Polymer Coated Titania for Analytical and Preparative Reversed-Phase Chromatography

EAS 2005

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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: \[
\text{TiOH} + \text{OH}^- \rightleftharpoons \text{TiO}^- + \text{H}_2\text{O}
\]

Weak Brönsted Base: \[
\text{Ti} + \text{H}^+ \rightleftharpoons \text{H}_2\text{O}
\]

Strong Lewis Acid: \[
\text{Ti} + \Theta\text{OPO}_3^\Theta\rightleftharpoons \Theta\text{POPO}_3^\Theta
\]

SCX mode
Polyethylene Coated Titania

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

<table>
<thead>
<tr>
<th>Nonpolar</th>
<th>Polar</th>
<th>HB Donor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene</td>
<td>Bromobenzene</td>
<td>Benzylalcohol</td>
</tr>
<tr>
<td>Toluene</td>
<td>p-Dichlorobenzene</td>
<td>3-Phenyl Propanol</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>Anisole</td>
<td>N-Benzyl Formamide</td>
</tr>
<tr>
<td>p-xylene</td>
<td>Methylbenzoate</td>
<td>Phenol</td>
</tr>
<tr>
<td>Propylbenzene</td>
<td>Naphthalene</td>
<td>p-Chlorophenol</td>
</tr>
<tr>
<td>Butylbenzene</td>
<td>Acetonphenone</td>
<td></td>
</tr>
</tbody>
</table>

- **Nonpolar**
  - Benzene
  - Toluene
  - Ethylbenzene
  - p-xylene
  - Propylbenzene
  - Butylbenzene

- **Polar**
  - Bromobenzene
  - p-Dichlorobenzene
  - Anisole
  - Methylbenzoate
  - Naphthalene
  - Acetonphenone

- **HB Donor**
  - 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.
**κ-κ Comparison**

**LC Conditions:** Mobile phase, 40/60 ACN/Water; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5μl; 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# T101-0546).
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 ºC. Wavelength: 254 nm. Column, 50 mm x 4.6 mm i.d., Sachtropore®-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 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, Sachtopore®-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 μl; Detection at 230 nm; Column, Sachtopore®-RP (300 Å, 3 μm, 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 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).
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

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