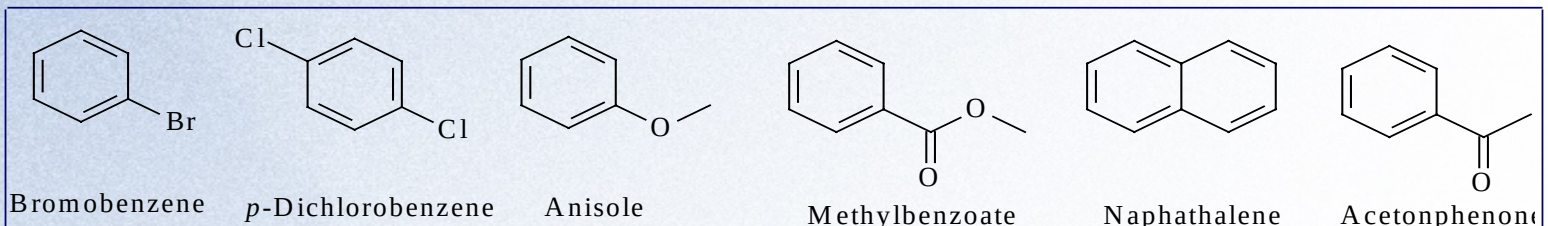
22 Non-electrolyte Solutes

ZirChrom®

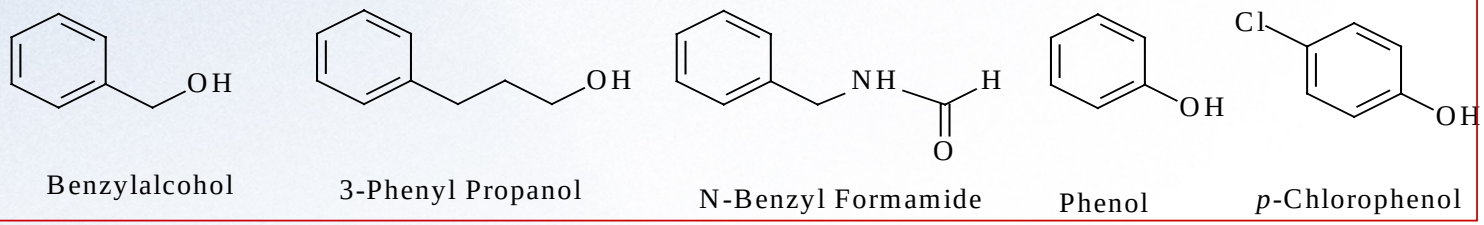
Nonpolar



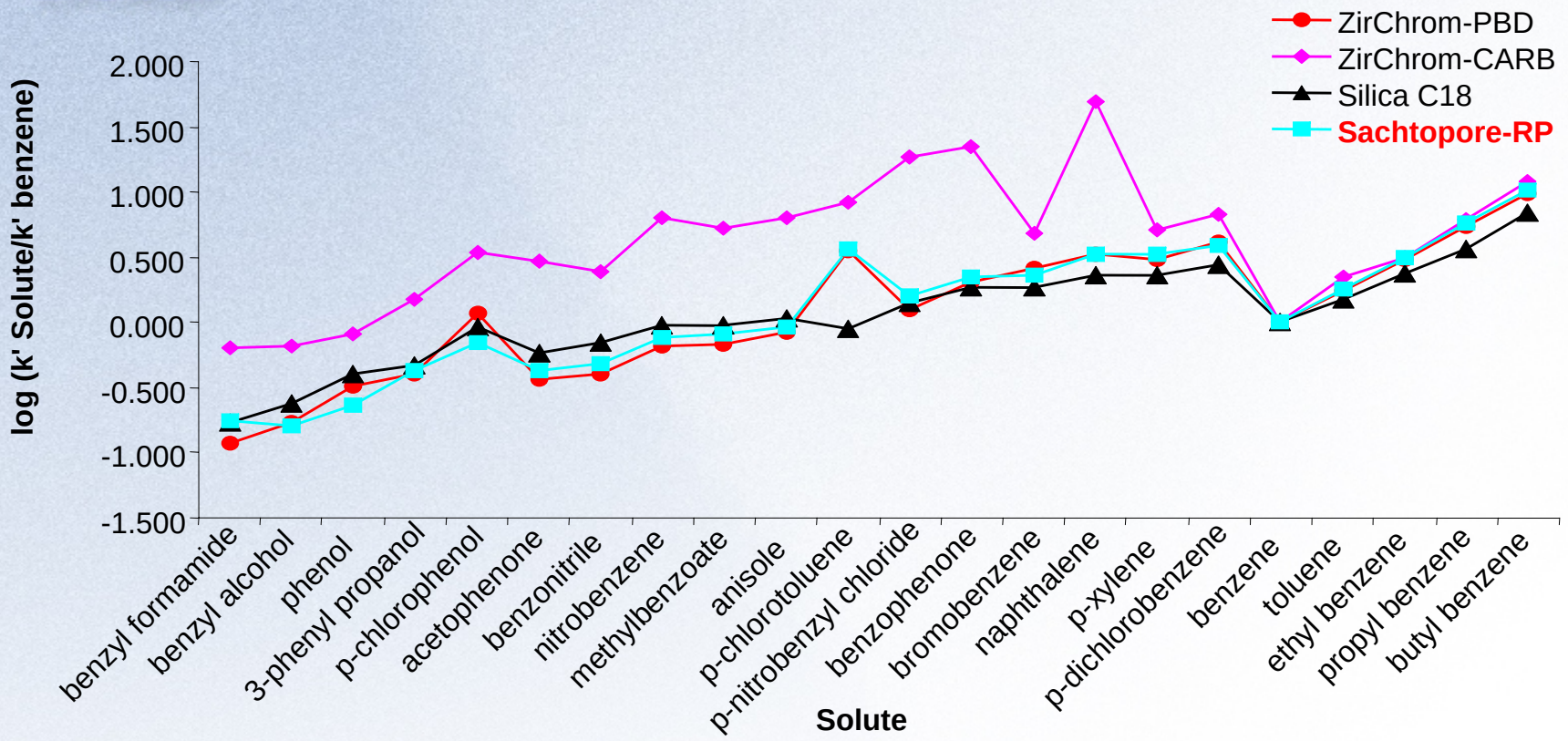
Polar



HB Donor

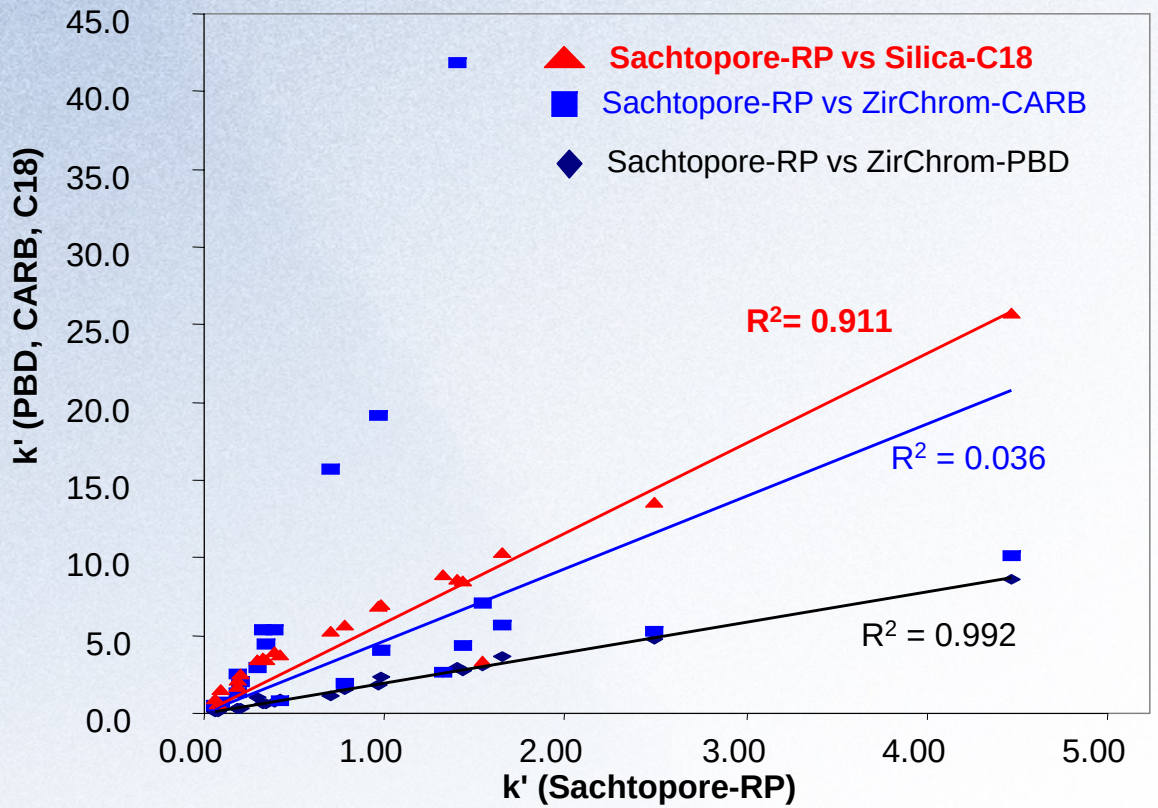


Selectivity Comparison



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

κ-κ Comparison

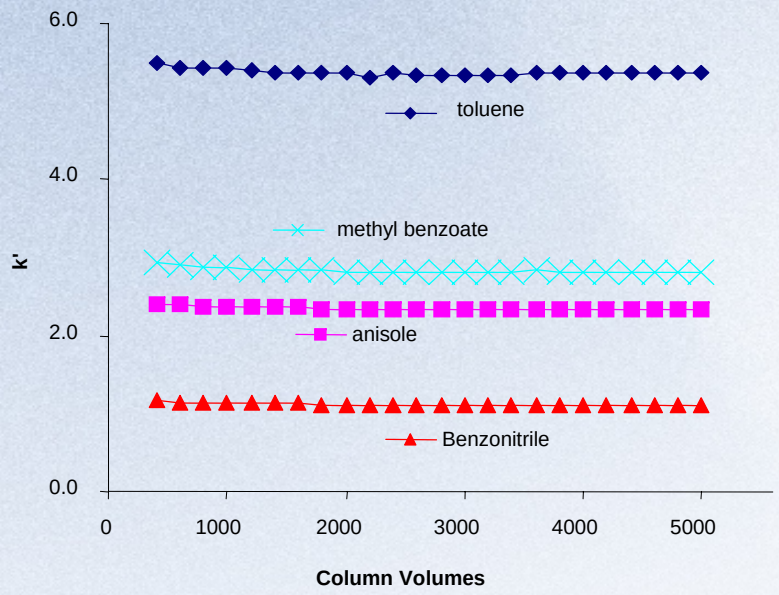


LC Conditions: Mobile phase, 40/60 ACN/Water; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5ul; Detection at 254nm.

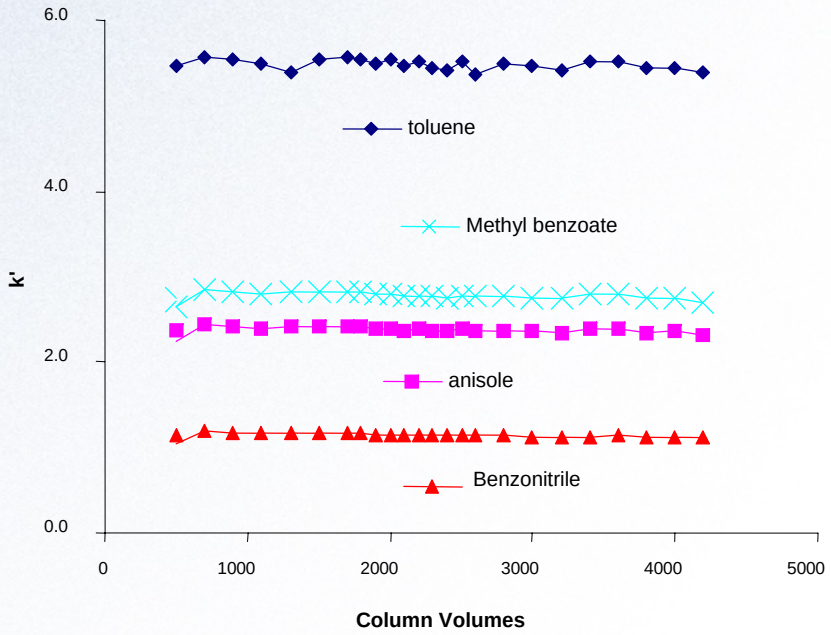
Reference: Melander, W.; Stoveken, J.; Horvath, C. *J. Chromatogr.* **1980**, *199*, 35-56.

Chemical Stability

pH 1.0

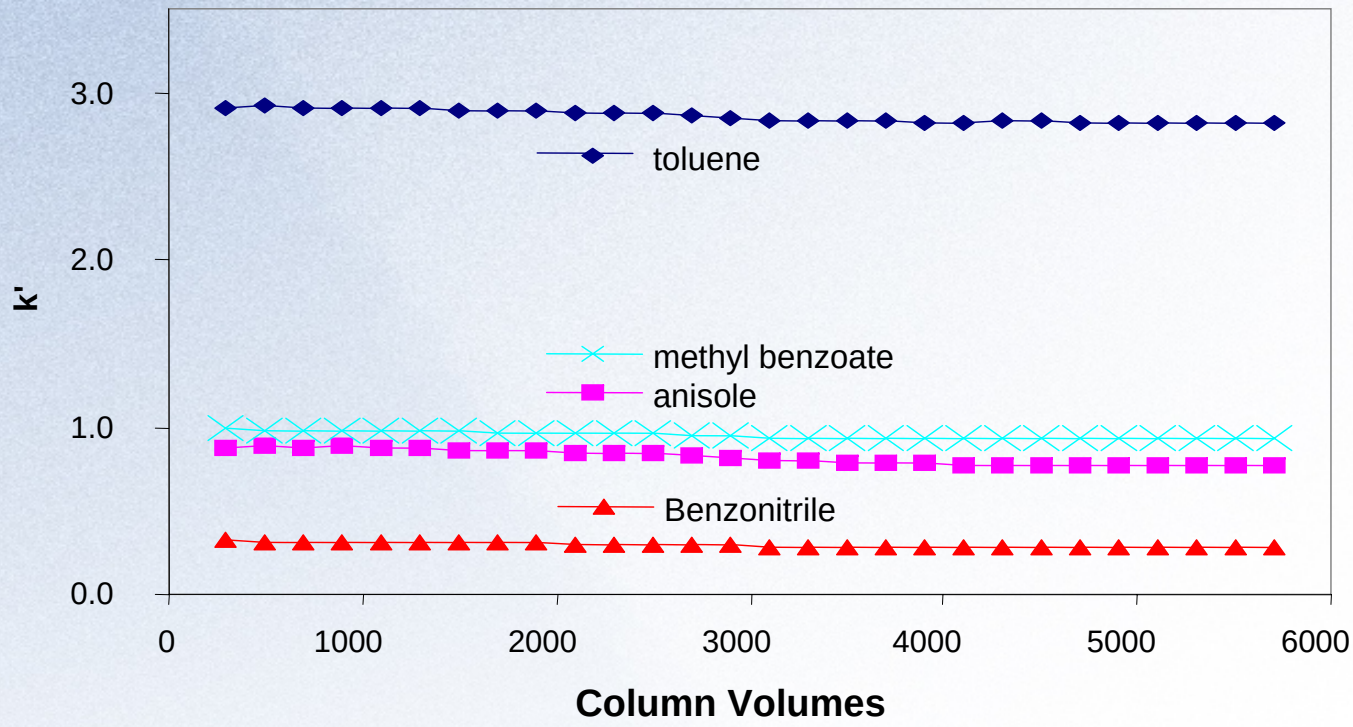


pH 12.0



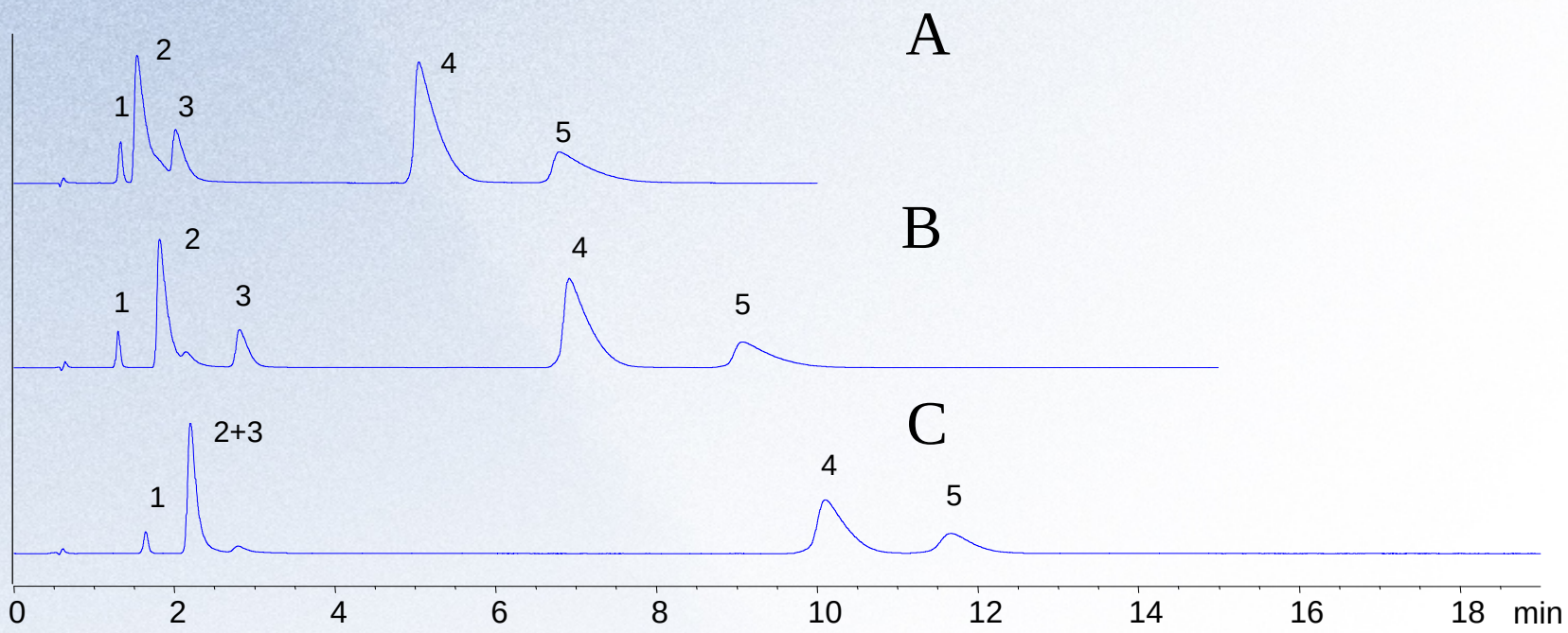
Exposure and Evaluation Conditions: Mobile phase, 15/85 ACN/0.1M Nitric acid, pH 1.0, or 0.01M Tetramethylammoniumhydroxide, pH 12.0; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5 µl; Detection at 254 nm; Column, 50 mm x 4.6 mm i.d., Sachtopore-RP (part# TI01-0546).

Temperature Stability at 100 °C



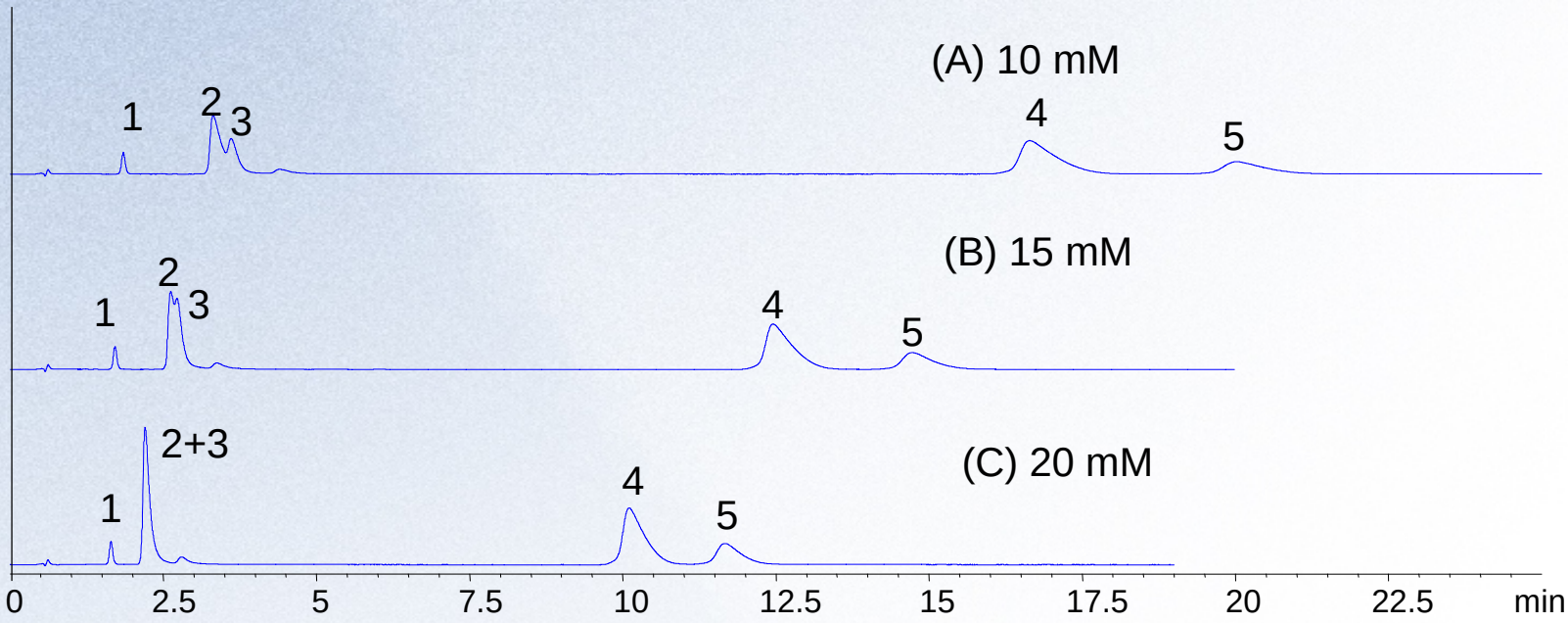
Exposure and Evaluation Conditions: Mobile phase, 15/85 ACN/water; Flow rate, 1.0 ml/min.; Temperature, 100 °C with Metalox heater; Injection volume, 5 ml; Detection at 254 nm; Column, 50 mm x 4.6 mm i.d., Sachtopore-RP (part# TI01-0546).

Effect of Lewis Base Additive on Separation of Basic Drugs



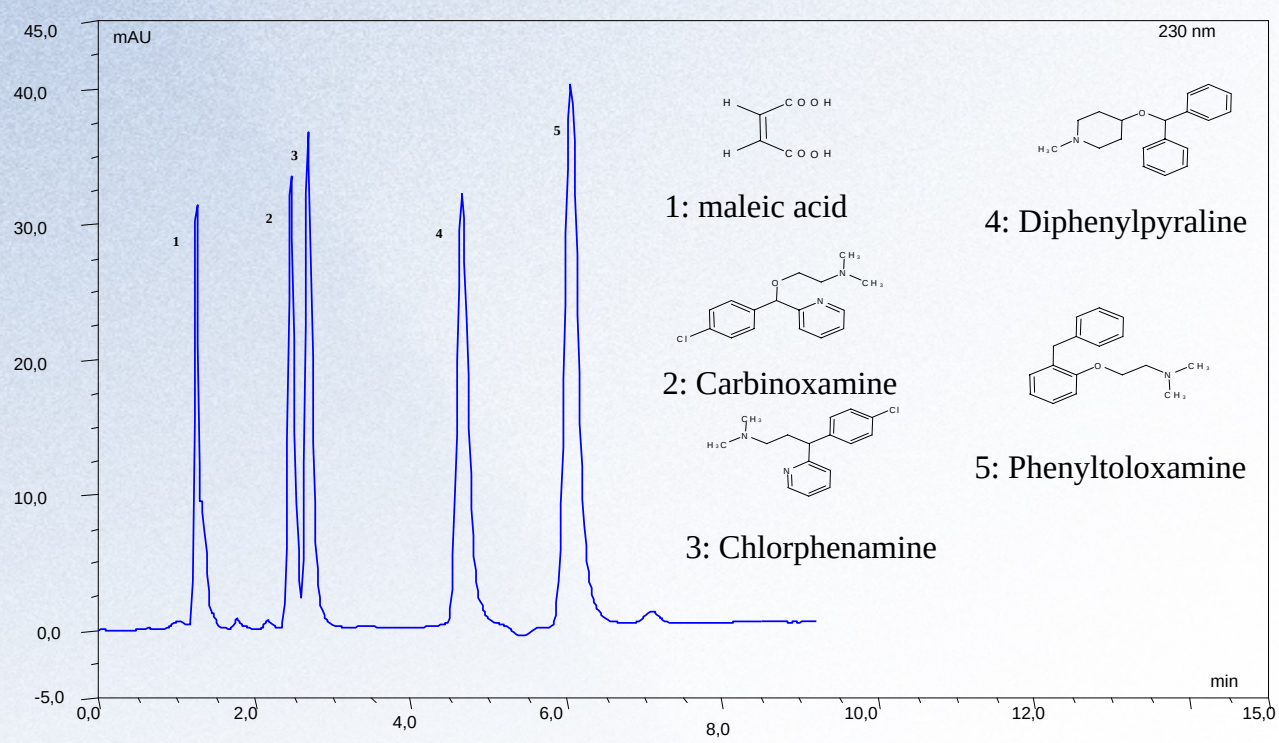
LC Conditions: Mobile phase: 30/70 ACN/20 mM buffer (pH=7). (A) ammonium acetate, (B) ammonium fluoride, (C) ammonium phosphate. flow rate: 1ml/min, temperature: 40 °C. Wavelength: 254 nm. Column, 50 mm x 4.6 mm i.d., Sachtopore-RP (part# TI01-0546). Solutes: (1) lidocaine, (2) quinidine, (3) tryptamine,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

Antihistimines Separation at pH 10

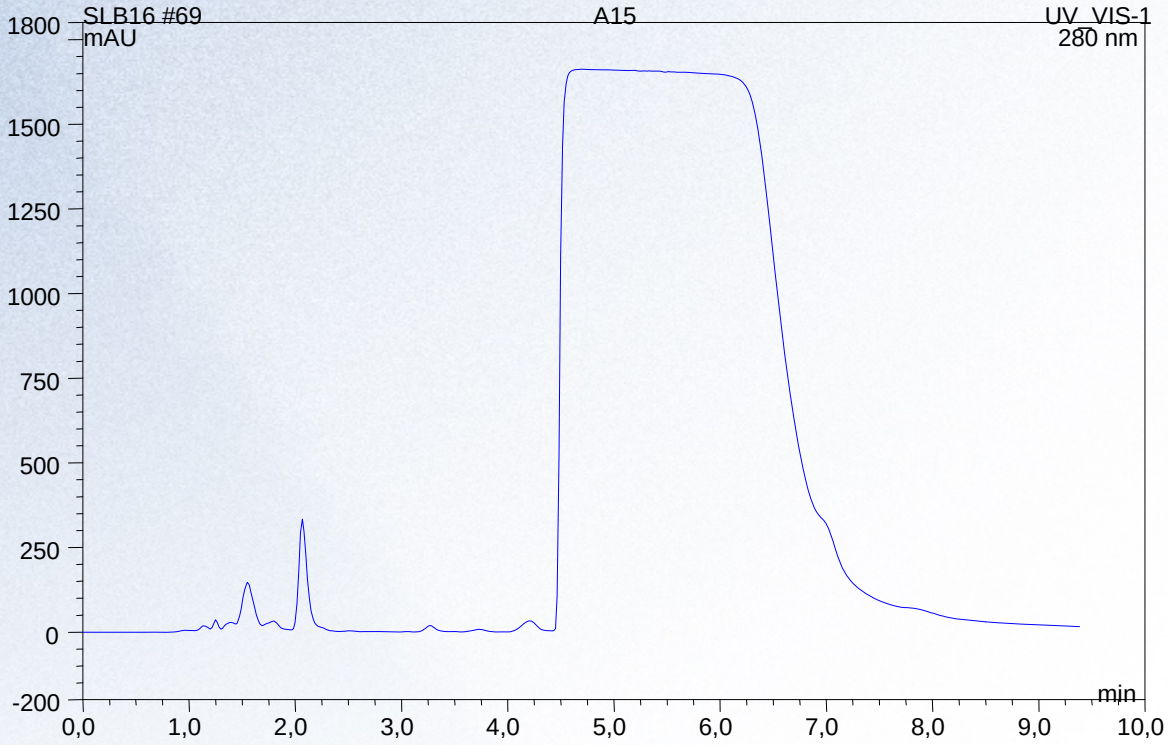


LC Conditions: Mobile phase, 74% (50 mM H_3PO_4 + 5 mM KH_2PO_4), 26% ACN, pH 10; Flow rate, 1.0 ml/min.; Temperature, Ambient; Injection volume, 20 µl; Detection at 220 nm; Column, Sachtopore-RP (300 Å, 3 µm, 150 x 4 mm).

Semi-Prep Separation of Pentifylline (vasodilator)

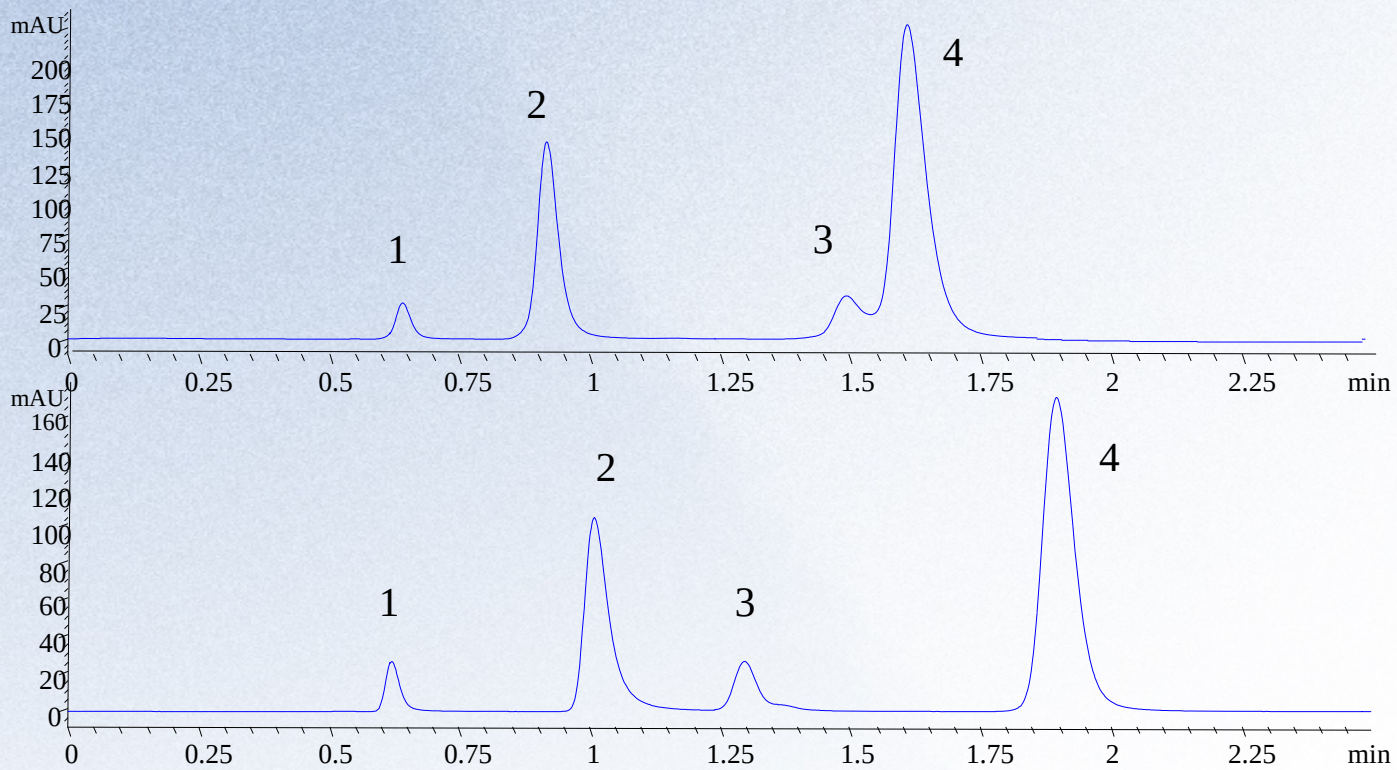
Particle Sizes:
3,5,10,20,40,80
100 micron
1 mm

Pores sizes:
60, 100, 300,
500, 1000,
2000 Angstroms



LC Conditions: Mobile phase, (+ 10 mM $Na_2B_4O_7$ + 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, Sachtopore®-RP (300 Å, 3 µm, 150 x 4 mm).

Comparison Between Sachtopore-CARB and ZirChrom-CARB



Sachtopore-CARB has somewhat similar selectivity compared to ZirChrom-CARB.

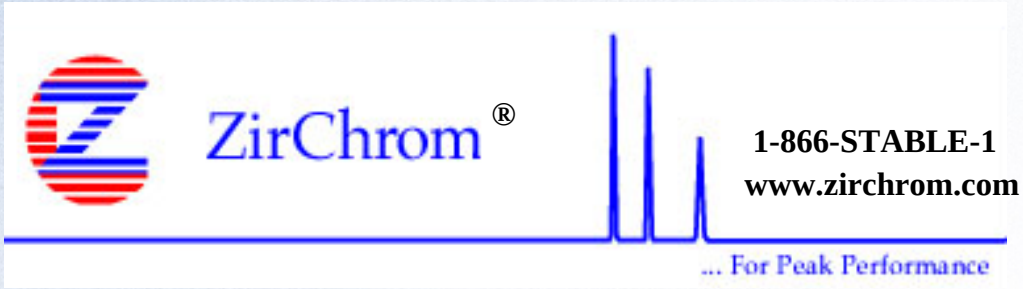
LC Conditions: Mobile phase, 50/50 ACN/water; Flow rate, 1.0 ml/min.; Temperature, Ambient; Injection volume, 5 µl; Detection at 254 nm; Column: 50 x 4.6 mm, 5 um.
Solutes: (1) acetone, (2) p-cresol, (3) ethylbenzene, (4) nitrobenzene

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^{o}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.
- Sachtopore-CARB has *somewhat similar* selectivity compared to ZirChrom-CARB.

ZirChrom®

For More Information, Visit Us at Booth 4623



For more information and web access to the free **Buffer Wizard:** www.zirchrom.com