Packing Materials for Column Chromatography

Open column chromatography is an excellent and easy technique for large-scale preparation and purification at low cost. COSMOSIL offers both normal and reversed phase packing materials based on totally porous spherical silica, which provides higher separation, less pressure and higher reproducibility than irregular silica.

### Material Characteristics

<table>
<thead>
<tr>
<th>Packing Material</th>
<th>C_{18}-OPN</th>
<th>C_{18}-PREP</th>
<th>SL-II-PREP</th>
<th>Silica Gel 60 (neutral)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica Gel</td>
<td>High Purity Porous Spherical Silica</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Particle Size</td>
<td>75, 140 µm</td>
<td>40, 75, 140 µm</td>
<td>75, 140 µm</td>
<td></td>
</tr>
<tr>
<td>Average Pore Size</td>
<td>approx. 120 Å</td>
<td>approx. 60 Å</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specific Surface Area</td>
<td>approx. 300 m²/g</td>
<td>approx. 500 m²/g</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stationary phase</td>
<td>Octadecyl Group</td>
<td>None</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbon content</td>
<td>-</td>
<td>approx. 19%</td>
<td>approx. 0%</td>
<td></td>
</tr>
<tr>
<td>End-capping treatment</td>
<td>None</td>
<td>Treated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Useful range</td>
<td>Open column chromatography / Flash column chromatography</td>
<td>Reversed phase chromatography</td>
<td>Normal phase chromatography</td>
<td></td>
</tr>
</tbody>
</table>

### Selection Guide

#### Reversed Phase

- **Open Column Chromatography**
  - Concentration of solvent: High (70% or more)
  - Suitable packing materials: 75C18-PREP, 140C18-PREP

- **Flash Column Chromatography**
  - Concentration of solvent: Low (70% or less)
  - Suitable packing materials: 75C18-OPN, 140C18-OPN

- **Medium Pressure Column Chromatography**
  - Suitable packing materials: 40C18-PREP

#### Normal Phase

- **Acid resistance of compounds**
  - Not decompose in low acid
  - Suitable packing materials: SL-II-PREP
  - Decomposed in low acid
  - Suitable packing materials: Slica Gel 60 (neutral)
  - Unknown

### COSMOSIL C_{18}-OPN

- A new “Water-Wet” C_{18} packing material for reversed phase open column chromatography
- Enables large-scale preparation and purification at low cost

### Characteristic

- The external surface of the C18-OPN gel is coated with hydrophilic groups.
- Conventional reversed phase C_{18} packing materials are restricted to about 30-50% water in the mobile phase. The COSMOSIL C_{18}-OPN is a new “Water-Wet” C_{18} packing material developed for reversed phase open column chromatography. The C_{18}-OPN material can be used in 100% aqueous eluents.

- Packing materials in water
  - Left: C18 OPN provides good resolution
  - Right: Conventional product float up
Application Data

Separation of hydrophilic compounds in aqueous solution

In reversed phase chromatography, hydrophilic compounds such as Theobromine and Theophylline could be separated under low concentration of organic solvent. The figure shows they are clearly separated by reversed open column chromatography with 70% of water.

Separation of \( p \)-Cresol and \( p \)-Ethylphenol by normal and reversed phase mode

Since the structural difference between \( p \)-Cresol and \( p \)-Ethylphenol is only one methyene group, it is difficult to separate such samples under normal phase condition. On the other hand, the samples are clearly separated under reversed phase condition with COSMOSIL C18®OPN packing material.

COSMOSIL Application Data

Column: 75C18-OPN
Column size: 20mm.I.D.-250mm
Mobile phase: Methanol/H2O = 30/70
Flow rate: 0.2 ml/min
Temperature: Room temperature
Detection: UV254nm
Sample: 1; Theobromine (100mg)
2; Theophylline (100mg)

COSMOSIL C18®PREP

· Standard reversed phase packing material for open chromatography
· Enables large-scale preparation and purification at low cost

COSMOSIL Application Data

Column: 40C18-PREP
Column size: 20mm.I.D.-300mm(Closed column)
Mobile phase: Methanol
Flow rate: 9.9 ml/min
Temperature: Room temperature
Detection: UV254nm
Sample: 1; DL-\( \alpha \)-Tocopherol (5mg)
2; DL-\( \alpha \)-Tocopherol Acetate (5mg)

COSMOSIL Application Data

Column: 40C18-PREP
Column size: 20mm.I.D.-300mm(Closed column)
Mobile phase: Methanol/0.05%TFA-H2O = 70/30
Flow rate: 9.9 ml/min
Temperature: Room temperature
Detection: UV254nm
Sample: 1; Baicalin (40μg)
2; B thecalon (120μg)
3; Wogonin (40μg)

Application Data

Vitamin E

Natural Compounds
**Basic Compounds**

**COSMOSIL Application Data**

- **Column:** 40C$_{18}$-PREP
- **Column size:** 20mm I.D.-300mm (Closed column)
- **Mobile phase:** MeOH/H$_2$O = 80/20
- **Flow rate:** 9.9 ml/min
- **Temperature:** Room temperature
- **Detection:** UV254nm
- **Sample:**
  1. Quinoline (200 µg)
  2. Naphthalene (200 µg)
  3. N,N-Diethylaniline (100 µg)

**Highly Polar Compounds**

**COSMOSIL Application Data**

- **Column:** 40C$_{18}$-PREP
- **Column size:** 20mm I.D.-300mm (Closed column)
- **Mobile phase:** MeOH/H$_2$O = 30/70
- **Flow rate:** 9.9 ml/min
- **Temperature:** Room temperature
- **Detection:** UV254nm
- **Sample:**
  1. Theobromine (250 µg)
  2. Theophylline (250 µg)
  3. Caffeine (250 µg)

**Reproducibility and washing methods**

Wash the COSMOSIL C$_{18}$-OPN packing material with tetrahydrofuran, chloroform or other solvents to remove the impurities. This packing material has excellent reproducibility and can be used repeatedly.

*CAUTION*

Do not wash with basic solvents of pH 7 or more which will dissolve the silica gel or pH 2 or less which will cleave the C$_{18}$ stationary phase. Dry the packing material at 50°C or less. See end of this chapter for packing method.

---

**COSMOSIL SL-II-PREP**

- **Standard packing materials for normal phase chromatography**
- **Ultra pure silica gel packing material more than 99.99% purity**

**Comparison**

Highly purified silica gel of COSMOSIL SL-II-PREP enables separation of metal coordination compounds without adsorption.

**Metal Coordination Compounds**

**COSMOSIL Application Data**

- **Column:** 10mm I.D.-250mm
- **Mobile phase:** Hexane/Ethanol = 95/5
- **Flow rate:** 5.0 ml/min
- **Temperature:** 30°C
- **Detection:** UV254nm
- **Sample:**
  1. Quinizarin
  2. p-Nitrobenzyl Alcohol

**Organic Acid and Amide**

**COSMOSIL Application Data**

- **Column:** 10mm I.D.-250mm
- **Mobile phase:** Hexane/Ethanol = 90/10
- **Flow rate:** 5.0 ml/min
- **Temperature:** 30°C
- **Detection:** UV254nm
- **Sample:**
  1. Salicylic Acid
  2. Salicylamide
Silica Gel 60 (spherical • neutral)

- The pH of Silica Gel is adjusted to neutral
- Suitable for the separation of pH sensitive compounds

Comparison with conventional silica gel

Since conventional silica gels are weakly acidic, some pH sensitive compounds may be decomposed during the purification by column chromatography with the acidic silica gels. The pH of Silica Gel 60 (spherical • neutral) is adjusted to nearly neutral for the separation of not only pH sensitive compounds but also new compounds that the physical properties are still unknown.

Purification of Acetal

**COSMOSIL Application Data**

<table>
<thead>
<tr>
<th>Column:</th>
<th>Column size:</th>
<th>Mobile phase:</th>
<th>Flow rate:</th>
<th>Temperature:</th>
<th>Detection:</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.6mm I.D. x 250mm</td>
<td>1.0 ml/min</td>
<td>Hexane/Ethyl Acetate = 99/1</td>
<td>30°C</td>
<td>UV254nm</td>
<td></td>
</tr>
</tbody>
</table>

Sample: 1; Methyl Benzoate (Standard) (10mg/ml)
Sample A (100mg/ml)

Inj.Vol: 3μl

**COSMOSIL C₁₈-OPN**

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Particle Size</th>
<th>PKG Size</th>
<th>Product Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>COSMOSIL 75C₁₈-OPN</td>
<td>75 μm</td>
<td>100 g</td>
<td>37842-66</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500 g</td>
<td>37842-95</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>37842-11</td>
</tr>
<tr>
<td>COSMOSIL 140C₁₈-OPN</td>
<td>140 μm</td>
<td>100 g</td>
<td>37878-16</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500 g</td>
<td>37878-45</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>37878-61</td>
</tr>
</tbody>
</table>

**COSMOSIL C₁₈-PREP**

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Particle Size</th>
<th>PKG Size</th>
<th>Product Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>COSMOSIL 40C₁₈-PREP</td>
<td>40 μm</td>
<td>100 g</td>
<td>37932-86</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500 g</td>
<td>37932-15</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>37932-31</td>
</tr>
<tr>
<td>COSMOSIL 75C₁₈-PREP</td>
<td>75 μm</td>
<td>100 g</td>
<td>37933-76</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500 g</td>
<td>37933-05</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>12061-11</td>
</tr>
<tr>
<td>COSMOSIL 140C₁₈-PREP</td>
<td>140 μm</td>
<td>100 g</td>
<td>37934-66</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500 g</td>
<td>37934-95</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>37934-11</td>
</tr>
</tbody>
</table>

**COSMOSIL SL-II PREP**

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Particle Size</th>
<th>PKG Size</th>
<th>Product Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>COSMOSIL 75SL-II-PREP</td>
<td>75 μm</td>
<td>100 g</td>
<td>38012-64</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500 g</td>
<td>38012-35</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>38012-51</td>
</tr>
<tr>
<td>COSMOSIL 140SL-II-PREP</td>
<td>140 μm</td>
<td>100 g</td>
<td>38013-54</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>38013-41</td>
</tr>
</tbody>
</table>

**Silica Gel (spherical • neutral)**

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Particle Size</th>
<th>PKG Size</th>
<th>Product Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica Gel 60 (spherical • neutral) for column chromatograph</td>
<td>75 μm</td>
<td>500 g</td>
<td>30511-35</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>30511-51</td>
</tr>
<tr>
<td></td>
<td>140 μm</td>
<td>500 g</td>
<td>30518-65</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 kg</td>
<td>30518-81</td>
</tr>
</tbody>
</table>

For research use only, not intended for diagnostic or drug use.