Titanium Dioxide - Properties and Applications in LC

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Abstract

For decades science and industry have been searching for sorbents useful in liquid chromatography with the following

- Excellent stability at pH values between pH 1 and pH 14
- Symmetric and narrow peaks for basic compounds along with
- High selectivity
- · High mechanical stability and
- Low column back pressures

This sorbent is now commercially available under the trade name Sachtopore.

Chemically, Sachtopore is is a porous, ceramic titanium dioxide in the anatase or rutile modification. Titanium dioxide has acid/base properties intermediate between silicas and aluminium oxides. Unlike these, however, it is practically insoluble in bases as well as acids. It exhibits very selective adsorption/desorption behaviour for organic molecules, especially for bases and isomers.

The poster presents characteristic examples for separations that are impossible to perform with inorganic stationary phases known so far. Furthermore, examples for other benefits – such as its durability in cleaning in place (CIP) procedures - are shown.

Introduction

By choosing suitable stationary phases and separation conditions the performance and productivity of a large scale separation process can be improved significantly. Porous titanium dioxide – namely SachtoporeTM - is different in selectivity compared to Silica and Alumina which are the most commonly used oxide packings for NPchromatography. This difference offers a high innovative potential in order to gain more selective separations and enhance productivity.

■ General Features of Sachtopore

- Spherical particle shape (Fig. 1)
- 100 % crystalline
- Interconnected crystals

Physical Properties of Sachtopore

Sachtopore	60 Å	100 Å	300 Å	2000 Å
Pore diameter [Å]	60	100	300	2000
Specific surface area [m²/g]	100	55	15	<5
Specific pore volume [ml/g]	0.2	0.21	0.12	n. d .
Particle size available	3, 5, 10, 20, 40, 80 μm, 1 – 2 mm			
Mechanical stability				
Applicable packing pressure [bar]	600	600	6 00	600
Applicable piston pressure in DAC™ columns [bar]	100	100	100	80

Chemical Properties of Sachtopore

- Pharmaceutical grade purity
- · Homogeneous surface chemistry • pH-stability 1-14, stable to CIP in 1 M NaOH
- 2 4 6 8 10 12 14
- · Moderate Lewis acidity
- Strong adsorption of multidendate ligands which bind specifically to TI(IV)

Chromatographic Properties of Sachtopore

- Scaleable from analytical to preparative
- Basic and acidic modifier applicable in NP-mode (Fig. 2)
- Extraordinarily useful for:
- Separation of basic compounds (Fig. 3)
- Large scale separations of isomers (Fig. 5)
- Purification of basic pharmaceuticals (Fig. 5, 6)

Conclus**io**n

Sachtopore is an ideal packing material for NP-separations which are difficult or even impossible to perform on other stationary phases. The unique selectivity of Sachtopore can enhance the selectivity coefficient and can dramatically improve the productivity of the entire large scale separation process. Thus Sachtopore can be much more than an alternative to commonly used packings; it is a serious candidate to substitute them.

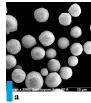
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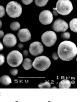
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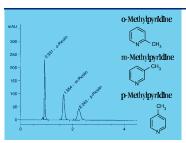


Fig. 2: Normal Phase separation of isomeric picolines using a basic modifier.c Column: Porous Titanium Dioxide (100 Å, 5 um, 150 x 4 mm) Eluent: n-Heptane/Ethanol (NH₃)* = 97/3 (v/v) *Ethanol/NH₃ (25 %) = 1/500 Flow: 1.5 ml/min Detection: UV @ 210 nm Temperature: 43 °C Injection: 2 µl

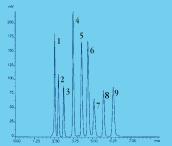
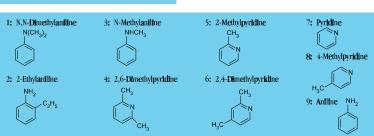


Fig. 3: Normal Phase separation of basic sample solutes.d Column: Porous Titanium Dioxide (100 Å, 5 µm, 150 x 4 mm) Eluent: n-Heptane/2-Propanol = 99.5/0.5 (v/v) Flow: 0.75 ml/min Detection: UV @ 254 nm Temperature: amblent Pressure: 16 bar



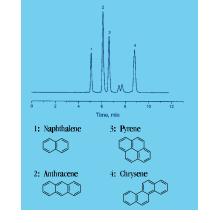


Fig. 4: Separation of unpolar sample solutes.d Column: Porous Titanium Dioxide (100 Å, 5 μm, 250 x 4 mm) Eluent: n-Heptane Flow: 0.5 ml/min Detection: UV @ 230 nm Temperature: ambient Pressure: 33 bar

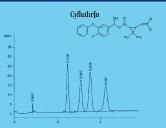


Fig. 5: Normal Phase separation of Isomeric pyrethroids. Column: Porous Titanium Dioxide (100 Å, 5 µm, 150 x 4 mm) Eluent: n-Heptane/MTBE = 80/20 (v/v) Detection: UV @ 220 nm Temperature: 43 °C Injection: 2 µl

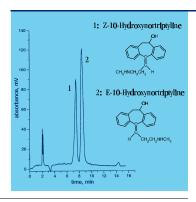


Fig. 6: Normal Phase separation of basic sample solutes.d Column: Porous Titanium Dioxide (100 Å. 5 um. 250 x 4 mm) Eluent; Dichloromethane/Methanol = 96.5/3.5 (v/v) Flow: 1.0 ml/min Detection: UV @ 254 nm Temperature: ambient



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 $^{^{}c} with \ courtesy \ of \ H. \ Schmidt, \ Prof. \ Dr. \ B. \ Wenclawiak, \ Universität \ Gesamt-Hochschule \ Siegen, \ Dr. \ E. \ Weber, \ Bayer \ AG$

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