

TITAN™

Totally Porous, Monodisperse Silica

1.9 µm UHPLC Columns

Patent-Pending Ecoporous
Monodisperse Silica Technology

Maximizes UHPLC Performance
with Monodisperse Porous Silica

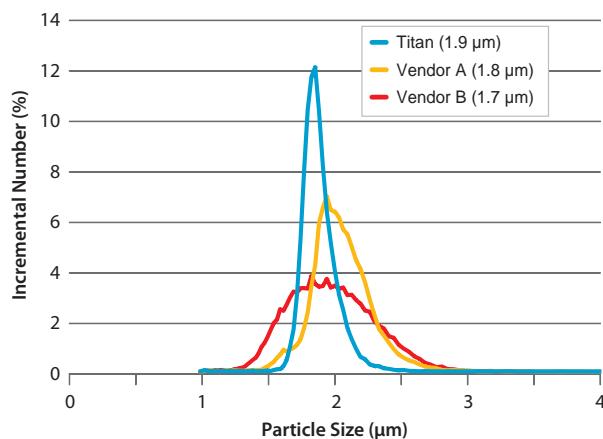
Delivers Exceptional
UHPLC Performance

Exceeds All Other UHPLC Columns
In Performance-to-Price Ratio

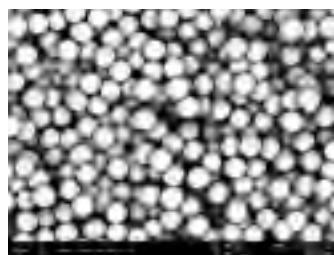
Titan™ UHPLC Columns

Using the New Cost-Efficient, Patent-Pending Ecoporous™ Silica Manufacturing Process

Particle Size Distribution (PSD) Comparison for Different Silica

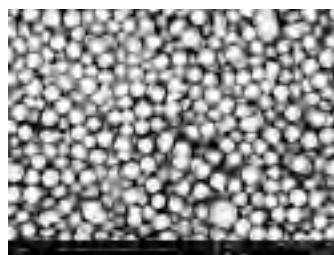


SEM Images of Different Silica



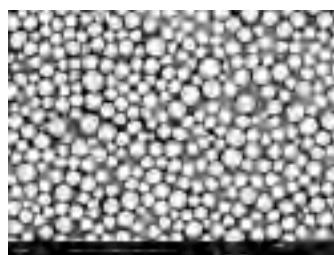
Titan, 1.9 μm

PSD with 6% standard deviation



Vendor A, 1.8 μm

Traditional silica with PSD of 15–25% standard deviation



Vendor B, 1.7 μm

Traditional silica with PSD of 15–25% standard deviation

Unique patent-pending process delivers higher performance-to-price ratio by:

- Providing high-purity monodisperse silica – characterized by particles of uniform size – at lower cost
- Eliminating wasteful silica sizing processes
- Improving performance across HPLC and UHPLC

Titan UHPLC columns are the outcome of the Ecoporous process, a patent-pending, silica manufacturing process that provides an economical UHPLC and HPLC grade silica. Titan UHPLC columns provide all of the performance of leading UHPLC columns at lower cost.

There is growing evidence that a narrow particle size distribution can provide:

- Higher efficiency as a result of a lower A term that is related to bed uniformity and flow equality
- Lower pressure drop (higher permeability) permitting use of higher flows and longer columns
- Smaller reduced plate heights confirming a better quality of the packed column bed
- Higher bed quality which leads to more rugged columns in use

Maximize UHPLC Performance With Monodisperse Porous Silica

Titan C18 is based on a silica particle platform that has the narrowest particle size distribution available of any totally porous particles. This provides performance advantages in the A-term of the Van Deemter equation and the elimination of fines associated with broader particle size distributions.

Monodisperse particles, owing to their narrow particle size distributions, are one of the key reasons core-type particles achieve higher efficiencies than comparative porous particles.

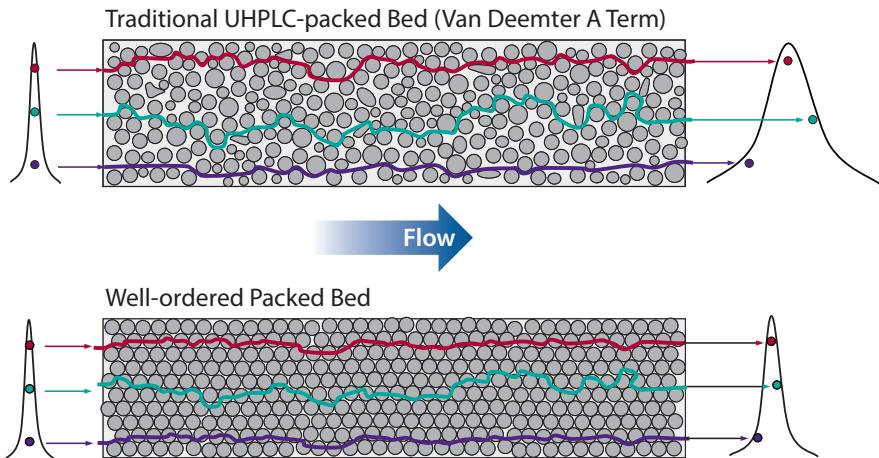
These monodisperse particles offer:

- Minimized voiding and channeling in silica bed compared to higher PSD particles as shown below
- A positive influence on column permeability, as evident by a Titan UHPLC column's low pressure drop compared to other traditional porous particle columns
- A profound affect on separation impedance or kinetic performance resulting in more robust and rugged columns
- Good kinetic performance, Desmet, et al.¹ observed a strong trend between narrow silica PSD and good column performance

Reference

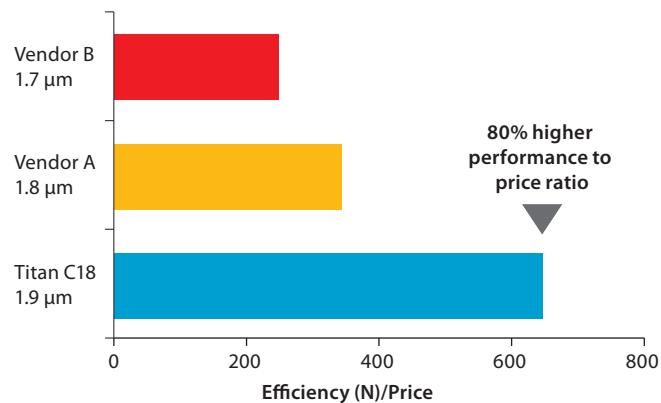
1. D. Cabooter, A. Fanigliulo, G. Bellazzi, B. Allieri, A. Rottigni, G. Desmet, *J. of Chromatography A*, 2010 1217, 7074–7081

Visual Depiction of UHPLC Column Flow Paths

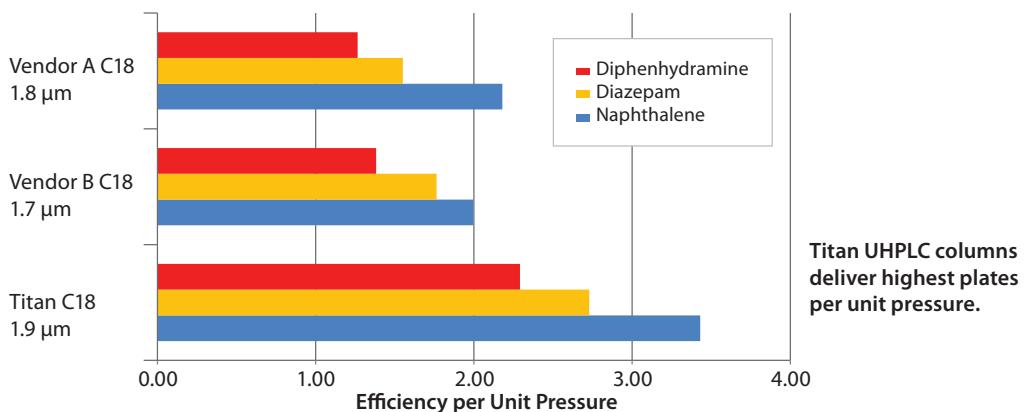


Titan UHPLC Columns Deliver Performance at Incredible Value

Ecoporous technology delivers Titan UHPLC columns at 80% higher performance-to-price ratio compared to key competition



Titan Outperforms Other Fully Porous UHPLC Columns



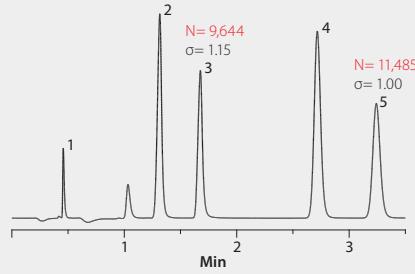
Titan C18, 1.9 μm Performance Comparisons in Methanol and Acetonitrile

Comparison of Titan performance to commercially available sub-2 μm columns

column: 5 cm x 2.1 mm
 mobile phase: 60:40 methanol: 0.1% ammonium acetate (pH 7.1)
 flow rate: 0.25 mL/min
 column temp: 35 °C
 detection: 220 nm
 instrument: Dionex® 3000 (Low D tubing)

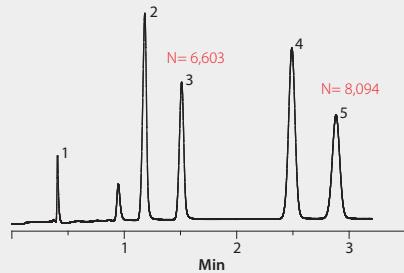
Titan C18, 1.9 μm

Pressure = 4,210 psi



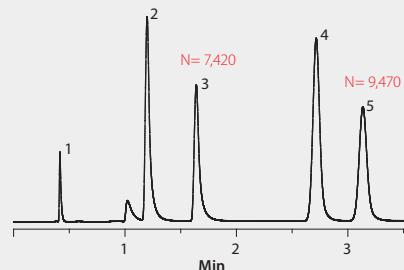
Vendor A C18, 1.8 μm

Pressure = 5,220 psi



Vendor B C18, 1.7 μm

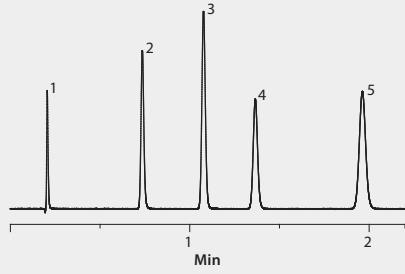
Pressure = 5,370 psi



column: 5 cm x 3.0 mm I.D.
 mobile phase: 40:60 water:acetonitrile
 flow rate: 0.9 mL/min (4 mm/s)
 column temp: 35 °C
 detection: 254 nm
 instrument: Dionex 3000 (Low D tubing)

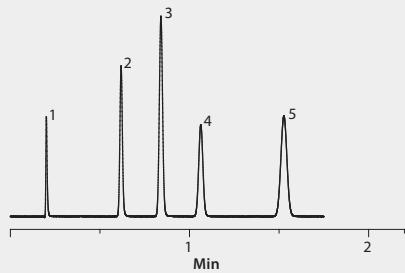
Titan C18, 1.9 μm

$N_{\text{Nap}} = 14,066$
 $N/m = 281,200$
 $H = 1.75$
 $\sigma = 1.05$
 Pressure = 4,100 psi



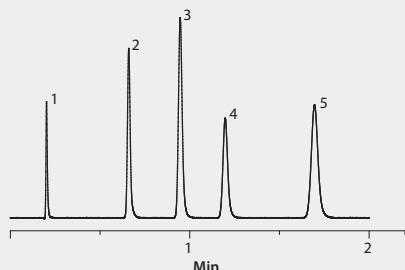
Vendor A C18, 1.8 μm

$N_{\text{Nap}} = 10,131$
 $N_{\text{Nap}}/m = 202,620$
 $H = 3.18$
 $\sigma = 1.02$
 Pressure = 4,650 psi



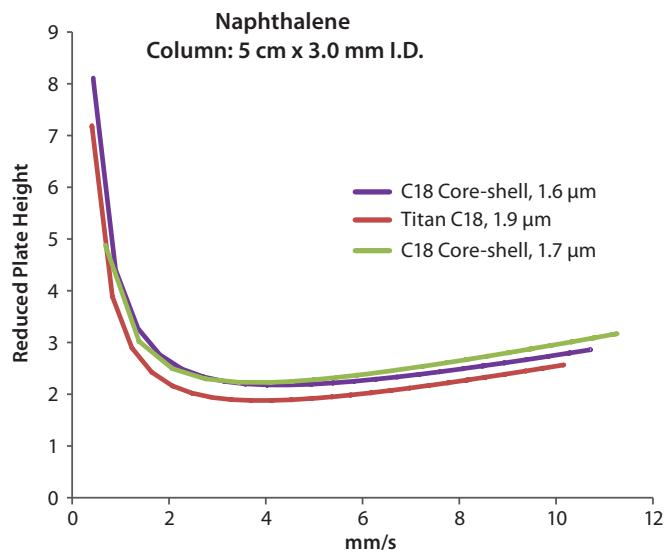
Vendor B C18, 1.7 μm

$N_{\text{Nap}} = 9,783$
 $N_{\text{Nap}}/m = 195,700$
 $H = 2.84$
 $\sigma = 1.16$
 Pressure = 4,900 psi



Titan vs. Core–Shell Performance Comparison

Titan C18, 1.9 μm UHPLC columns based on monodisperse particles are designed to deliver optimum UHPLC performance comparable to latest sub-2 μm core-type particles.



Titan 1.9 μm monodisperse silica is the first porous particle to match or even exceed performance levels for core-type particles with lowest plate height value. A reduced plate height, h , has no units and is determined by dividing plate height, H , by average particle diameter; h values allow performance comparison between columns having different particle sizes. Previously, core-type particles have produced the lowest values for reduced plate height.

A low value for reduced plate height indicates:

- Greater performance from a given particle size; higher efficiency for a given pressure
- Ability to operate longer columns (greater resolution) or the same column at higher flows (greater speed)
- Higher quality of the packed bed, usually the result of a lower A term and more uniform flow path
- Better column preparation or superior particle design (or both)

All column data should be obtained on the same low dispersion instrument in order to make accurate comparisons as shown.

References

1. J. H. Knox, Band Dispersion in Chromatography – A New View of the A Term, *Journal of Chromatography A* 1999, 831, 3–15.
2. J. H. Knox, A Universal Expression for Bandspeading in the Mobile Zone, *Journal of Chromatography A* 2002, 960, 7–18.

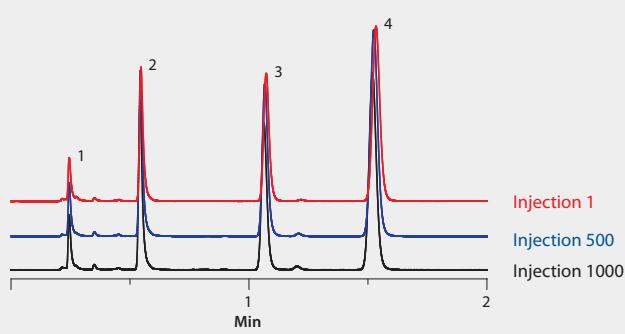
Maximize Reproducibility and Minimize Risk With Titan UHPLC Columns

Titan C18, 1.9 μm Acid Stability (pH 2)

column: Titan C18, 5 cm x 2.1 mm I.D., 1.9 μm (577122-U)
Acidic Conditions (pH 2)

mobile phase: 60% acetonitrile with 0.1% TFA (pH=2)
flow rate: 0.4 mL/min (continuous flow for 1000 injections)
column temp: 35 °C
detector: 220 nm
injection: 0.75 μL
instrument: Agilent® 1290

1. Uracil
2. 2,4-Dichlorophenoxyacetic acid
3. Toluene
4. *p*-Xylene



Percent Change

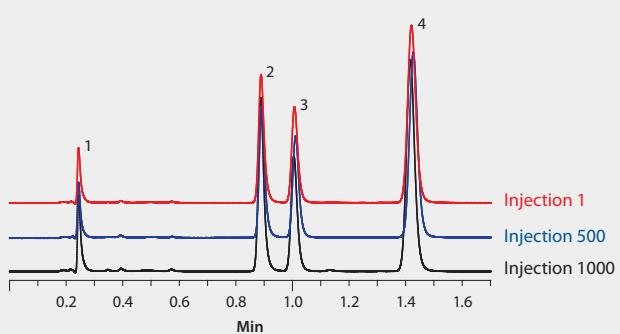
| | k' | Efficiency | USP tailing |
|------------------|------|------------|-------------|
| <i>p</i> -Xylene | 0.68 | -0.24 | -0.88 |

Titan C18, 1.9 μm Base Stability (pH 8)

column: Titan C18, 5 cm x 2.1 mm I.D., 1.9 μm (577122-U)
Basic Conditions (pH 8)

mobile phase: 60% acetonitrile:25 mM ammonium phosphate (pH=8)
flow rate: 0.4 mL/min (continuous flow for 1000 injections)
column temp: 35 °C
detector: 220 nm
injection: 0.4 μL
instrument: Agilent 1290

1. Uracil
2. Dimethylaniline (DMA)
3. Toluene
4. *p*-Xylene



Percent Change

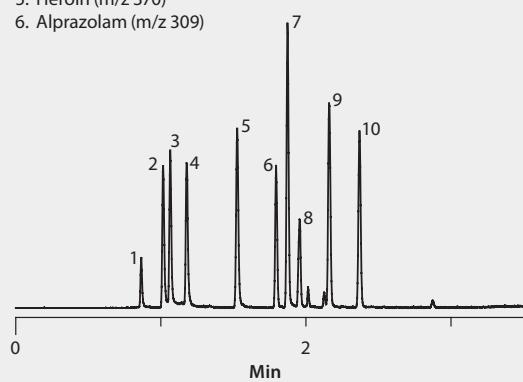
| | k' | Efficiency | USP tailing |
|------------------|------|------------|-------------|
| <i>p</i> -Xylene | 0.85 | 0.15 | -0.87 |

Selected Titan C18, 1.9 µm UHPLC Applications

UHPLC Analysis of Drugs of Abuse on Titan C18: Heroin

column: Titan C18, 5 cm x 2.1 mm I.D., 1.9 µm (577122-U)
 mobile phase: (A) 5 mM ammonium acetate in 95:5, water:acetonitrile;
 (B) 5 mM ammonium acetate in 5:95, water:acetonitrile
 flow rate: 0.6 mL/min
 gradient: 0 to 100% B in 3 min, held at 100% B for 1 min
 pressure: 6770 psi (467 bar)
 column temp.: 35 °C
 detector: MS-TOF, XIC
 injection: 0.5 µL
 sample: 1 µg/mL in 95:5, water:methanol
 instrument: Agilent® 1290; TOF6210

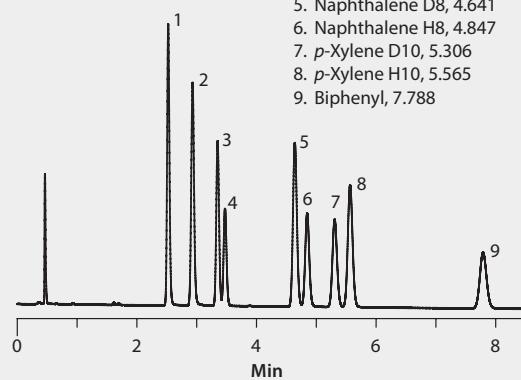
1. Morphine (m/z 286)
2. Procaine (m/z 237)
3. Codeine (m/z 300)
4. 3-Monoacetylmorphine (m/z 328)
5. Heroin (m/z 370)
6. Alprazolam (m/z 309)
7. Papaverine (m/z 340)
8. Flunitrazepam (m/z 314)
9. Diazepam (m/z 285)
10. Noscapine (m/z 414)



High Speed and High Resolution UHPLC Analysis of Deuterated Analogs on Titan C18

column: Titan C18, 10 cm x 2.1 mm I.D., 1.9 µm (577124-U)
 mobile phase: 50% acetonitrile
 flow rate: 0.4 mL/min
 column temp: 35 °C
 detector: UV @ 254 nm
 pressure: 5650 psi (390 bar)
 instrument: Dionex® 3000

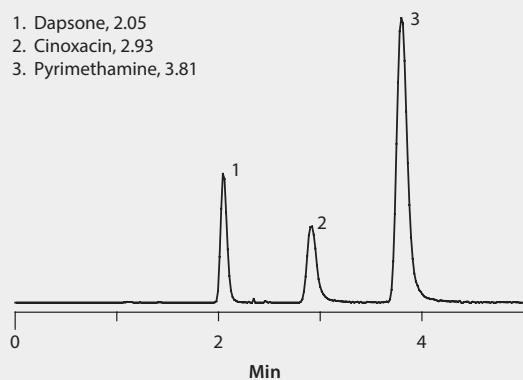
1. Diazepam, 2.523
2. N,N-Dimethylaniline, 2.931
3. Toluene D8, 3.349
4. Toluene H8, 3.473
5. Naphthalene D8, 4.641
6. Naphthalene H8, 4.847
7. p-Xylene D10, 5.306
8. p-Xylene H10, 5.565
9. Biphenyl, 7.788



UHPLC Analysis of Antibiotics/Antimalarials on Titan C18

column: Titan C18, 10 cm x 2.1 mm I.D., 1.9 µm (577124-U)
 mobile phase: (A) 10 mM ammonium acetate, pH 7.0;
 (B) acetonitrile (70:30, A:B)
 flow rate: 0.3 mL/min
 column temp.: 35 °C
 detector: MS, XIC m/z 249.09, 249.07, 263.14
 injection: 2 µL
 sample: 300 ng/mL each in acetonitrile:water (3:1)
 instrument: Agilent 1290; TOF6210

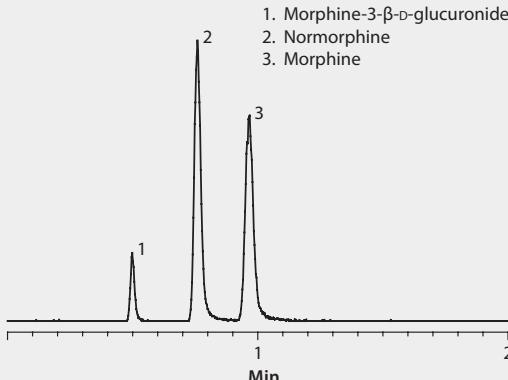
1. Dapsone, 2.05
2. Cinoxacin, 2.93
3. Pyrimethamine, 3.81



UHPLC Analysis of Morphine and Metabolites on Titan C18 Using MS Detection

column: Titan C18, 5 cm x 2.1 mm I.D., 1.9 µm (577122-U)
 mobile phase: (A) water with 0.1% formic acid;
 (B) acetonitrile with 0.1% formic acid; (95:5, A:B)
 flow rate: 0.4 mL/min
 pressure: 4960 psi (342 bar)
 column temp.: 25 °C
 detector: MS-TOF, XIC
 injection: 0.5 µL
 instrument: Agilent 1290; TOF6210

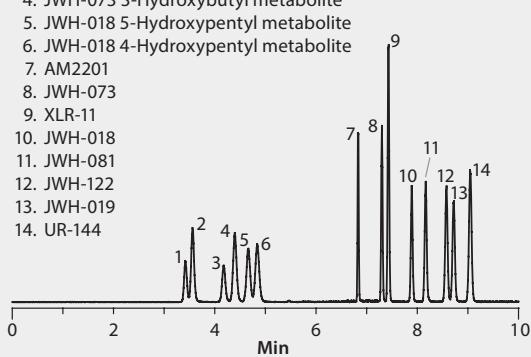
1. Morphine-3-β-D-glucuronide
2. Normorphine
3. Morphine



UHPLC-MS Analysis of Spice Cannabinoids and Metabolites on Titan C18

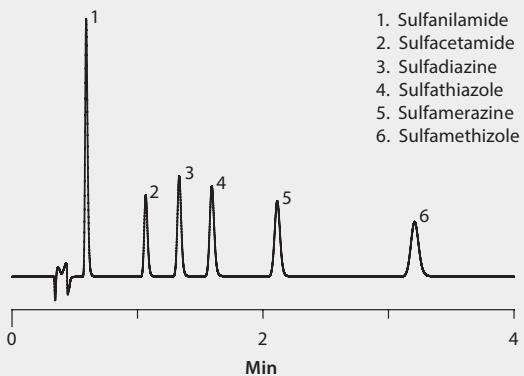
column: Titan C18, 10 cm x 2.1 mm I.D., 1.9 μ m (577124-U)
 mobile phase: (A) 0.1% formic acid in 95:5, water:acetonitrile;
 (B) 0.1% formic acid in 9:5, water:acetonitrile (70:30, A:B)
 gradient: 45% B held for 4.5 min; to 80% B in 1.5 min;
 80% B held for 4 min
 flow rate: 0.5 mL/min
 pressure: 7208 psi (497 bar)
 column temp.: 35 °C
 detector: MS- ESI+, 100 - 1000 m/z scan, XIC
 instrument: Agilent® 1290

1. JWH-073 4-Butanoic acid metabolite
2. JWH-073 4-Hydroxybutyl metabolite
3. JWH-018 5-Pentanoic acid metabolite
4. JWH-073 3-Hydroxybutyl metabolite
5. JWH-018 5-Hydroxypentyl metabolite
6. JWH-018 4-Hydroxypentyl metabolite
7. AM2201
8. JWH-073
9. XLR-11
10. JWH-018
11. JWH-081
12. JWH-122
13. JWH-019
14. UR-144



HPLC Analysis of Sulfa Drugs on Titan C18

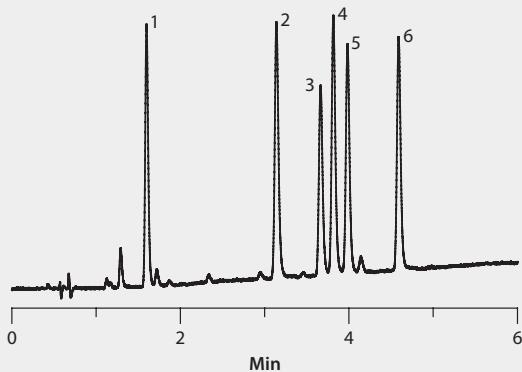
column: Titan C18, 5 cm x 2.1 mm I.D., 1.9 μ m (577122-U)
 mobile phase: (A) water with 0.1% acetic acid;
 (B) acetonitrile with 0.1% acetic acid; (90:10, A:B)
 flow rate: 0.4 mL/min
 pressure: 2670 psi (184 bar)
 column temp.: 40 °C
 detector: UV @ 250 nm
 injection: 2 μ L
 instrument: Dionex 3000



UHPLC Analysis of Taxanes on Titan C18 Column

column: Titan C18, 10 cm x 2.1 mm I.D., 1.9 μ m (577124-U)
 mobile phase: (A) water; (B) (50:50) methanol:acetonitrile
 gradient: 60 to 80% B in 4.5 min; 80% B held for 1.5 min
 flow rate: 0.4 mL/min
 column temp.: 40 °C
 detector: UV @ 227 nm
 injection: 2 μ L
 sample: 300 ng/mL each in acetonitrile:water (3:1)
 instrument: Dionex® 3000

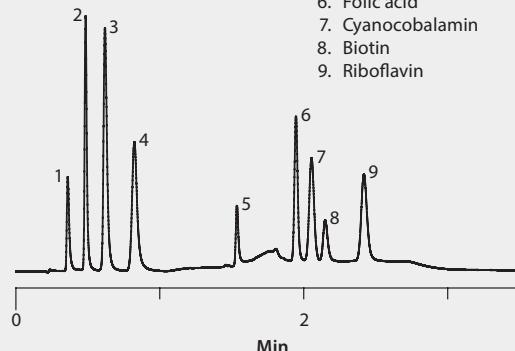
1. Baccatin III
2. 10-Deacetyl paclitaxel
3. Cephalomannine
4. Paclitaxel
5. 10-Deacetyl-7-epipaclitaxel
6. 7-Epipaclitaxel



UHPLC Analysis of B Vitamins on Titan C18 Using UV Detection

column: Titan C18, 5 cm x 2.1 mm I.D., 1.9 μ m (577122-U)
 mobile phase: (A) 20 mM potassium phosphate, pH 3; (B) methanol
 gradient: 0.5% B held for 0.5 minutes, to 30% B in 1.3 minutes,
 held at 30% B for 1 minute, held at 0.5% B for 0.8 minutes
 flow rate: 0.5 mL/min
 pressure: 7700 psi (550 bar)
 column temp.: 30 °C
 detector: UV, 210 nm
 instrument: Dionex 3000

1. Thiamine
2. Nicotinic acid
3. Pyridoxine
4. Niacinamide
5. Calcium pantothenate
6. Folic acid
7. Cyanocobalamin
8. Biotin
9. Riboflavin



Get Started

Additional resources are available for helping you implement Titan UHPLC columns into your laboratory.



Web

Visit sigma-aldrich.com/titan for product information, webinars, ordering and real-time availability information.



Email

Our technical service staff is ready to answer questions.
EU: eurtechserv@sial.com
US: techserv@sial.com



In Person

A technical seminar can be arranged on-site or via the web. Request via seminars@sial.com.

Selecting a Titan UHPLC Column

Which column I.D. is best for my needs?

- If you are doing Mass Spec 2.1 mm I.D.
- If you want high loading 3.0 mm I.D.
- If you want high sensitivity 2.1mm I.D.
- If you want high performance 3.0 mm I.D.

Which column length is best for my needs?

- If you want to maximize the speed of your application 2 to 5 cm
- If you want a balance of resolution and speed 10 cm

What flow rate is best for my needs?

- If you are using a column with a 3.0 mm I.D. 0.8 to 2.0 mL/min*
- If you are using a column with a 2.1 mm I.D. 0.4 to 1.8 mL/min*

*Higher flow rates may be allowed by certain instruments.

For more information, visit
sigma-aldrich.com/titan

Titan Porous Silica Characteristics

| | |
|------------------------|--------------|
| Carbon Load | 13.5% |
| Operating pH Stability | 2–8 |
| Max Temp | 60 °C |
| Features | Endcapped |
| USP Suitability | L1 |
| Particle Size* µm | 1.9 |
| Pore Diameter Å | 80 |
| Surface Area m²/g | 410 |
| Pore Volume cc/g | 0.76 |
| Pressure psi (bar) | 18000 (1241) |

*Very narrow distribution: D (90/10) < 1.15

Ordering Information

| I.D. (mm) | Length (cm) | Qty. | C18 | HILIC | PFP |
|---|-------------|------|----------|----------|----------|
| Titan 1.9 µm U/HPLC Columns | | | | | |
| 2.1 | 2 | 1 | 577120-U | 581528-U | 569675-U |
| 2.1 | 3 | 1 | 577121-U | 581529-U | 569676-U |
| 2.1 | 5 | 1 | 577122-U | 581530-U | 569677-U |
| 2.1 | 7.5 | 1 | 577123-U | 581531-U | 569678-U |
| 2.1 | 10 | 1 | 577124-U | 581532-U | 569679-U |
| 2.1 | 15 | 1 | 577119-U | 581533-U | 569680-U |
| 3 | 3 | 1 | 577125-U | 581534-U | 569681-U |
| 3 | 5 | 1 | 577126-U | 581535-U | 569682-U |
| 3 | 10 | 1 | 577132-U | 581536-U | 569683-U |
| Titan 1.9 µm U/HPLC Guard Cartridges | | | | | |
| 2.1 | — | 3 | 577127-U | 581537-U | 569684-U |
| 3 | — | 3 | 577128-U | 581538-U | 569685-U |
| Titan Guard Cartridge Holder (cartridge not included) | | | | | |
| | | | | | 577133-U |

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