Polymer and Carbon Coated Porous Titania for Reversed Phase and Preparative Liquid Chromatography

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Specialists in High Efficiency, \textit{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 °C.
Surface Chemistry of Titania-Based Supports for HPLC

Weak Brönsted Acid: \( \text{TiOH} + \text{OH}^- \leftrightarrow \text{TiO}^- + \text{H}_2\text{O} \)

Weak Brönsted Base:

Strong Lewis Acid:

SCX mode
Polyethylene Coated Titania

\[
\left[ \text{CH}_2-\text{CH}_2 \right]_n \quad \text{Polyethylene}
\]
## 22 Non-electrolyte Solutes

### Nonpolar
- Benzene
- Toluene
- Ethylbenzene
- $p$-xylene
- Propylbenzene
- Butylbenzene

### Polar
- Bromobenzene
- $p$-Dichlorobenzene
- Anisole
- Methylbenzoate
- Naphthalene
- Acetonphenone

### HB Donor
- Benzonitrile
- Nitrobenzene
- $p$-Nitrotoluene
- $p$-Nitrobenzyl Chloride
- Benzophenone

### HB Donor Substances
- Benzylalcohol
- 3-Phenyl Propanol
- N-Benzyl Formamide
- Phenol
- $p$-Chlorophenol
**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.

<table>
<thead>
<tr>
<th>Solute</th>
<th>ZirChrom-PBD</th>
<th>ZirChrom-CARB</th>
<th>Silica C18</th>
<th>Sachtopore-RP</th>
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<tbody>
<tr>
<td>benzyl formamide</td>
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<td>benzy alcohol</td>
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<td>3-phenyl propanol</td>
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<tr>
<td>butyl benzene</td>
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Log (k' Solute/k' benzene)
**LC Conditions:** Mobile phase, 40/60 ACN/Water; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5ul; Detection at 254nm.

Chemical Stability

**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.
LC Conditions: Mobile phase, 74% (50 mM H$_3$PO$_4$ + 5 mM KH$_2$PO$_4$), 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 Na₂B₄O₇ + 1 mM H₃BO₃), 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 μm.

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°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.
For More Information, Visit Us at Booth 220

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