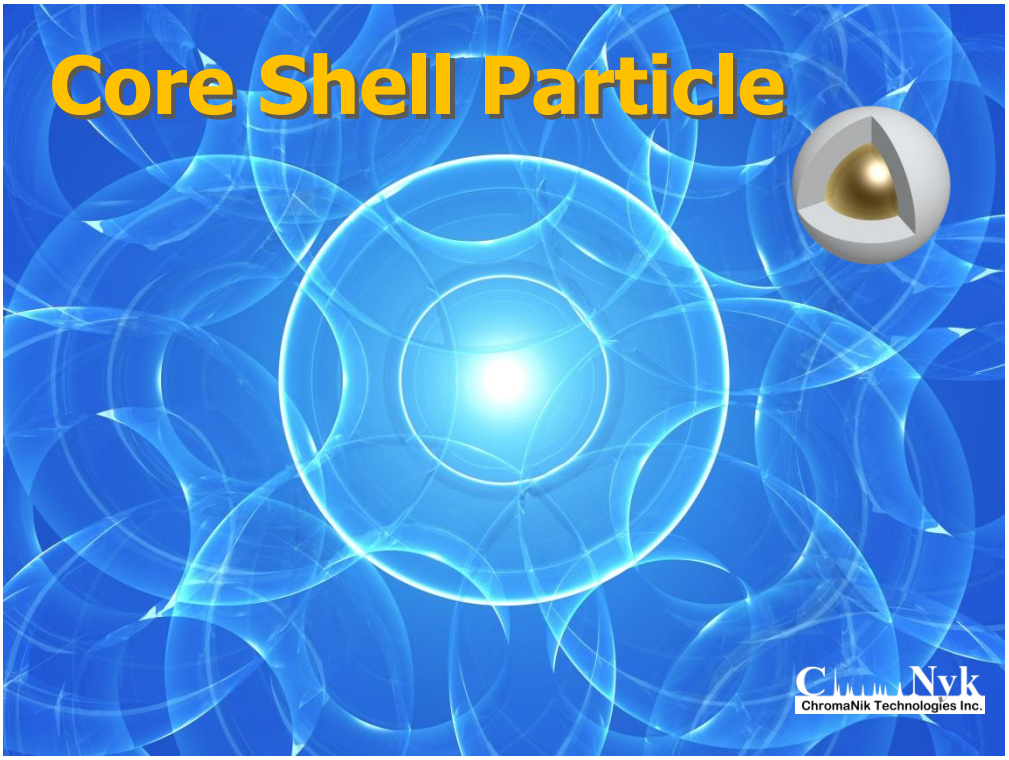
C18, C18-WP, HFC18-16, HFC18-30,RP-AQUA, C8, C30, PFP, Phenyl, C8-30, C8-30HT, C4-30, C4-100, HILIC-Amide, HILIC-S and 2-EP

SunShell

2 µm, 2.6 µm, 3.4 µm and 5 µm HPLC column



SunShell Guard Cartridge Column

ChromaNik Technologies Inc.

ChromaNik Technologies Inc.

"SunShell " is a core shell silica column made by ChromaNIk Technologies.

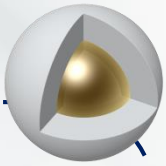
The next generation to Core Shell particle

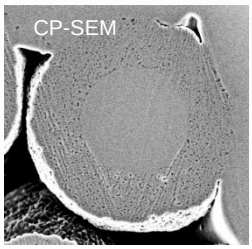
SUNSHELL

Superficially porous silica

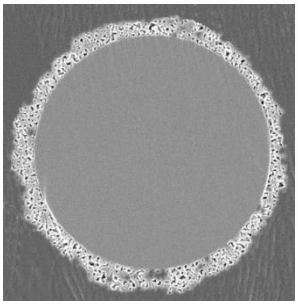
Features of SunShell

* 1.2 µm, 1.6 µm, 3.0 µm and 3.4 µm of core and 0.4 µm, 0.5 µm, 0.2 µm and 0.6 µm of superficially porous silica layer
*Higher efficiency and higher throughput to compare with totally porous silica with same size
*Same chemistry as Sunniest technology (reference page 6)
*Good peak shape for all compounds such as basic, acidic and chelating compounds
*High stability (pH range for SunShell C18, 1.5 to 10) * Low breeding

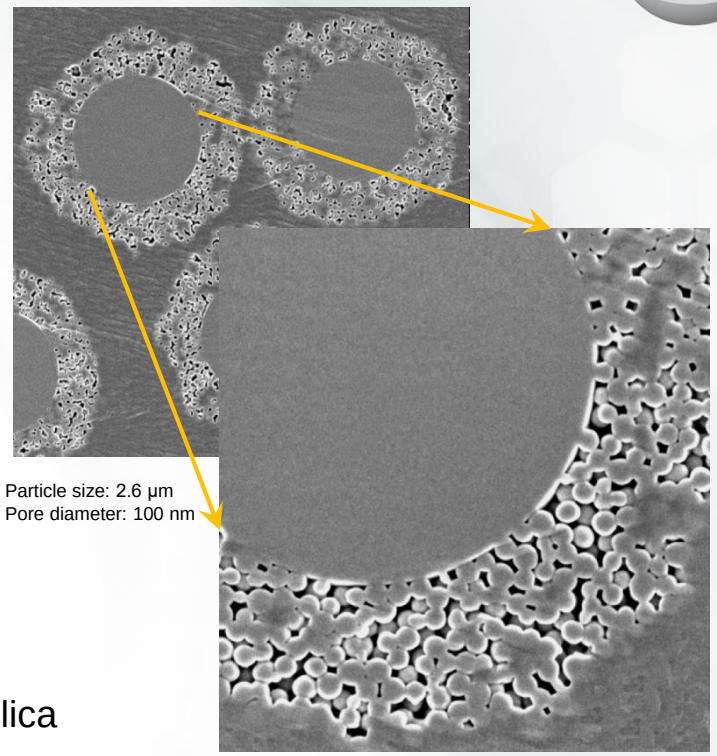




Particle size: 2.6 μm
Pore diameter: 16 nm



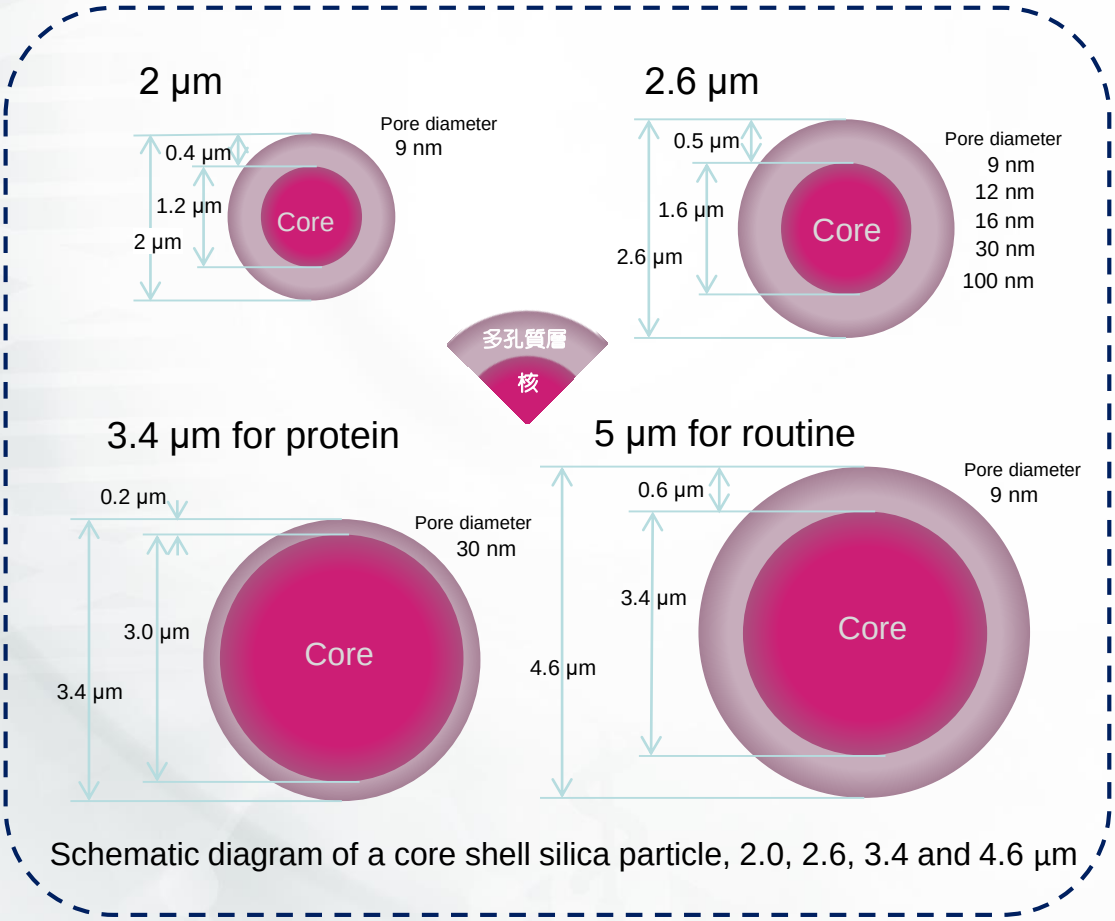
Particle size: 3.4 μm
Pore diameter: 30 nm



Particle size: 2.6 μm
Pore diameter: 100 nm

Electron micrograph of core shell silica

Core shell silica particles were embedded in resin, cross-section processed by Ar ion milling, Os (osmium) vapor deposited for conduction treatment, and observation. You can see the core (fused silica) and the porous layer around it。



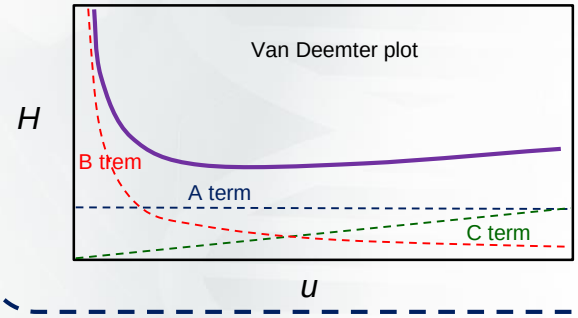
Schematic diagram of a core shell silica particle, 2.0, 2.6, 3.4 and 4.6 μm

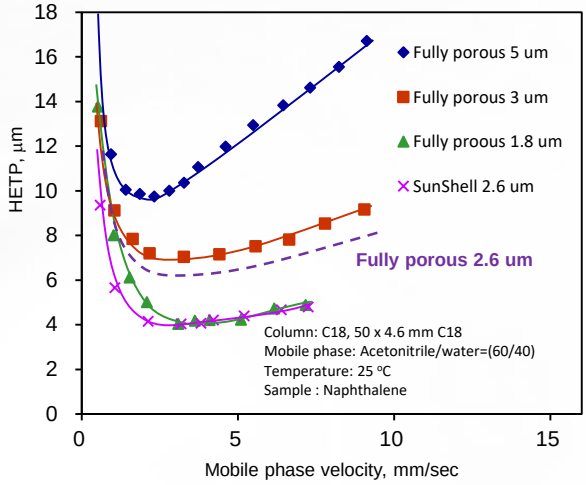
Why does a 2.6 µm core shell particle show the same performance as a sub 2 µm particle?

Van Deemter Equation

$$H = Ad_p + B\frac{D_m}{u} + C\frac{d_p^2}{D_m}u$$

A term : Eddy diffusion(dp is particle diameter)
B term : Longitudinal diffusion
(Dm is diffusion coefficient)
C term : Mass transfer



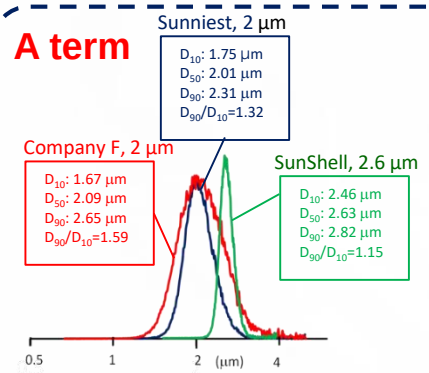


Comparison of plate height plots

SunShell C18 shows same efficiency as a sub 2 µm C18. In comparison between fully porous 2.6 µm and core shell 2.6 µm (SunShell), SunShell shows lower values for A term, B term and C term of Van Deemter equation. The core shell structure leads higher performance to compare with the fully porous structure.

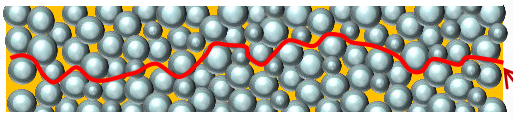
All terms in Van Deemter Equation reduce.

A term



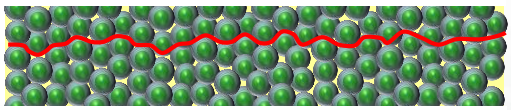
Comparison of Particle Size Distribution

Wide particle distribution (Conventional silica gel D_{90}/D_{10}=1.50)



Flow of mobile phase

Narrow particle distribution (core shell silica D_{90}/D_{10}=1.15)



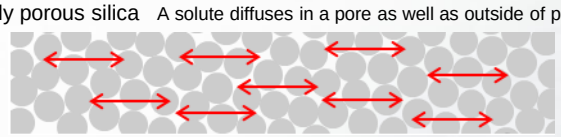
Packing state of core shell and fully porous silica

The size distribution of a core shell (SunShell) particle is much narrower than that of a conventional totally porous particle, so that the space among particles in the column reduces and efficiency increases by reducing Eddy Diffusion (multi-path diffusion) as the A term in Van Deemter Equation.

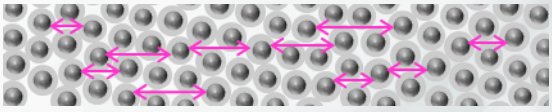
B term

Diffusion of a solute is blocked by the existence of a core, so that a solute diffuses less in a core shell silica column than in a totally porous silica column. Consequently B term in Van Deemter Equation reduces in the core shell silica column.

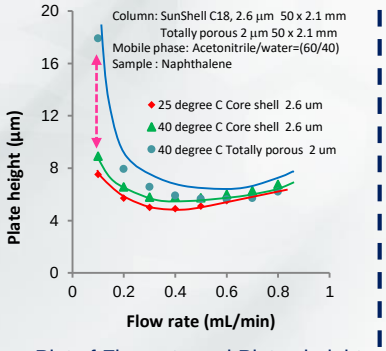
Totally porous silica A solute diffuses in a pore as well as outside of particles.



Core shell silica A core without pores blocks diffusion of a solute.

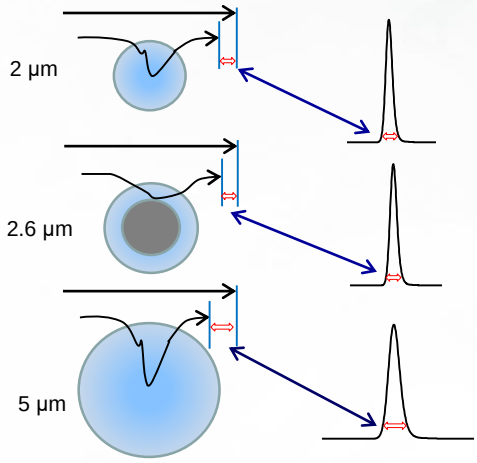


Difference of longitudinal diffusion



Plot of Flow rate and Plates height

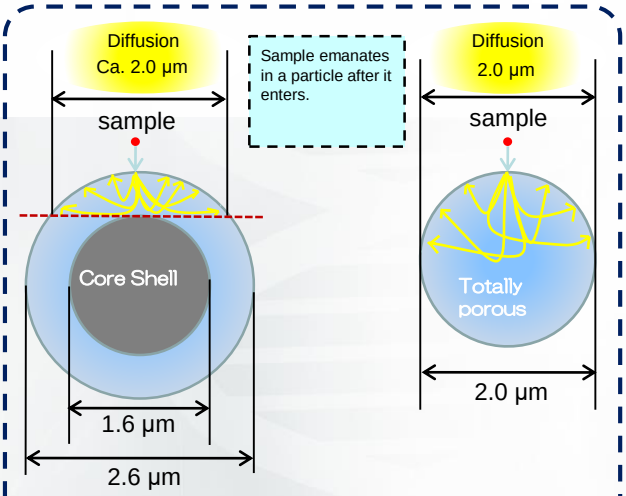
C term



Comparison of diffusion path

As shown in the left figure, a core shell particle has a core so that the diffusion path of samples shortens and mass transfer becomes fast. This means that the C term in Van Deemter Equation reduces. In other words, HETP (theoretical plate) is kept even if flow rate increases. A 2.6 μm core shell particle shows as same column efficiency as a totally porous sub-2 μm particle.

Considering diffusion of solute within pore

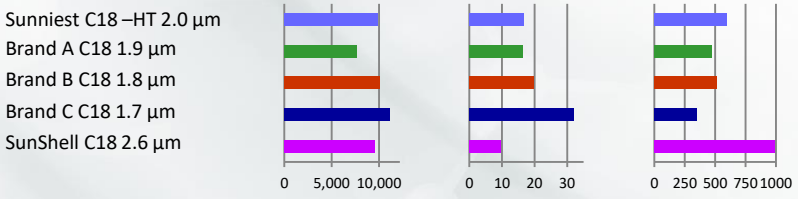


Diffusion of sample in core shell and totally porous silica

The left figure shows that a diffusion width of a sample in a 2.6 μm core shell particle and a 2 μm totally porous particle. Samples or solutes enter into the particle and move by diffusion, then they go out of a particle. In this moment, sample peak width is broadened. This broadening width is statistically same for 2.6 μm core shell particle and 2 μm fully porous particle. The 2.6 μm core shell particle is superficially porous, so that the diffusion width becomes narrower than particle size. Same diffusion means same efficiency.

Comparison of Performance by Plate/Pressure

	Plate	Back press. (MPa)	Plate/back press.
Sunniest C18 –HT 2.0 μm	9,900	16.7	593
Brand A C18 1.9 μm	7,660	16.3	470
Brand B C18 1.8 μm	10,100	19.6	515
Brand C C18 1.7 μm	11,140	32.0	348
SunShell C18 2.6 μm	9,600	9.7	990



Back pressure and theoretical plate were compared for 2 μm and sub 2 μm C18 and 2.6 μm SunShell C18. All columns showed almost the same theoretical plate except for brand A C18 1.9 μm. However back pressure was not same. Especially Brand C C18 1.7 μm showed the highest back pressure. And SunShell C18 2.6 μm showed the lowest back pressure. On the comparison of theoretical plate per back pressure, SunShell indicated the largest value. This is a big advantage.

Column: 50 x 2.1 mm C18, Mobile phase: Acetonitrile/water=(70/30), Temperature: 25 °C

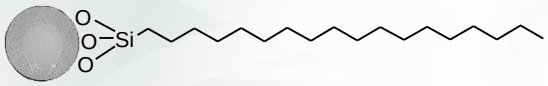
ChromaNik Technologies Inc.

SUNSHELL

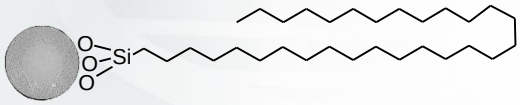
STATIONARY PHASE

Reversed phase

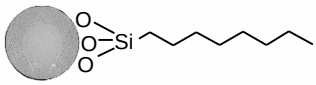
C18, C18-WP (7 page, 16 page, 20 page)



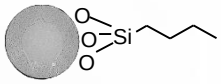
RP-AQUA, C30 (16 page, 19 page)



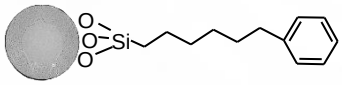
C8, C8-30, C8-30HT (16 page, 20 page, 21 page)



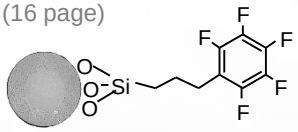
C4-30, C4-100 (20 page, 21 page)



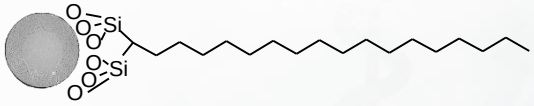
Phenyl (16 page)



PFP (16 page)



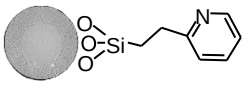
HFC18-16, HFC18-30 (20 page)



**All revered phases except for PFP was end-capped at high temperature using Sunniest Endcapping technique.

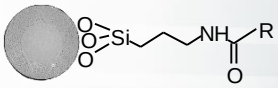
SFC

2EP (22 page)

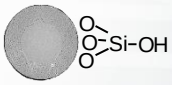


HILIC

HILIC-Amide (23 page)



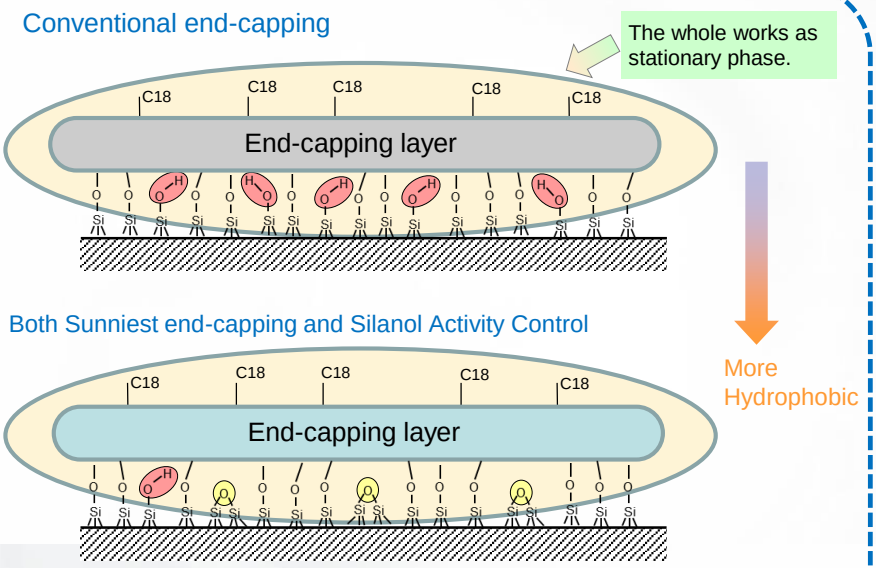
HILIC-S (23 page)



*Stationary phase for both SFC and HILIC was not end-capped.

Unique end-capping by new concept

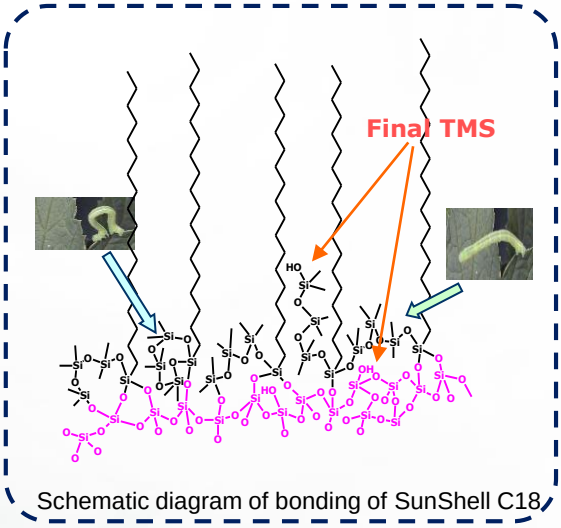
This figure shows comparison of hydrophobicity between two C18 stationary phases. We developed silanol activity control technique which was a reaction at extremely high temperature. This technique makes residual silanol groups change to siloxane bond. The upper one is a C18 phase with conventional end-capping and the lower one is a C18 phase with both Sunniest end-capping and silanol activity control. A residual silanol group contributes as a polar site and makes hydrophobicity of stationary phase decrease. On the other hand siloxane bond in the lower one doesn't make hydrophobicity decrease. Consequently the lower one is more hydrophobic than the upper one.



State of stationary phase with unique end-capping and comparison of hydrophobicity

End-capping method

1) Unique end-capping reagent
<<Hexamethetyltrisiloxane>>
2) Secondly TMS end-capping



Schematic diagram of bonding of SunShell C18

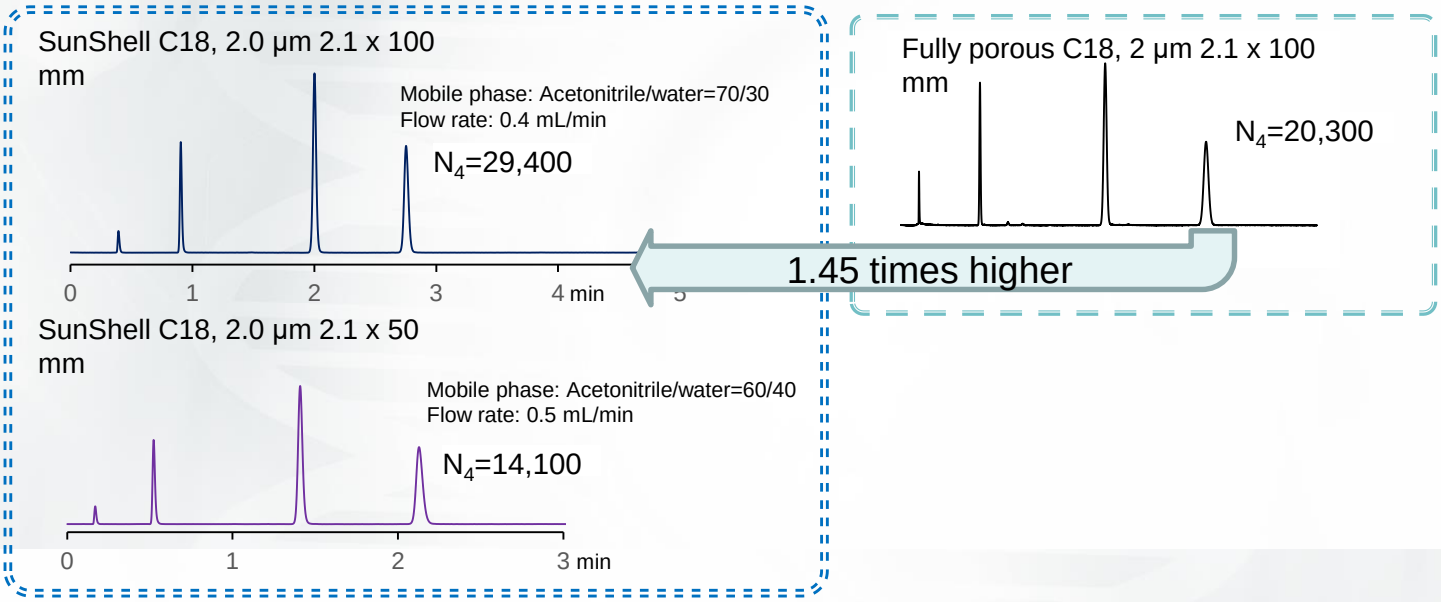
An end-capping of hexamethyltrisiloxane works as an arm. This arm moves like a Geometrid caterpillar, so that a functional group on the tip of the arm can bond with a silanol group which is located anywhere.
Finally TMS reagent is bonded to a remaining silanol group.

SunShell C18, 2 µm, 2.6 µm, 5 µm

Characteristics of SunShell C18

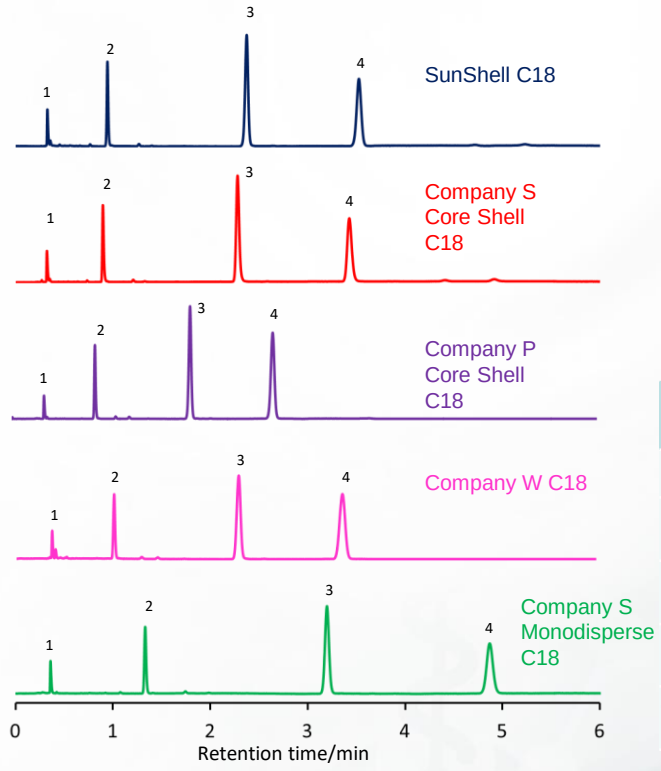
	Core shell silica			C18 (USP L1)				
	Particle size	Pore diameter	Specific surface area	Carbon content	Bonded phase	End-capping	Maximum operating pressure	Available pH range
SunShell C18	2.0 µm	9 nm	120 m^2/g	6.5%	C18	Sunniest endcapping	100 MPa or 14504 psi	1.5 - 10
SunShell C18	2.6 µm	9 nm	150 m^2/g	7%	C18	Sunniest endcapping	60 MPa or 8,570 psi	1.5 - 10
SunShell C18	4.6 µm	9 nm	90 m^2/g	5.5%	C18	Sunniest endcapping	50 MPa or 7,141 psi	1.5 - 10

Core Shell particle shows 1.4 to1.5 times higher plate than fully porous particle.



Theoretical plate and tailing factor

Used columns: SunShell C18 2 µm, Ascentis Express C18 2 µm, Kinetex C18 1.7 µm, Acquity BEH C18 1.7 µm, Titan C18 1.9 µm

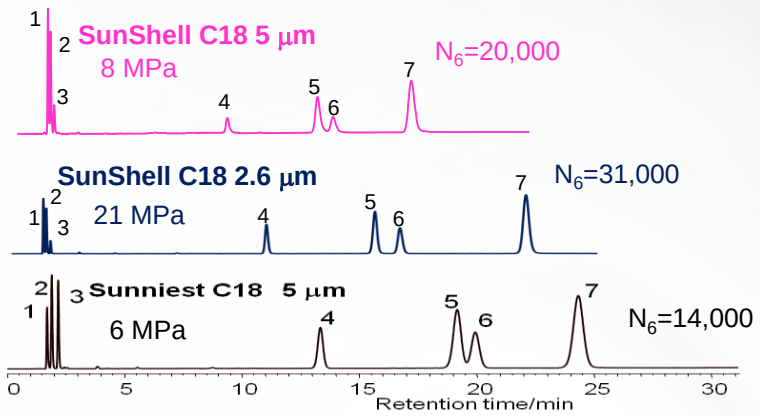


Column: SunShell C18, 2 µm 100 x 2.1 mm
Company S Core Shell C18, 2 µm 100 x 2.1 mm
Company P Core Shell C18, 1.7 µm 100 x 2.1 mm
Company W C18, 1.7 µm 100 x 2.1 mm
Company S Monodisperse C18, 1.9 µm 100 x 2.1 mm
Mobile phase: CH_3CN/H_2O=60/40
Flow rate: 0.5 mL/min
Temperature: 40 °C
Sample: 1 = Uracil, 2 = Ethylbenzoate, 3 = Acenaphthene, 4 = Butylbenzene

	Acenaphthene		Pressure (MPa)	Theoretical plate /pressure (MPa^{-1})
	Theoretical plate	Tailing factor		
SunShell C18	29,721	1.04	48.5	613
Company S Core Shell C18	25,533	1.19	53.5	477
Company P Core Shell C18	24,700	0.97	53.8	458
Company W C18	14,511	1.01	54.1	269
Company S Monodisperse C18	26,592	1.05	43.9	605

*Ascentic Express is a registered trade mark of Sigma Aldrich. Titan is a registered trade mark of Sigma Aldrich.
Comparative separations may not be representative of all applications.

Comparison of retention and plate using HPLC



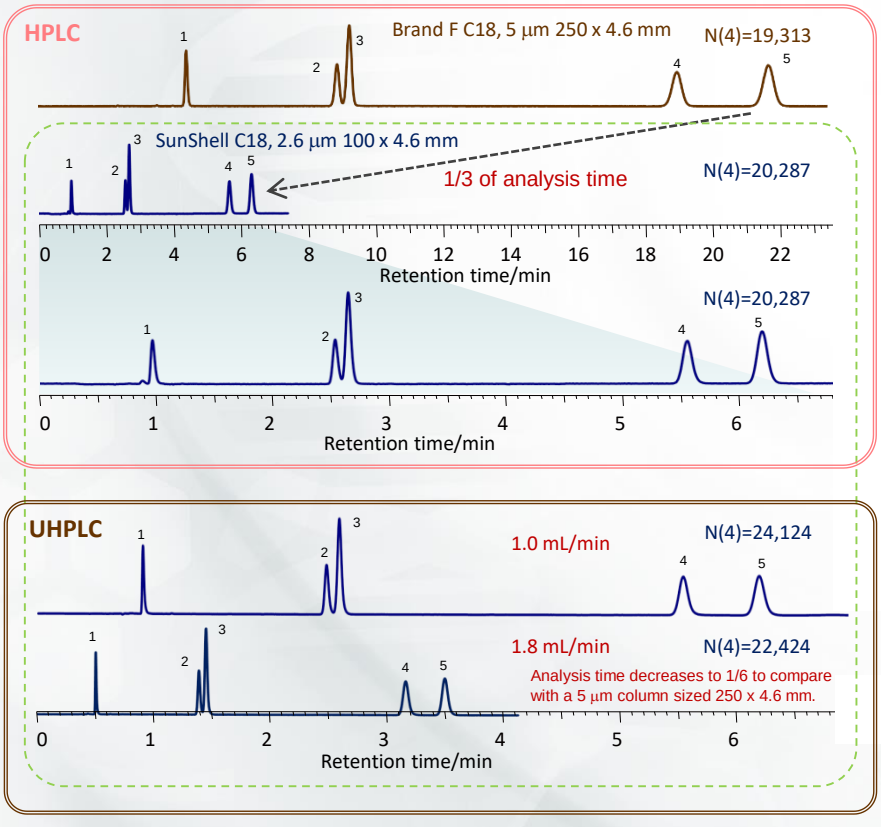
Column size: 150 x 4.6 mm
Mobile phase: CH_3OH/H_2O=75/25
Flow rate: 1.0 mL/min
Temperature: 40 °C
Sample: 1 = Uracil
2 = Caffeine
3 = Phenol
4 = Butylbenzene
5 = o-Terphenyl
6 = Amylbenzene
7 = Triphenylene
HPLC: Hitachi LaChrom ELITE
(Tubing, 0.25 mm i.d.)

	Totally porous silica Sunniest C18, 5 µm		Core shell silica SunShell C18, 2.6 µm		Core shell silica SunShell C18, 5 µm	
Specific surface area	340 m^2/g		150 m^2/g		90 m^2/g	
Packings weight (150x4.6mm)	1.5 g		2.7 g		3.2 g	
Surface area in a column	510 m^2/g (100%)		405 m^2/g (79%)		288 m^2/g (56%)	
	Retention time (t_R)	Retention factor (k)	Retention time (t_R)	Retention factor (k)	Retention time (t_R)	Retention factor (k)
1) Uracil	1.70	0	1.34	0	1.30	0
6) Amylbenzene	19.96	10.74	16.56	11.36	13.43	9.33
Relative value of Amylbenzene	100%	100%	83%	106%	67%	87%

There is a little difference of k between totally porous and core shell particles.

Examples of transfer from a conventional 5 µm column to SunShell column

Isocratic separation



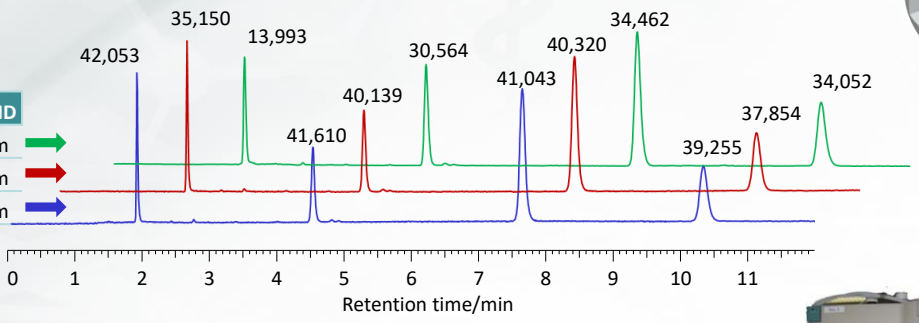
Column:
Brand F C18, 5 µm 250 x 4.6 mm
SunShell C18, 2.6 µm 100 x 4.6 mm
Mobile phase:
CH_3CN/20mM Phosphoric acid = 45/55
Flow rate: 1.0 mL/min,
1.8 mL/min at the lowest chromatogram
Temperature: 25 °C
Pressure: 9.5 MPa for Brand F C18 5 µm
13.4 MPa for SunShell C18 2.6 µm
Detection: UV@230 nm

Sample: 1 = Benzydamine
2 = Ketoprofen
3 = Naproxen
4 = Indomethacin
5 = Ibuprofen

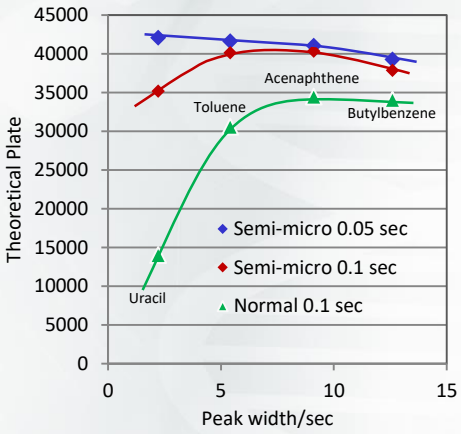
HPLC: Hitachi LaChrom ELITE (Tubing, 0.25 mm i.d.)
UHPLC: Jasco X-LC

Comparison between normal and semi-micro HPLC

Flow cell	Response	Sampling	Tubing ID
Normal	0.1 sec	0.4 sec	0.25 mm
Semi-micro	0.1 sec	0.4 sec	0.13 mm
Semi-micro	0.05 sec	0.05 sec	0.13 mm



Comparison of chromatograms



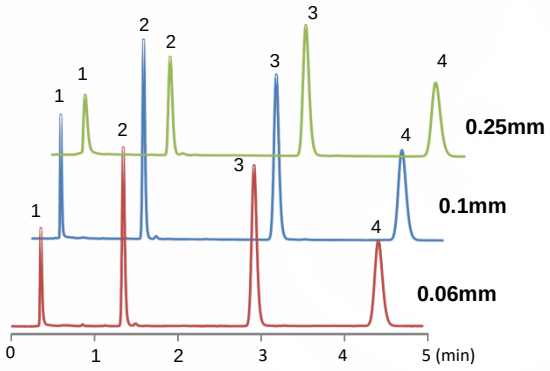
Relationship between Peak width and theoretical plate

Column:
SunShell C18, 5 µm 250 x 4.6 mm
Mobile phase:
CH_3CN/H_2O= 70/30
Flow rate: 1.0 mL/min
Temperature: 40 °C
Pressure: 6.7 MPa
Detection: UV@250 nm
Sample: 1 = Uracil
2 = Toluene
3 = Acenaphthene
4 = Butylbenzene

HPLC: Hitachi LaChrom ELITE

Semi-micro HPLC derives near 100% performance of a core shell column. Even if normal HPLC is used, it derives 80% performance except for a narrow peak whose width is less than 5 second

Effect of inner diameter of tubing



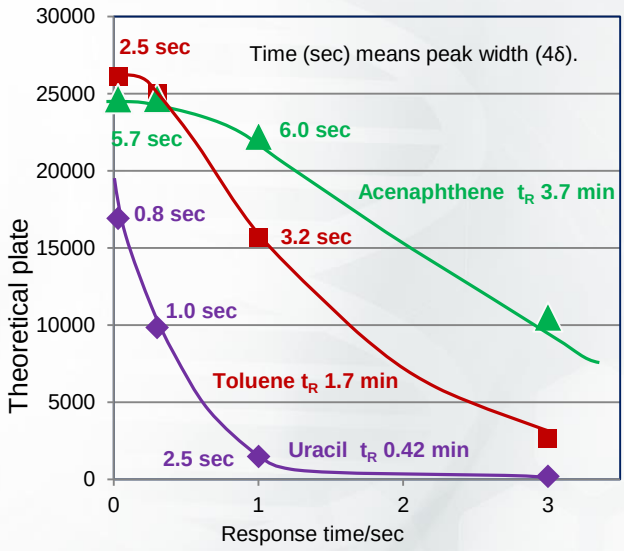
Average of theoretical plate (n=3)

Inner diameter of tubing	0.06mm	0.1mm	0.25mm
Peak (1)	792	785	246
Peak (2)	7790	7652	3535
Peak (3)	10704	10345	7998
Peak (4)	10113	9772	7689

Column: SunShell C18, 2.6 µm 50 x 2.1 mm
Mobile phase: CH_3CN/H_2O=60/40
Flow rate: 0.3 mL/min Temperature: Ambient
Tube length: 30 cm (Peek, from the column to the flow cell)
Instrument: X-LC(JASCO) Response time: 0.01 sec

The above theoretical plate was compared changing the inner diameter of tubing between a column and a flow cell of the detector. A tubing with a large inner diameter has a large dead volume, so that it makes the peak width be wide. As a result, theoretical plate decreases. I recommend to use the tubing with 0.1 mm or less than 0.1 mm inner diameter for core shell columns.

Effect of response time of detector



Column: SunShell C18, 2.6 µm 100 x 4.6 mm
Mobile phase: CH_3CN/H_2O=60/40
Flow rate: 1.8 mL/min Temperature: Ambient
Sample: Toluene Tube: i.d.0.1㎜ x 20 cm Peeksil
Instrument: X-LC(JASCO)

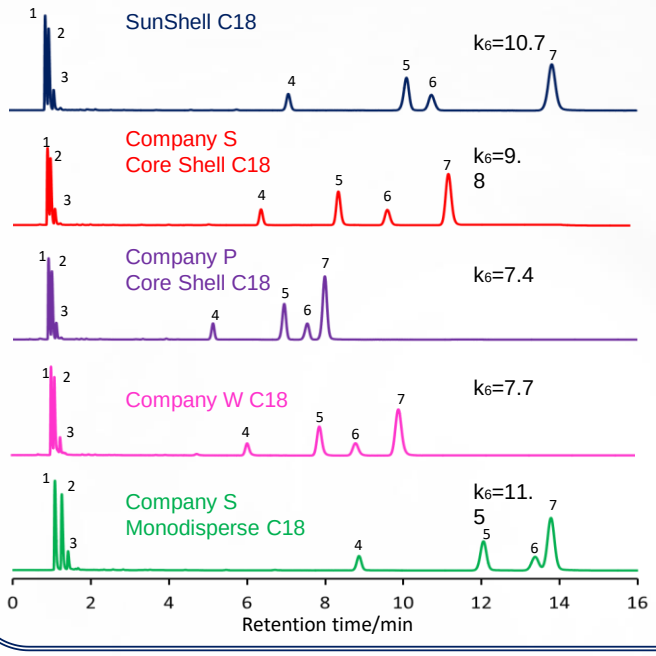
The response time of a detector is important. Regarding uracil, the real peak width is less than 0.8 sec. When the peak width is less than 1 sec, 0.03 sec of response time is needed. Furthermore, the sampling rate of an integrator should be set to be 0.1 sec.

SunShell C18 2 µm

Comparison of core shell 2 µm and totally porous sub 2 µm

Used columns: SunShell C18 2 µm, Ascentis Express C18 2 µm, Kinetex C18 1.7 µm, Acquity BEH C18 1.7 µm, Titan C18 1.9 µm

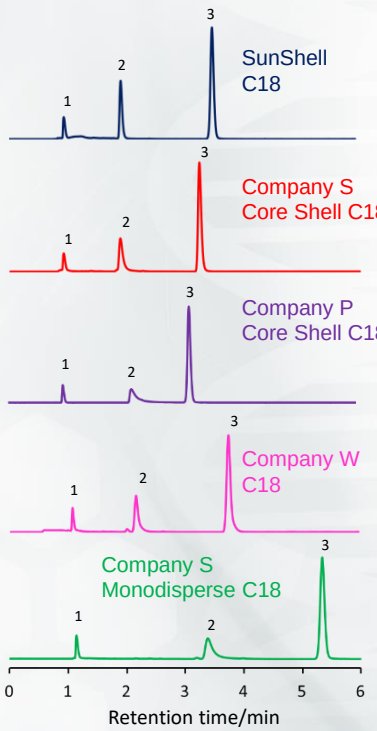
Separation of standard samples



Column: SunShell C18, 2 µm 100 x 2.1 mm
Company S Core Shell C18, 2 µm 100 x 2.1 mm
Company P Core Shell C18, 1.7 µm 100 x 2.1 mm
Company W C18, 1.7 µm 100 x 2.1 mm
Company S Monodisperse C18, 1.9 µm 100 x 2.1 mm
Mobile phase: CH_3OH/H_2O=75/25
Flow rate: 0.2 mL/min
Temperature: 40 °C
Sample: 1 = Uracil, 2 = Caffeine, 3 = Phenol, 4 = Butylbenzene
5 = o-Terphenyl, 6 = Amylbenzene, 7 = Triphenylene

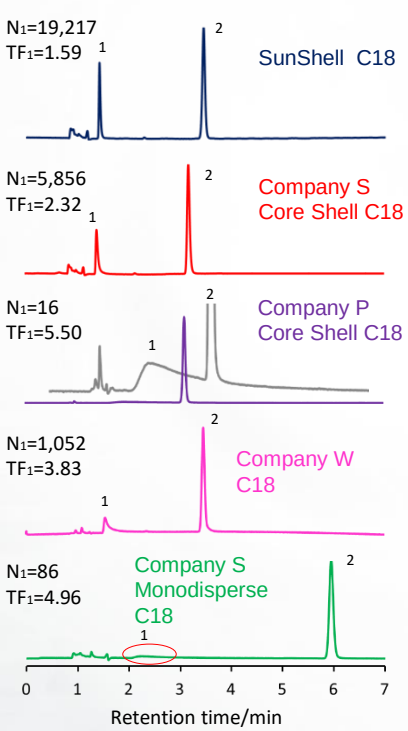
	Hydrogen bonding (Caffeine/Phenol)	Hydrophobicity (Amylbenzene/Butylbenzene)	Steric selectivity (Triphenylene/o-Terphenyl)
SunShell C18	0.43	1.59	1.41
Company S Core Shell C18	0.37	1.59	1.38
Company P Core Shell C18	0.45	1.57	1.17
Company W C18	0.35	1.55	1.30
Company S Monodisperse C18	0.53	1.58	1.16

Comparison of Pyridine (2) as a basic compound



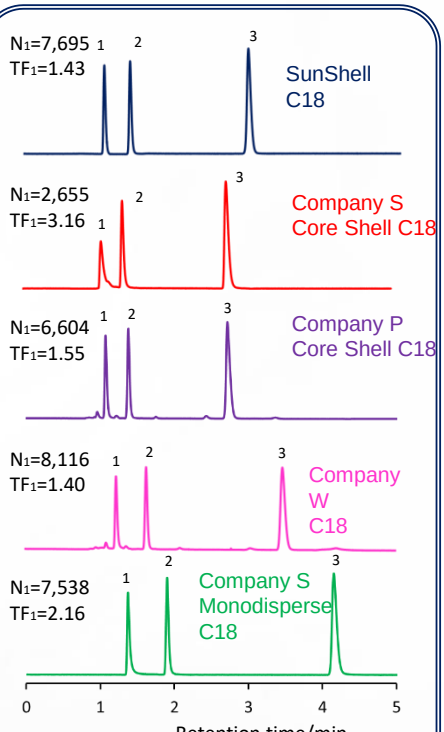
Column dimension: 100 x 2.1 mm
Mobile phase: CH_3OH/H_2O=30/70
Flow rate: 0.2 mL/min
Temperature: 40 °C
Detection: UV@250nm
Sample: 1 = Uracil
2 = Pyridine
3 = Phenol

Comparison of Oxine (1) as a metal chelating compound



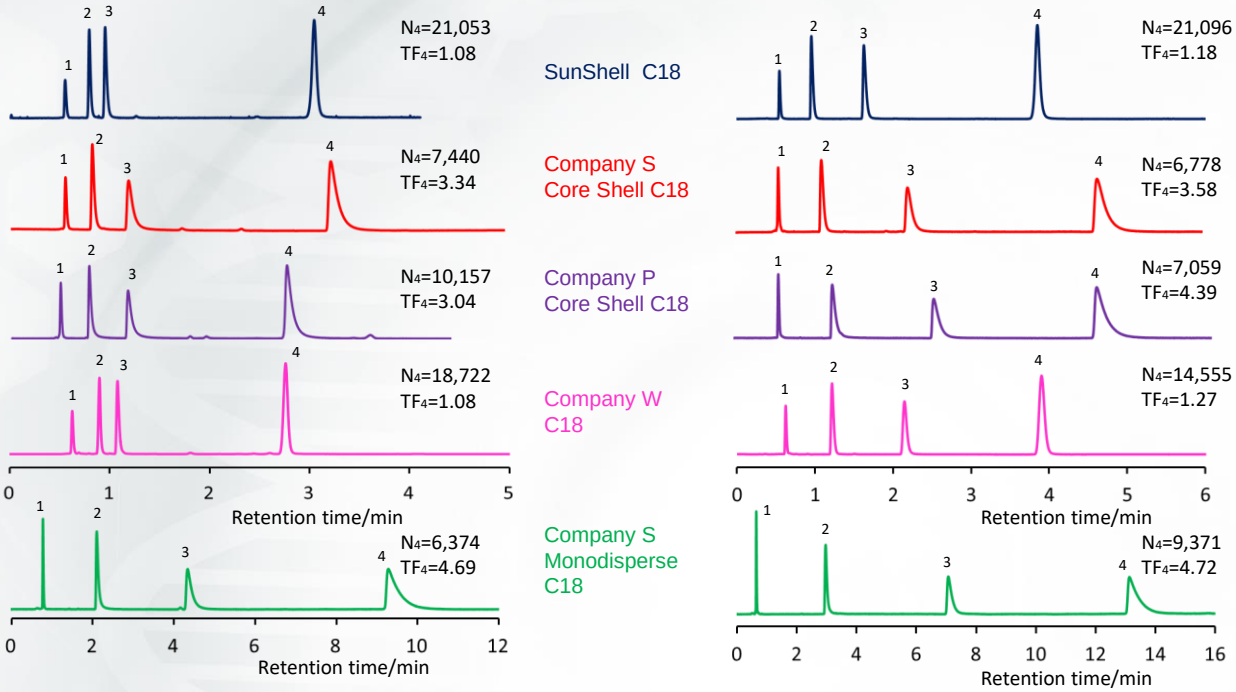
Column dimension: 100 x 2.1 mm
Mobile phase: CH_3CN/20mM H_3PO_4=10/90
Flow rate: 0.2 mL/min
Temperature: 40 °C
Detection: UV@250nm
Sample: 1 = 8-Quinolinol (Oxine)
2 = Caffeine

Comparison of Formic acid (1) as an acidic compound

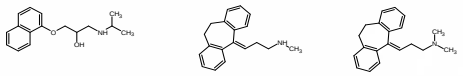


Column dimension: 100 x 2.1 mm
Mobile phase: CH_3CN/0.1% H_3PO_4=2/98
Flow rate: 0.2 mL/min
Temperature: 40 °C
Detection: UV@210nm
Sample: 1 = Formic acid
2 = Acetic acid
3 = Propionic Acid

Comparison of Amitriptyline (4) as a strong basic compound

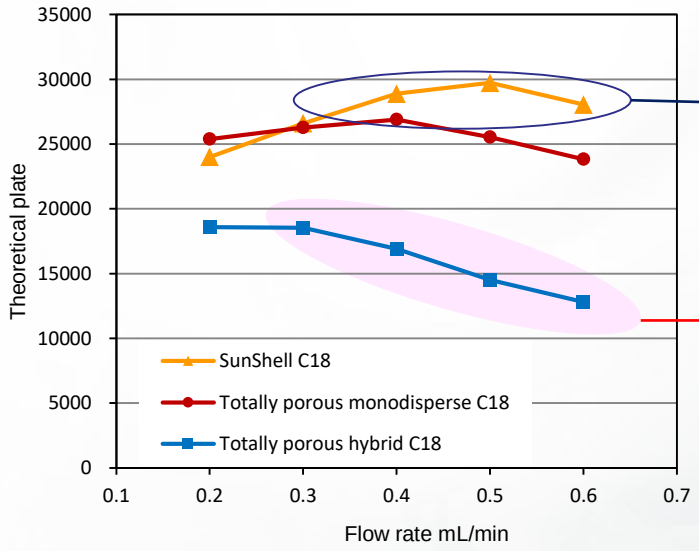


Column dimension: 100 x 2.1 mm
Mobile phase: CH_3CN/20 mM Phosphate buffer pH 7.0=60/40
Flow rate: 0.3 mL/min
Temperature: 40 °C
Detection: UV@250 nm
Sample: 1 = Uracil, 2 = Propranolol, 3 = Nortriptyline, 4 = Amitriptyline



Column dimension: 100 x 2.1 mm
Mobile phase: CH_3CN/10 mM ammonium acetate pH 6.8=40/60
Flow rate: 0.3 mL/min
Temperature: 40 °C
Detection: UV@250 nm
Sample: 1 = Uracil
2 = Propranolol
3 = Nortriptyline
4 = Amitriptyline

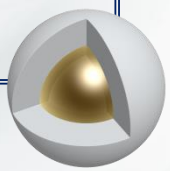
Decreasing of theoretical plate due to frictional heating effect



Core shell silica has a solid core (non-porous silica), so that thermal conductivity is high in the column. There is no influence of reducing theoretical plate by frictional heating.

Regarding totally porous hybrid silica, not only totally porous structure but also including ethylene groups make thermal conductivity be low in the column. It is considered that frictional heating deflects thermal distribution in the column and theoretical plate decreases..

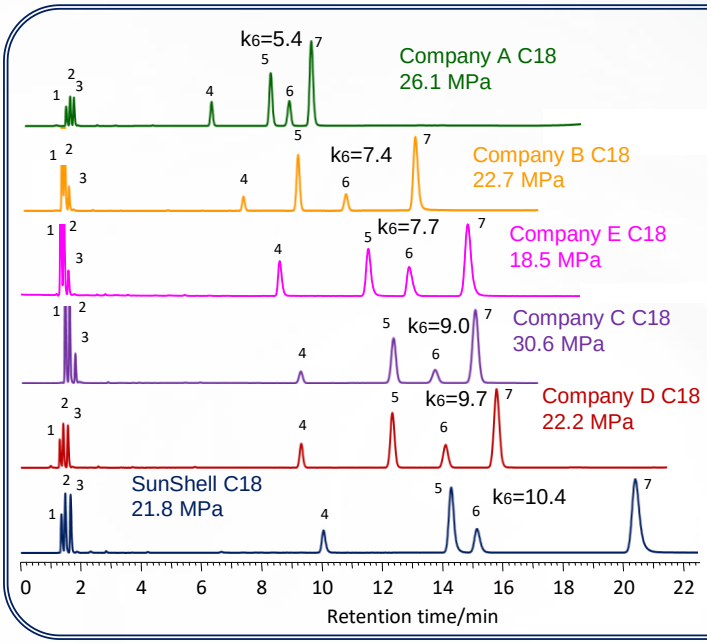
Column: 100 x 2.1 mm
Mobile phase: CH_3CN/H_2O=60/40
Temperature: 40 °C
Sample: Acenaphthene,



Comparison of core shell 2.6 µm columns

Used columns
1. Kinetex C18, 2.6 µm
2. Accucore C18, 2.6 µm
3. PoroShell C18 EC, 2.7 µm
4. Ascentis Express C18, 2.7 µm
5. Cortecs C18, 2.7 µm
6. SunShell C18, 2.6 µm

Comparison of standard samples between core shell C18s

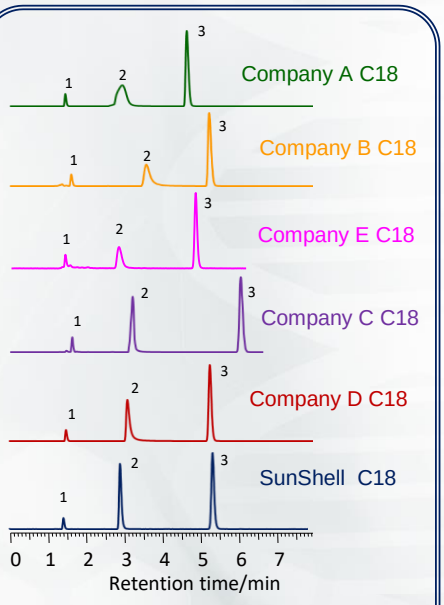


Column: Company A C18, 2.6 µm 150 x 4.6 mm (26.1 Mpa, 30,800 plate)
Company B C18, 2.6 µm 150 x 4.6 mm (22.7 MPa , 31,600 plate)
Company W C18, 2.7 µm 150 x 4.6 mm (18.5 MPa , 23,300 plate)
Company C C18, 2.7 µm 150 x 4.6 mm (30.6 MPa , 30,200 plate)
Company D C18, 2.7 µm 150 x 4.6 mm (22.2 MPa , 31,800 plate)
SunShell C18, 2.6 µm 150 x 4.6 mm (21.8 MPa , 31,900 plate)
Mobile phase: CH_3OH/H_2O=75/25
Flow rate: 1.0 mL/min, Temperature: 40 °C
Sample: 1 = Uracil, 2 = Caffeine, 3 = Phenol, 4 = Butylbenzene
5 = o-Terphenyl, 6 = Amylbenzene, 7 = Triphenylene

	Hydrogen bonding (Caffeine/Phenol)	Hydrophobicity (Amylbenzene/Butylbenzene)	Steric selectivity (Triphenylene/o-Terphenyl)
Company A C18	0.48	1.54	1.20
Company B C18	0.35	1.56	1.50
Company E C18	0.38	1.59	1.32
Company C C18	0.42	1.57	1.25
Company D C18	0.44	1.60	1.31
SunShell C18	0.39	1.60	1.46

Retention of standard samples and back pressure were compared for six kinds of core shell type C18s. Company A C18 showed only a half retention to compare with SunShell C18. Steric selectivity becomes large when ligand density on the surface is high. SunShell C18 has the largest steric selectivity so that it has the highest ligand density. This leads the longest retention time.

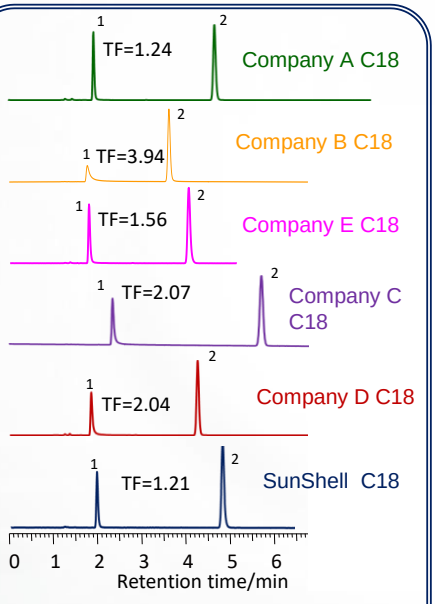
Comparison of pyridine



Column dimension: 150 x 4.6 mm
Mobile phase: CH_3OH/H_2O=30/70
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@250nm
Sample: 1 = Uracil
2 = Pyridine
3 = Phenol

Residual silanol groups make pyridine be tailing under methanol/water mobile phase condition. SunShell C18 shows a sharp peak for pyridine.

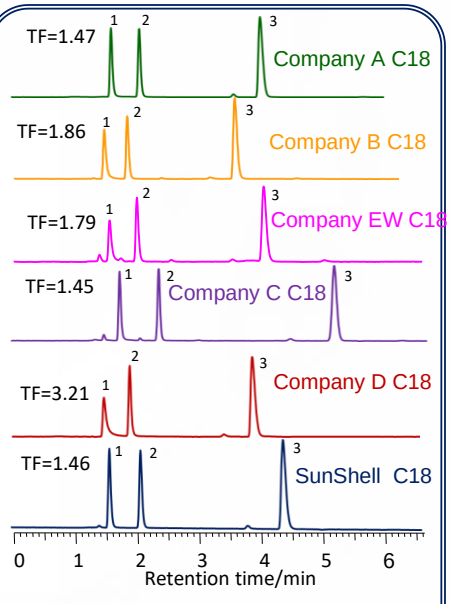
Comparison of Oxine



Column dimension: 150 x 4.6 mm
Mobile phase: CH_3CN/20mM H_3PO_4=10/90
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@250nm
Sample: 1 = 8-Quinolinol (Oxine)
2 = Caffeine

8-Quinolinol (Oxine) is a metal chelating compound. Metal impurities in the core shell particle leads the tailing for oxine peak.

Comparison of formic acid



Column dimension: 150 x 4.6 mm
Mobile phase: CH_3CN/0.1% H_3PO_4=2/98
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@210nm
Sample: 1 = Formic acid
2 = Acetic acid
3 = Propionic Acid

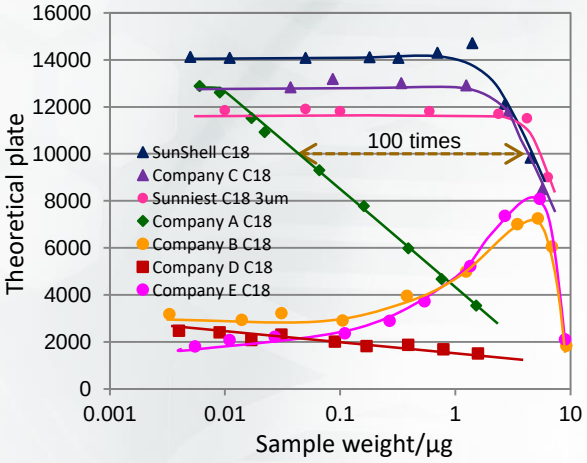
Formic acid is used as an indicator for a acidic inertness. SunShell and Company A and C C18 show a sharp peak.

Loading capacity of amitriptyline as a basic compound

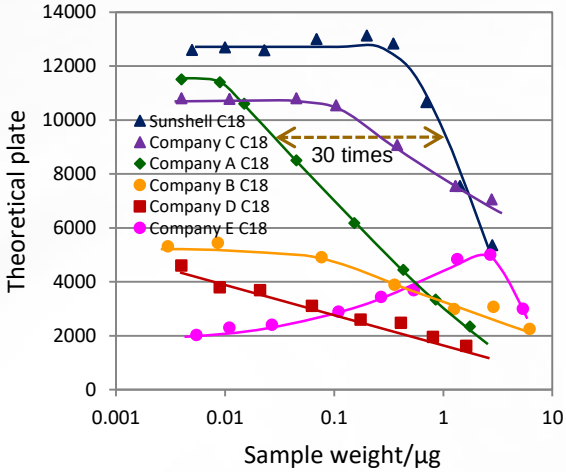
Amitriptyline overlords much more at acetonitrile/buffer mobile phase than methanol/buffer. Three kinds of core shell C18s were compared loading capacity of amitriptyline at three different mobile phases.

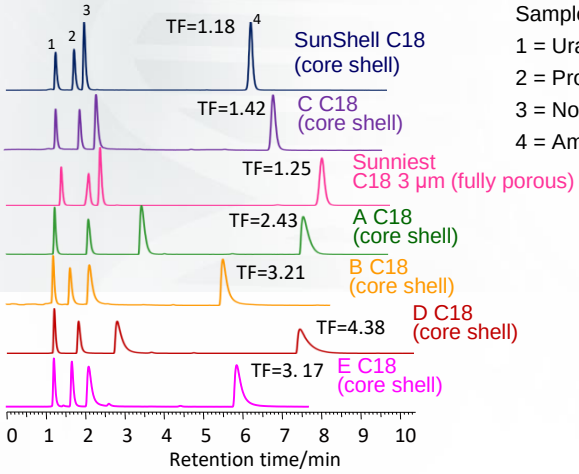
Common condition: Column dimension,150 x 4.6 mm, flow rate; 1.0 mL/min, temperature; 40 °C

Mobile phase: Acetonitrile/**20mM phosphate buffer pH7.0**=(60:40)

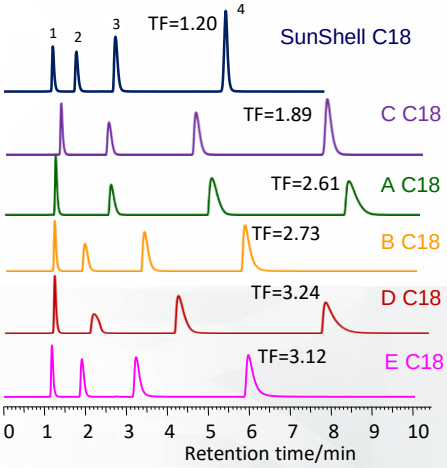


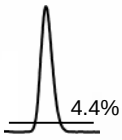
Mobile phase: Acetonitrile/**10mM acetate ammonium pH6.8**=(40:60)





Sample:
1 = Uracil (0.07μg)
2 = Propranolol (1.53μg)
3 = Nortriptyline (0.32μg)
4 = Amitriptyline (0.32μg)





Theoretical plate was calculated by 5σ method using peak width at 4.4% of peak height.

Physical properties

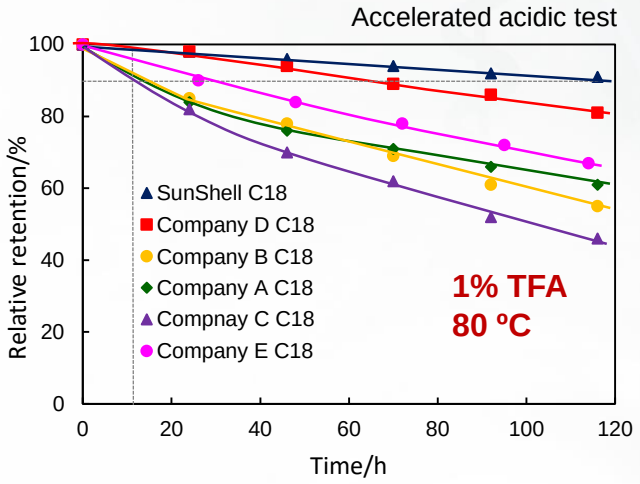
	Carbon loading (%)	Specific surface area [a] (m^2/g)	Pore volume [a] (mL)	Pore diameter [a] (nm)
SunShell C18	7.3 (7)[b]	125 (150)[b]	0.261	8.34 (9)[b]
Ascentis Express C18	8.0	133 (150)[b]	0.278	8.20 (9)[b]
PoroShell C18 EC	8.5 (8)[b]	135 (130)[b]	0.414	12.3 (12)[b]
Accucore C18	8.8 (9)[b]	130 (130)[b]	0.273	8.39 (8)[b]
Cortecs C18	7.3 (6.6)[b]	113	0.264	9.32
Kinetex C18	4.9 (12 effective)[b]	102 (200 effective)[b]	0.237	9.25 (10)[b]

Comparison column
1. Kinetex C18, 2.6 μm
2. Accucore C18, 2.6 μm
3. PoroShell C18 EC, 2.7 μm
4. Ascentis Express C18, 2.7 μm
5. Cortecs C18 2.7 μm
6. SunShell C18, 2.6 μm

a. Measured after sintered at 600 degree Celsius for 8 hours.
b. Value cited in company brochure or literature.

All columns are core shell type. All columns sized 150 x 4.6 mm except for company E show 38,000 to 40,000 plates for a neutral compound. However regarding a basic compound like amitriptyline, SunShell C18 and company C C18 showed a good peak, while Company A, B and D C18 showed a poor peak. Company A C18 overloaded at more than 0.01 μg of amitriptyline while SunShell C18 overloaded at more than from 0.3 to 1 μg of amitriptyline. Surprisingly loading capacity of company A C18 was only one hundredth to compare with SunShell C18 under acetonitrile/20mM phosphate buffer pH7.0=(60:40) mobile phase. Company D C18 always showed poor peak of amitriptyline.

◆Evaluation of Stability



Durable test condition

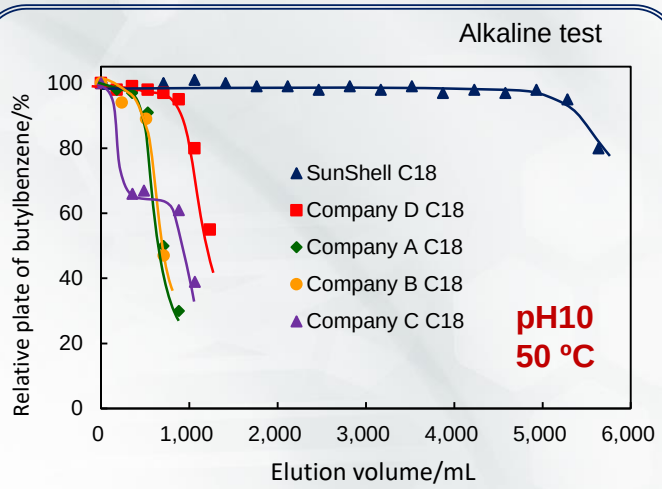
Column size: 50 x 2.1 mm
Mobile phase: CH_3CN/1.0% TFA, pH1=10/90
Flow rate: 0.4 mL/min
Temperature: 80 ºC

Measurement condition

Column size: 50 x 2.1 mm
Mobile phase: CH_3CN/H_2O=60/40
Flow rate: 0.4 mL/min
Temperature: 40 ºC
Sample: 1 = Uracil (t_0)
2 = Butylbenzene

Stability under acidic pH condition was evaluated at 80 ºC using acetonitrile/1% trifluoroacetic acid solution (10:90).

★Sunshell C18 has kept 90% retention for 100 hours under such a severe condition. SunShell C18 is 5 to 10 times more stable than the other core shell C18.



Durable test condition

Column Size: 50 x 2.1 mm
Mobile phase:
CH_3OH/20mM Sodium borate/10mM NaOH=30/21/49 (pH10)
Flow rate: 0.4 mL/min
Temperature: 50 ºC

Measurement condition

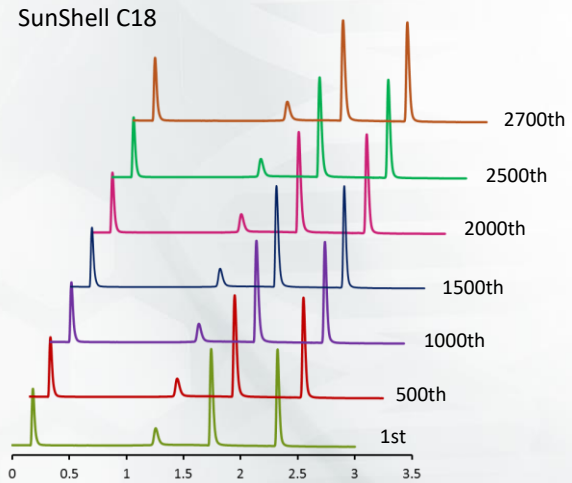
Column Size: 50 x 2.1 mm
Mobile phase: CH_3CN/H_2O=60/40
Flow rate: 0.4 mL/min
Temperature: 40 ºC
Sample: 1 = Butylbenzene

Stability under basic pH condition was evaluated at 50 ºC using methanol/Sodium borate buffer pH 10 (30:70) as a mobile phase. Sodium borate is used as a alkaline standard solution for pH meter, so that its buffer capacity is high.

Elevated temperature of 10 ºC makes column life be one third. The other company shows stability test at ambient (room temperature). If room temperature is 25 ºC, column life at room temperature (25 ºC) is sixteen times longer than that at 50 ºC.

★ SunShell C18 is enough stable even if it is used under pH 10 condition. Regarding stability under basic pH condition, there is little C18 column like SunShell C18 except for hybrid type C18. It is considered that our end-capping technique leads high stability.

◆Continuous analysis under pH9.5 condition

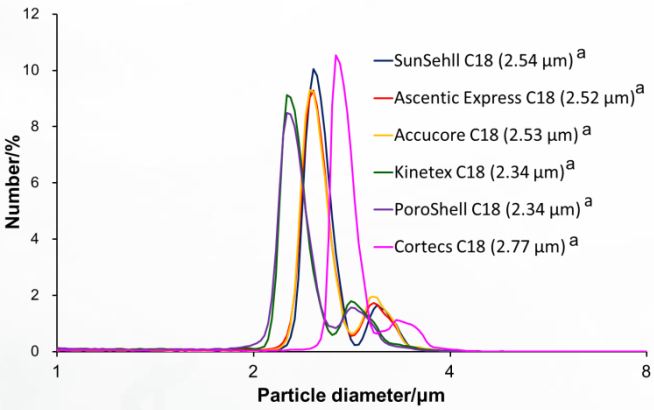


Column: SunShell C18, 2.6 µm 50 x 2.1 mm
Mobile phase: A) 10 mM Ammonium bicarbonate pH 9.5
B) Acetonitrile
Gradient program:

Time (min)	0	1	3	3.1	5
%B	30	90	90	30	30

Flow rate: 0.5 mL/min
Temperature: 40 ºC
Detection: UV@250 nm
Sample: 1=Uracil, 2=Propranolol, 3= Nortriptyline, 4=Amitriptyline

◆Comparison of particle size

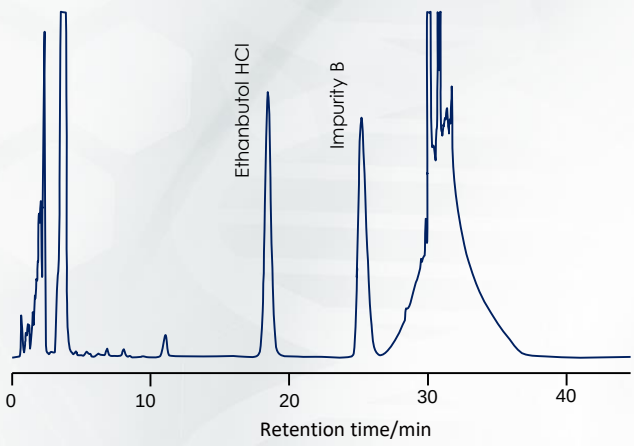


*Measured using Beckman Coulter Multisizer 3 after C18 materials were sintered at 600 degree Celsius for 8 hours. The measured value of each sintered core shell silica is considered to be different from that of the original core shell silica.

a. Median particle size

SunShell

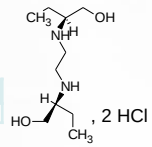
Ethambutol Hydrochloride



Column: SunShell C18, 2.6 µm 100 x 4.6 mm
Mobile phase: A) Methanol/water (50/50 V/V)
B) Methanol

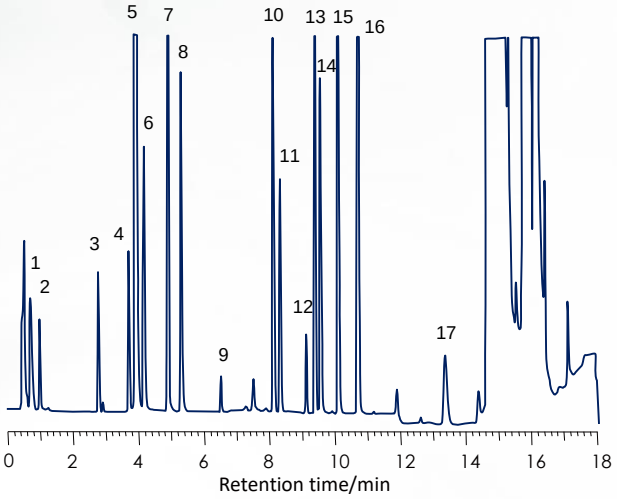
Time (min)	0	30	35	37	38
%B	29	29	100	100	29

Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV 215 nm
Injection volume: 10 µL



Ethambutol Hydrochloride

Amino Acids derivatized with OPA and FMOC

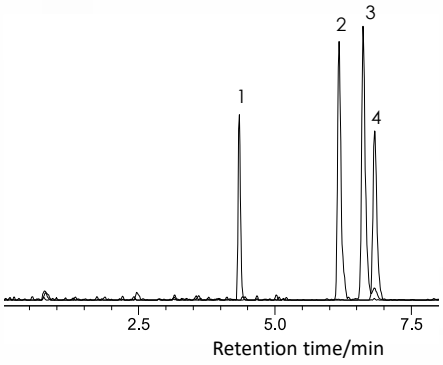


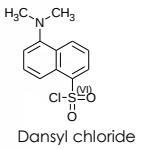
Column: SunShell C18 2.6 µm, 150 x 2.1 mm
Mobile phase: A) 10mM Na_2PO_4 + 10mM $Na_2B_4O_7$ + 0.5mM NaN_3 (pH7.8)
B) Acetonitrile/Methanol/Water (45/45/10 %V)

Time (min)	0	0.4	12.8	13.8
%B	5	5	50	100

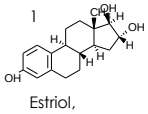
Flow rate: 0.61 mL/min
Temperature: 40 °C
Detection: UV@338 nm
Sample: 1=Aspartic acid, 2=Glutaminc acid, 3=Serine, 4=Histidine, 5=Glycine, 6=Threonine, 7=Arginine, 8=Alanine, 9=Tyrosine, 10=Valine, 11=Methionine, 12=Tryptophan, 13=Pnehylalanine, 14=Isoleucine, 15=Leucine, 16=Lysine, 17=Proline

Dansylated estrogen hormones

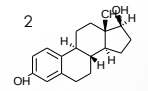




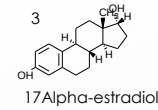
Dansyl chloride



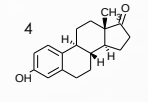
Estriol,



17Beta-estradiol,



17Alpha-estradiol



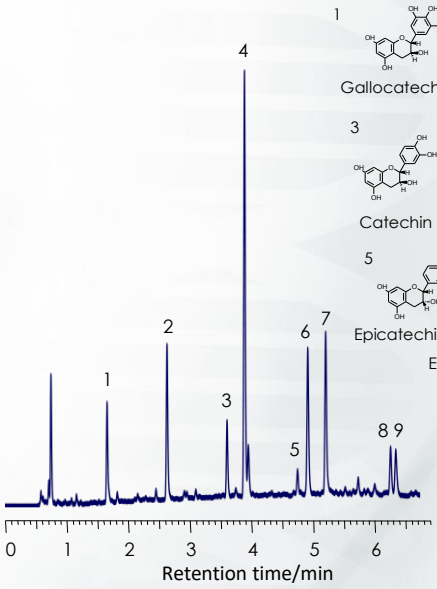
Estrone

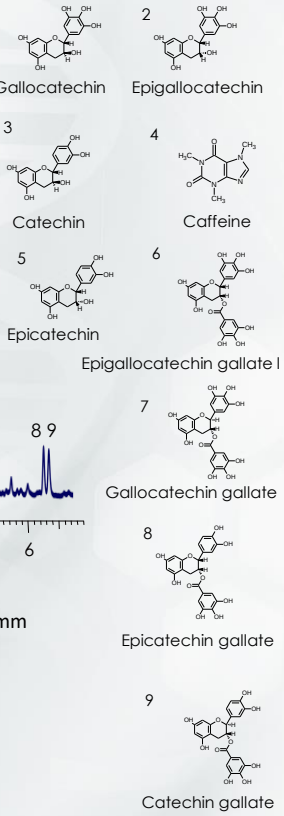
Column: SunShell C18 2.6 µm, 100 x 2.1 mm
Mobile phase:
A) H_2O with 0.1% formic acid.
B) CH_3CN with 0.1% formic acid.
Gradient program:
0 - 0.5 min: 10% B
0.51 – 3.0 min: 10 - 72% B
3.01 - 6.0 min: 72% B
6.01 - 7.0 min: 72 - 100% B
7.01 - 10.0 min: 100% B

Flow rate: 0.45 mL/min.
Temperature: 40 °C
Detection: MS(sim), m/z, 522.20, 506.20, 504.20
Samples: 1. Dansylated estriol, 2. Dansylated 17beta-estradiol,
3. Dansylated 17alpha-estradiol , 4. Dansylated estrone

Courtesy of Department of Chemistry & Biochemistry, The University of Texas at Arlington

Oolong tea





Column: SunShell C18 2.6 µm, 75 x 4.6 mm
Mobile phase:
A) 0.1% Phosphoric acid
B) CH_3CN
Gradient program

Flow rate: 1.0 mL/min,
Temperature: 25 °C
Detection: UV@250 nm
Sample: Oolong tea

SunShell C18-WP, RP-AQUA, C8, Phenyl, PFP, 2.6 µm

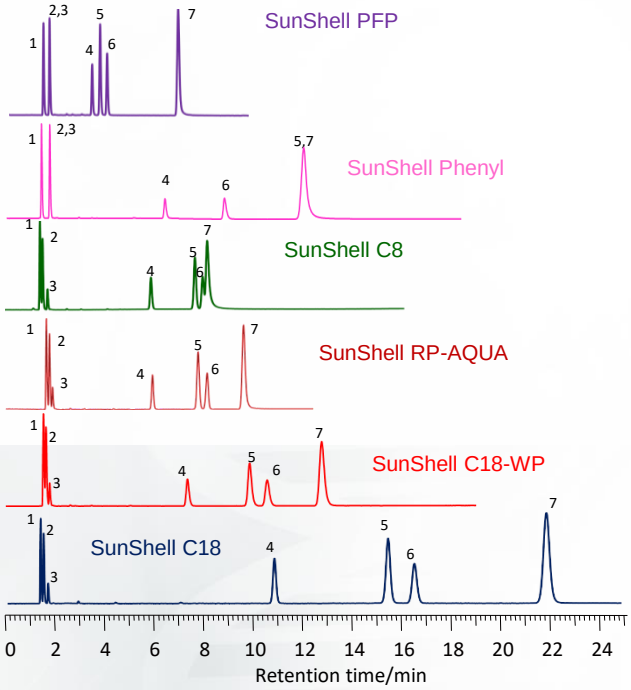
(Pentafluoropheny)

◆ Characteristics of SunShell

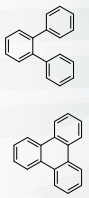
	Core shell silica			Bonding phase					
	Particle size	Pore diameter	Specific surface area	Carbon content	Bonded phase	USP L line	End-capping	Maximum operating pressure	Available pH range
SunShell C18	2.6 µm	9 nm	150 m^2/g	7%	C18	L1	Sunniest endcapping	60 MPa	1.5 - 10
SunShell C18-WP	2.6 µm	16 nm	90 m^2/g	5%	C18	L1	Sunniest endcapping	60 MPa	1.5 - 10
SunShell RP-AQUA	2.6 µm	16 nm	90 m^2/g	4%	C28	Equivalent to L62	Sunniest endcapping	60 MPa	2 - 8[a)]
SunShell C8	2.6 µm	9 nm	150 m^2/g	4.5%	C8	L7	Sunniest endcapping	60 MPa	1.5 - 9
SunShell Phenyl	2.6 µm	9 nm	150 m^2/g	5%	Phenylhexyl	L11	Sunniest endcapping	60 MPa	1.5 - 9
SunShell PFP	2.6 µm	9 nm	150 m^2/g	4.5%	Pentafluorophenyl	L43	TMS endcapping	60 MPa	2 - 8

a) Under 100% aqueous condition

◆ Comparison of standard samples

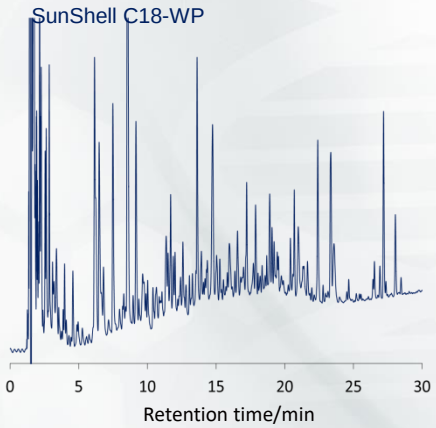


Column: SunShell C18, C18-WP, RP-AQUA, C8, Phenyl, PFP, 2.6 µm
150 x 4.6 mm
Mobile phase: CH_3OH/H_2O=75/25
Flow rate: 1.0 mL/min
Temperature: 40 °C
Sample: 1 = Uracil
2 = Caffeine
3 = Phenol
4 = Butylbenzene
5 = o-Terphenyl
6 = Amylbenzene
7 = Triphenylene



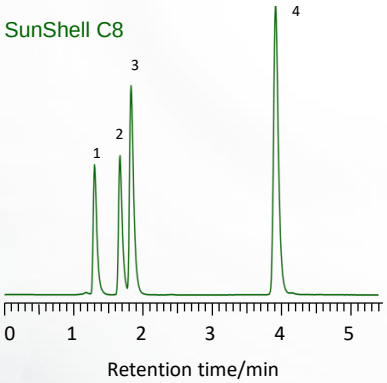
	Hydrogen bonding (Caffeine/Phenol)	Hydrophobicity (Amylbenzene/Butylbenzene)	Steric selectivity (Triphenylene/o-Terphenyl)
PFP	1.00	1.31	2.38
Phenyl	1.00	1.48	1.01
C8	0.32	1.46	1.08
RP-AQUA	0.52	1.52	1.30
C18-WP	0.40	1.55	1.35
SunShell C18	0.39	1.60	1.46

Separation of peptides



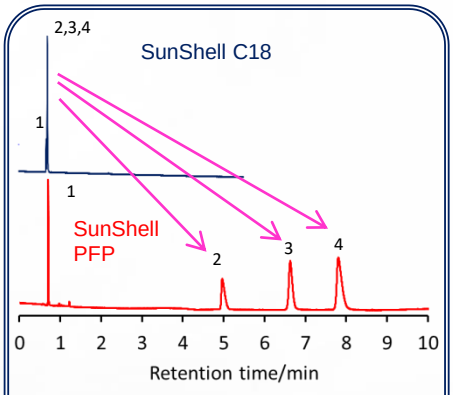
Column: SunShell C18-WP, 2.6 µm (16 nm) 150 x 4.6 mm,
Mobile phase: A) 0.1% TFA in Acetonitrile/water(10:90)
B) 0.1 % TFA in Acetonitrile
Gradient program: %B 5% – 50% in 50 min
Flow rate: 1.0 mL/min , Temperature: 25 °C,
Detection: UV@210 nm,
Sample: Tryptic digest of BSA

Separation of amitriptyline using C8



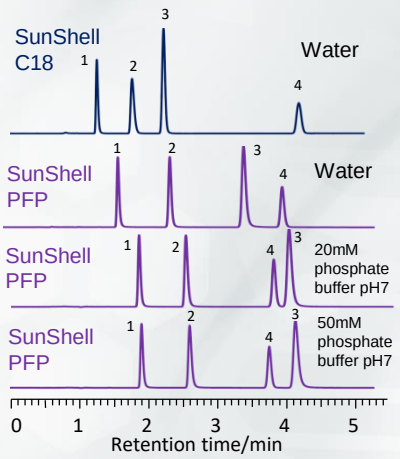
Column: SunShell C8, 2.6 µm 150 x 4.6 mm
Mobile phase:
CH_3CN/20mM phosphate buffer H7.0=60/40
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@250nm
Sample: 1 = Uracil, 2 = Propranolol,
3 = Nortriptyline, 4 = Amitriptyline

Separation of basic compounds



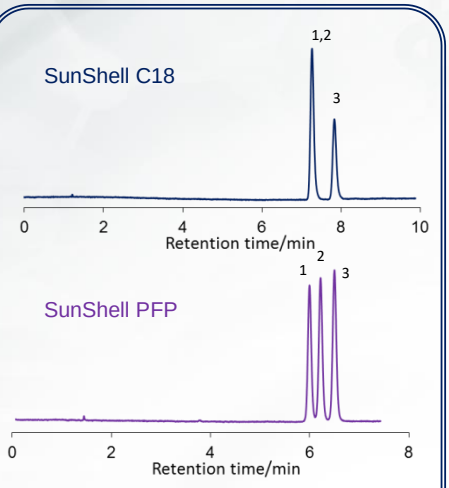
Column: SunShell C18, 2.6 µm 150 x 4.6 mm
SunShell PFP, 2.6 µm 150 x 4.6 mm
Mobile phase:
CH_3CN/10mM phosphate buffer pH7.0=80/20
Flow rate: 1.8 mL/min
Temperature: 25 °C
Sample: 1 = Uracil, 2 = Propranolol,
3 = Nortriptyline, 4 =Amitriptyline

Separation of xanthines



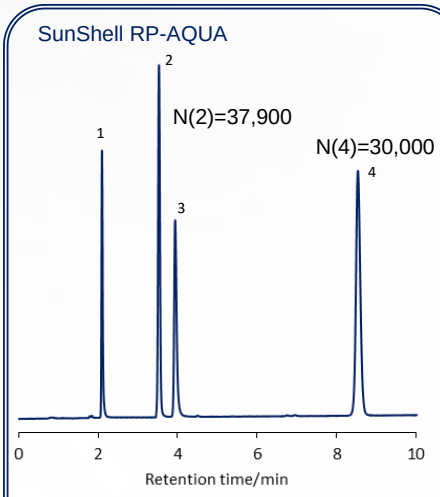
Column: SunShell C18, PFP, 2.6 µm 150 x 2.1 mm
Mobile phase: CH_3OH/water or buffer=30/70
Flow rate: 0.3 mL/min
Temperature: 25 °C
Detection: UV@250nm
Sample: 1 = Theobromine
2 = Theophyline
3 = Caffeine
4 = Phenol

Separation of cresol isomers



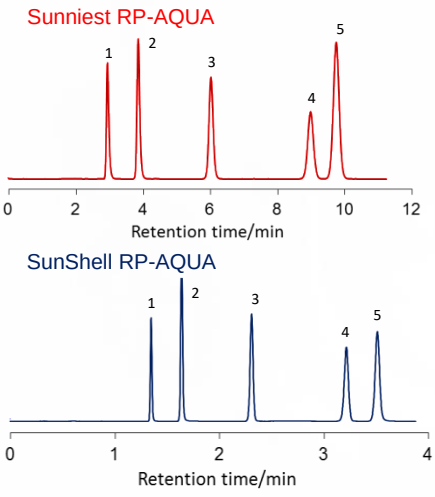
Column: SunShell C18, PFP, 2.6 µm 150 x 4.6 mm
Mobile phase: CH_3OH/H_2O=40/60
Flow rate: 1.0 mL/min
Temperature: 25 °C
Detection: UV@250nm
Sample: 1 = p-Cresol
2 = m-Cresol
3 = o-Cresol

Separation of nucleotides



Column:
SunShell RP-AQUA, 2.6 µm 150 x 4.6 mm
Mobile phase: 20mM Phosphate buffer pH6.0
Flow rate: 1.0 mL/min
Temperature: 25 °C
Detection: UV@250nm
Sample: 1 = 5'-GDP
2 = 5'-ATP
3 = 5'-ADP
4 = 5'-AMP

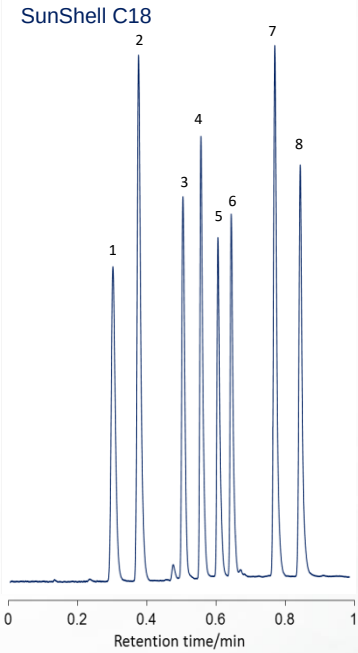
Separation of nucleic acid bases



Column:
Sunniest RP-AQUA, 5 µm 150 x 4.6 mm
SunShell RP-AQUA, 2.6 µm 150 x 4.6 mm
Mobile phase:
10mM Phosphate buffer pH7.0
Flow rate: 1.0 mL/min for Sunniest
1.5 ml/min for SunShell
Temperature: 24 °C
Sample: 1 = Cytosine, 2 = Uracil, 3 = Thymidine,
4 = Uridine, 5 = Thymine

	Plate(5)	Resolution (4,5)
Sunniest	14,000	1.98
SunShell	30,000	3.79

High-throughput separation

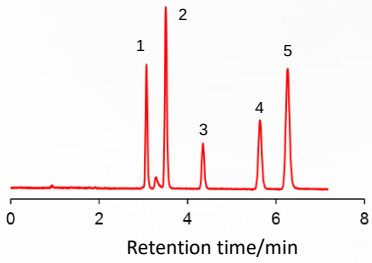


Column: SunShell C18, 30 x 3.0 mm.
Mobile phase: A) Water, B) Acetonitrile; Gradient (Acetonitrile %), 0.00 min - 35%, 0.40 min - 100%, 0.80 min - 100%, 0.85 min - 35%, 1cycle; 1.8min, (High-pressure gradient).
Flow rate: 1.0 mL/min.
Temperature: 40 °C.
Injection Volume: 1 µL.
Wavelength: 200 - 500nm, CH-9, 215 - 500nm (Max Abs.).
Sample: Mixture of ultraviolet absorbers,
1 = 2,2',4,4'-Tetrahydroxybenzophenone,
2 = Ethyl *p*-aminobenzoate,
3 = 2, 4-Dihydroxybenzophenone,
4 = 2,2'-Dihydroxy-4-methoxybenzophenone,
5 = 2,2'-Dihydroxy-4,4'-dimethoxybenzophenone,
6 = 2-Hydroxy-4-methoxybenzophenone,
7 = 2-(2'-Hydroxy-5'-metylphenyl) benzotriazole,
8 = 4-tert-Butylphenyl salicylate.
Courtesy of Jasco.

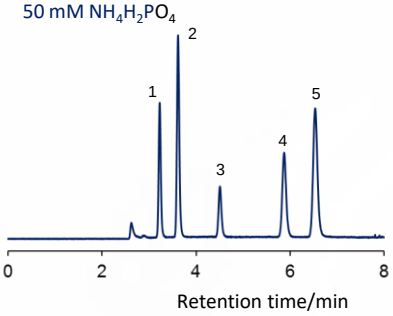
A peak width is just one second!!

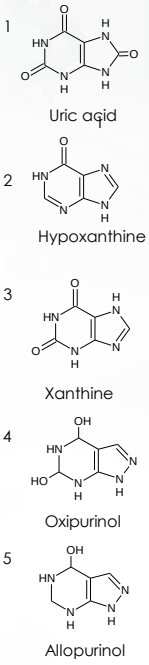
Purine analogue

10 mM ammonium acetate (pH 4.7)



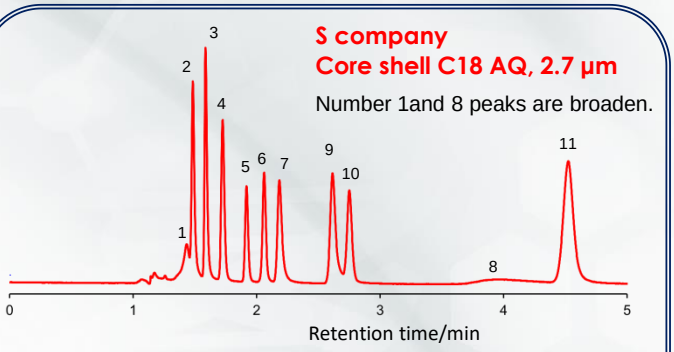
50 mM $NH_4H_2PO_4$

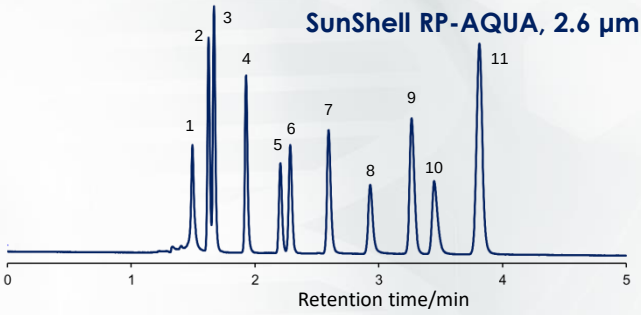




Column: SunShell RP-AQUA, 2.6 µm 100 x 4.6 mm
Mobile phase: 50 mM $NH_4H_2PO_4$ or 10 mM ammonium acetate (pH 4.7)
Flow rate: 1.0 mL/min
Temperature: Ambient
Detection: UV@250 nm
Sample: 1 = Uric acid, 2 = Hypoxanthine, 3 = Xanthine, 4 = Oxipurinol, 5 = Allopurinol

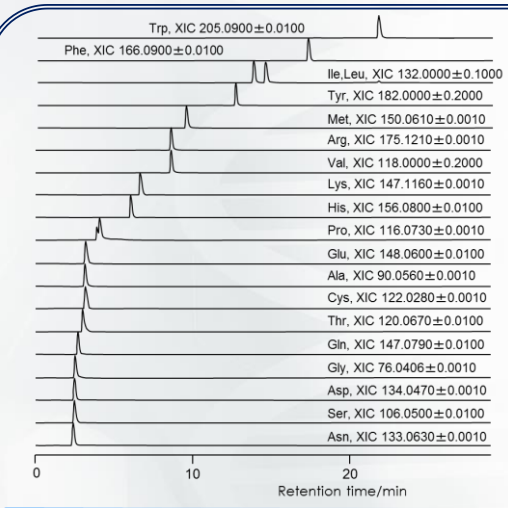
Organic acid



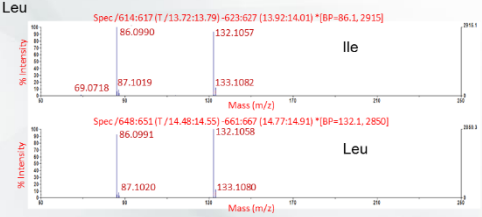


Column dimension: 150 x 4.6 mm
Mobile phase: 0.1% H_3PO_4
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@210nm
Sample:
1 = Oxalic acid, 2 = Tartaric acid, 3 = Formic acid, 4 = Malic acid,
5 = Lactic acid, 6 = Acetic acid, 7 = Diglycolic acid, 8 = Maleic acid,
9 = Citric acid, 10 = Succinic acid, 11 = Fumaric acid.

Amino acids (LC/MS)

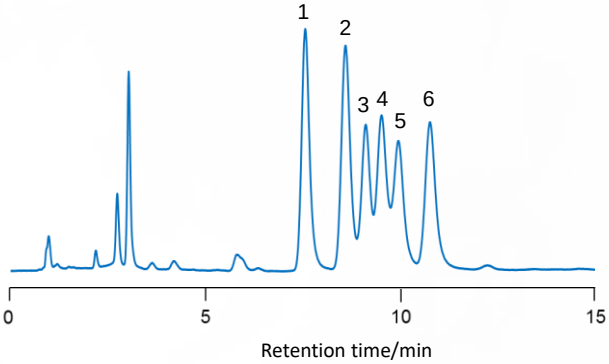


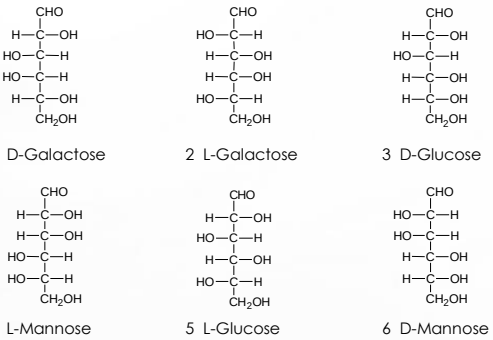
Mass spectra of Ile and Leu



Column: SunShell RP-AQUA, 2.6 µm, 150 x 2.1 mm
Mobile phase: A) 5 mM HFBA, B) 5 mM HFBA in CH_3CN / H_2O (9/1)
%B 0% to 20% in 20 min (HFBA: Heptafluorobutyric acid)
Flow rate: 0.2 mL / min
Temperature: 40 °C
Detection: MS (NanoFrontier LD) ESI Positive,
Extracted ion chromatogram (EIC)
Courtesy of Dr Takeo Kaneko, Hokohama National University

Monosaccharides derivatized with L-Tryptphan

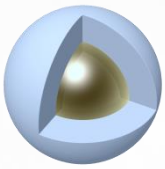




Column: SunShell RP-AQUA, 2.6 µm 100 x 4.6 mm
Mobile phase: 5 mM Phosphate and 25 mM tetraborate (pH 9.6)
Flow rate: 1.0 mL/min
Temperature: 20 °C
Detection: UV@220 nm
Sample: Monosaccharides derivatized with L-Tryptphan

Courtesy of Dr Shuji Kodama, Tokai University

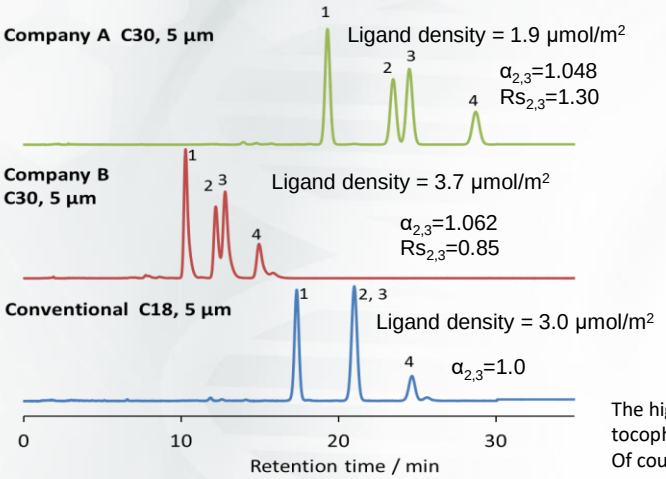
SunShell C30, 2.6 µm



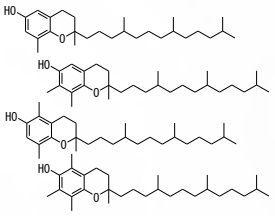
Specification of SunShell C30

	Core shell silica				Bonding phase					
	Particle size (µm)	Core size (µm)	Pore size (nm)	Specific surface area (m^2/g)	Carbon loading (%)	Ligand	USP L category	End-capping	Maximum pressure	pH range
SunShell C30	2.6	1.6	12	95	7	C30	L62	TMS	60 MPa	1.5 - 9

Problem of C30 column

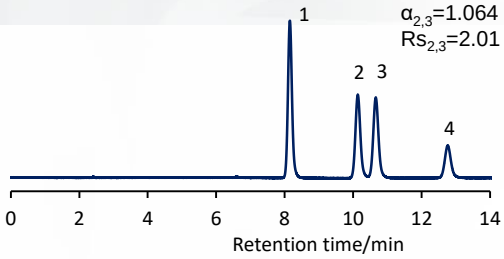


Column dimension: 250 x 4.6 mm
Mobile phase: methanol/water = 97/3
Flow rate: 1.0 mL/min
Temperature: 30 °C
Detection: UV@295 nm
Sample,
1 = δ-tocopherol
2 = γ-tocopherol
3 = β-tocopherol
4 = α-tocopherol



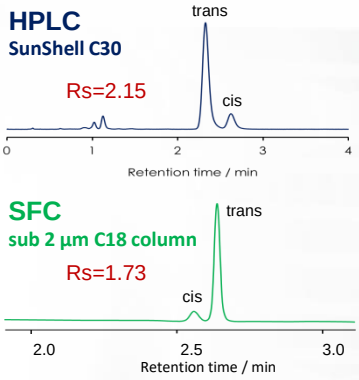
The higher a ligand density, the larger a separation factor of β-tocopherol and γ-tocopherol. Too high ligand density causes low theoretical plate and large tailing of a peak. Of course C18 columns can't separate β-tocopherol and γ-tocopherol.

Separation of tocopherols



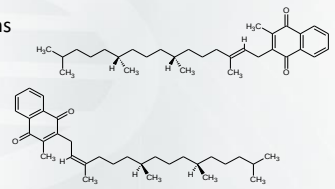
Column: SunShell C30, 2.6 µm 150 x 3.0 mm
Mobile phase: Methanol/water = 97/3
Flow rate: 0.43 mL/min
Temperature: 25 °C
Detection: UV@295 nm
Sample: 1 = δ-tocopherol, 2 = γ-tocopherol, 3 = β-tocopherol, 4 = α-tocopherol

Fast separation of vitamin K1 isomers



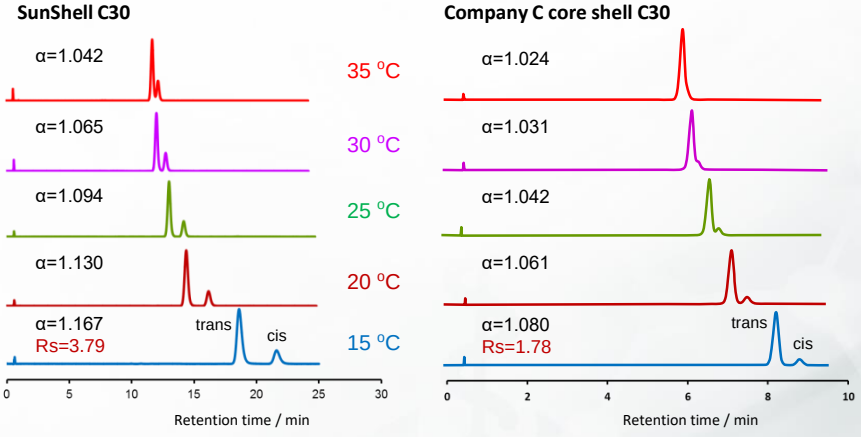
HPLC condition
Column: SunShell C30, 2.6 µm 50 x 3.0 mm
Mobile phase: Methanol
Flow rate: 0.80 mL/min
Temperature: 15 °C
Detection: UV@250 nm
Sample: Vitamin K1 isomers (trans and cis)

trans
cis



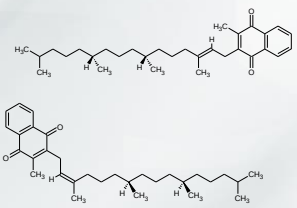
Both HPLC and SFC separations were finished within 3 minute.
Regarding resolution of isomers, HPLC won SFC.

Comparison of isomers separation of Vitamin k1



Column: SunShell C30, 2.6 µm 100 x 2.1 mm
Company C core shell C30, 2.6 µm 100 x 2.1 mm
Mobile phase: methanol/water = 96/4
Flow rate: 0.35 mL/min
Detection: UV@250 nm
Sample: vitamin K1 isomers (trans and cis).

trans
cis



SunShell 2.6 µm C18-WP, HFC18-16, HFC18-30, C8-30, C8-30HT, C4-30, C4-100

For separation of peptides and proteins

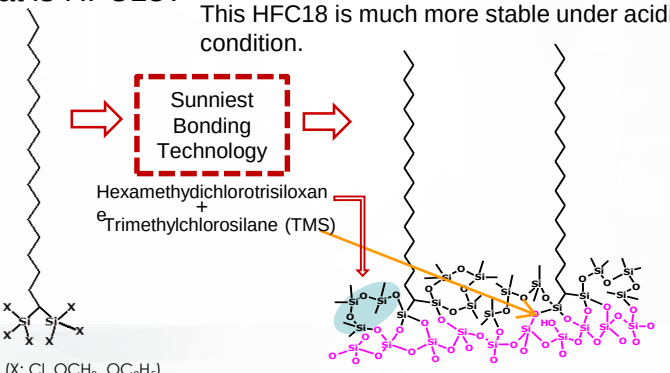
Characteristics of SunShell

	Core shell silica			Bonding phase						
	Particle size	Pore diameter	Specific surface area	Stationary phase	Carbon content	Ligand density	End-capping	Maximum operating pressure	Available pH range	USP L line
SunShell C18-WP	2.6 µm	16 nm	90 m^2/g	C18	5 %	2.5 µmol/m^2	Sunniest endcapping	60 MPa or 8,570 psi	1.5 - 10	L1
SunShell HFC18-16	2.6 µm	16 nm	90 m^2/g	C18	2.5%	1.2 µmol/m^2	Sunniest endcapping	60 MPa or 8,570 psi	1.5 – 9	L1
SunShell HFC18-30	2.6 µm	30 nm	40 m^2/g	C18	1.3%	1.2 µmol/m^2	Sunniest endcapping	60 MPa[a] or 8,570 psi[a]	1.5 - 9	L1
SunShell C8-30	2.6 µm	30 nm	40 m^2/g	C8	1.2%	2.5 µmol/m^2	Sunniest endcapping	60 MPa[a] or 8,570 psi[a]	1.5 – 9	L7
SunShell C8-30HT	3.4 µm	30 nm	15 m^2/g	C8	0.5%	2.5 µmol/m^2	Sunniest endcapping	60 MPa[a] or 8,570 psi[a]	1.5 – 9	L7
SunShell C4-30	2.6 µm	30 nm	40 m^2/g	C4	0.9%	3 µmol/m^2	Sunniest endcapping	60 MPa[a] or 8,570 psi[a]	1.5 – 8	L26
SunShell C4-100	2.6 µm	100 nm	22 m^2/g	C4	0.6%	3 µmol/m^2	Sunniest endcapping	60 MPa[a] or 8,570 psi[a]	1.5 – 8	L26

a: 50MPa, 7141psi for 4.6 mm i.d. column

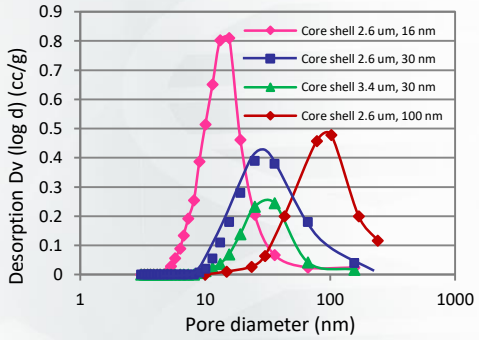
What is HFC18?

Hexa-Functional C18 has six functional groups. This HFC18 is much more stable under acidic condition.

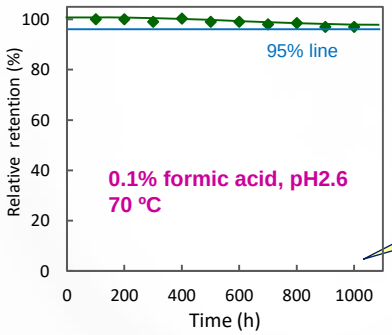


Schematic diagram of reagent

Schematic diagram of the state of bonding on silica surface



Pore distribution of core shell particle



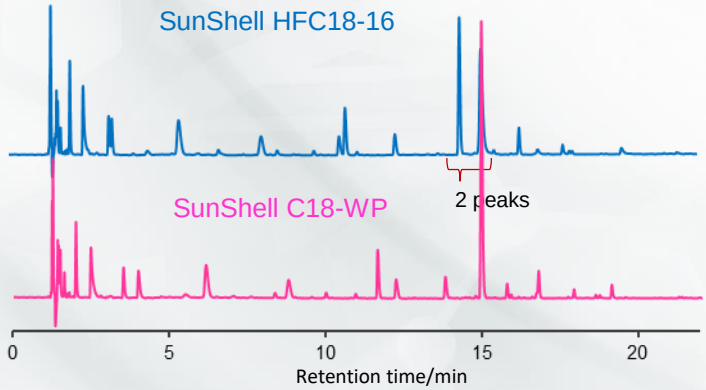
Durable test condition
Column : SunShell HFC18-16 2.6 µm, 50 x 2.1 mm
Mobile phase:
CH_3CN/0.1% formic acid, pH2.6=40/60
Flow rate: 0.4 mL/min
Temperature: 70 ºC

More than 1000 hours

Measurement condition
Mobile phase: CH_3CN/H_2O=60/40
Flow rate: 0.4 mL/min
Temperature: 40 ºC
Sample: 1 = Uracil
2 = Butylbenzene

Stability under LC/MS mobile phase condition

Separation of peptides



Column: SunShell HFC18-16, 2.6 µm (16 nm) 150 x 4.6 mm
SunShell C18-WP, 2.6 µm (16 nm) 150 x 4.6 mm
Mobile phase: A) 0.1% TFA in Acetonitrile/water(10:90)
B) 0.1 % TFA in Acetonitrile

Gradient program:

Time	0 min	5 min	40 min
%B	5%	5%	50%

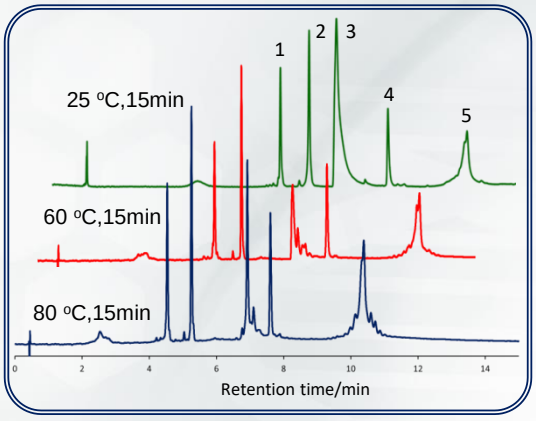
Flow rate: 1.0 mL/min
Temperature: 25 ºC
Detection: UV@210 nm
Sample: Tryptic digest of cytochrome C

ChromaNik Technologies Inc.

SunShell 2.6 µm C8-30, C8-30HT, C4-30, C4-100

For separation of peptides and proteins

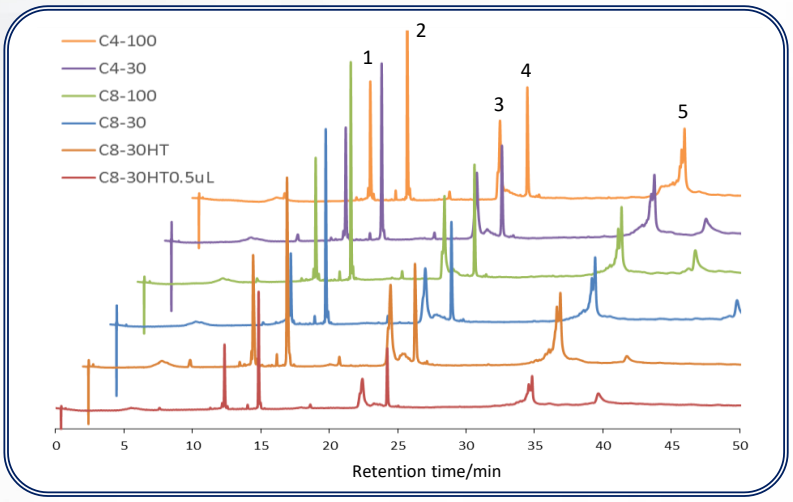
Comparison of column temperature



Column: SunShell C8-30, 2.6 µm (30 nm) 100 x 2.1 mm
Mobile phase: A) 0.1% TFA in water
B) 0.08 % TFA in acetonitrile
Gradient program: Time 0 min 15 min
%B 20% 65%
Flow rate: 0.5 mL/min ,
Temperature: 25 °C 60 °C or 80 °C
Detection: UV@215 nm,
Sample:1 = Cytochrome C, 2 = Lysozyme, 3 = BSA,
4 = Myoglobin, 5 = Ovalbumin

A macromolecule compound like a protein diffuses very slowly, so that an elevated temperature makes a peak be shaper and improves separation. BSA peak seemed to be tailing at 25 degree Celsius. BSA, however, was separated several peaks at 80 degree Celsius.

Comparison of SunShell stationary phase



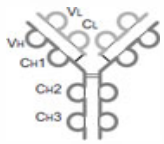
Column dimension: 100 x 2.1 mm,
Mobile phase: A) 0.1% TFA in water, B) 0.1 % TFA in Acetonitrile
Gradient program: Time 0 min 60 min
%B 20% 65%
Flow rate: 0.5 mL/min, Temperature: 80 °C, Detection: UV@215 nm, Injection volume: 1.0 µL
Sample:1 = Cytochrome C, 2 = Lysozyme, 3 = BSA, 4 = Myoglobin, 5 = Ovalbumin,
UHPLC instrument: HITACIHI Chromaster

Comparison of peak width (W0.5, min)

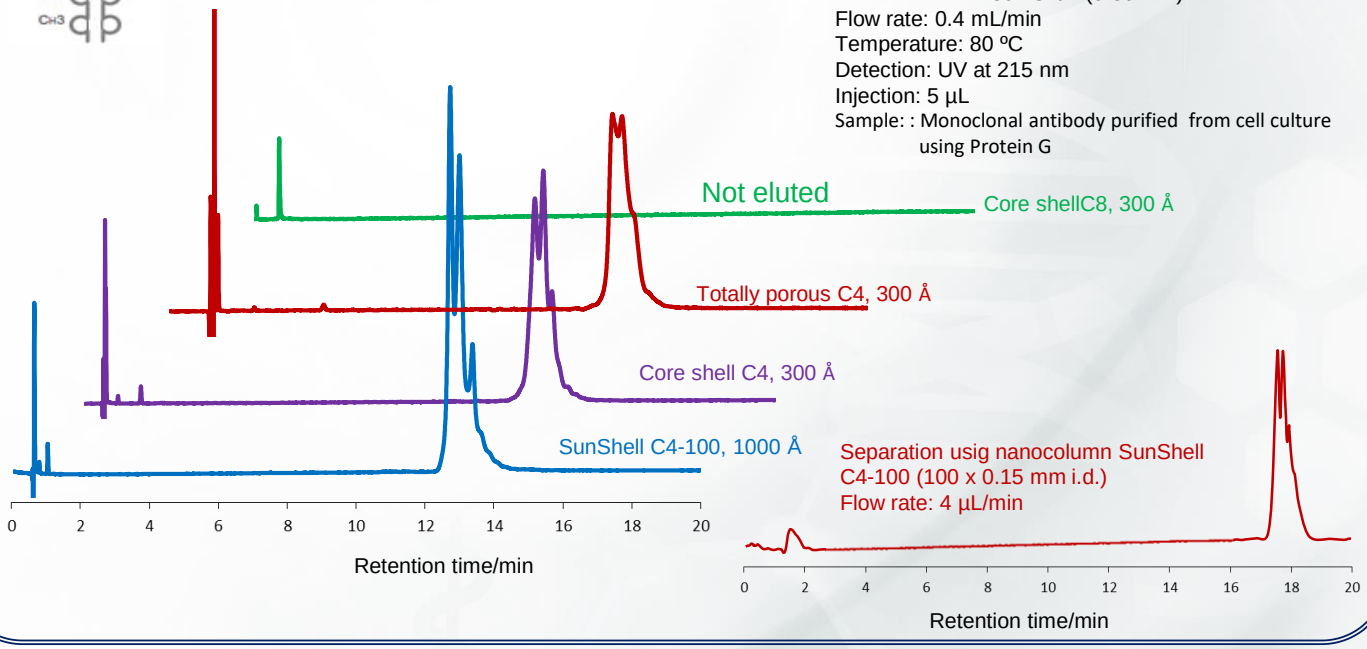
	C4-100	C4-30	C8-100	C8-30	C8-30HT	C8-30HT 0.5µL	Sample concentration
Cytochrome C	0.167	0.177	0.160	0.155	0.212	0.144	0.050%
Lysozyme	0.164	0.180	0.153	0.166	0.196	0.145	0.050%
BSA	0.308	0.410	0.276	0.514	0.422	0.330	0.100%
Myoglobin	0.197	0.221	0.180	0.199	0.238	0.176	0.050%
Ovalbmin	0.391	0.889	0.247	0.428	0.184	0.176	0.050%

The above table indicated that C4-100 with 1000Å of pore showed a sharper peak than the other. C8-30HT has a thin porous layer and low surface area, so that low sample loadnig made a peak sharper.

Separation of monoclonal antibody



Column dimension: 100 x 2.1 mm i.d.
Mobile phase: A) water/TFA (100/0.1)
B) acetonitrile/TFA (100/0.085)
30-45%B (0-30 min)
Flow rate: 0.4 mL/min
Temperature: 80 °C
Detection: UV at 215 nm
Injection: 5 µL
Sample: : Monoclonal antibody purified from cell culture using Protein G



Regarding reversed phase separation of monoclonal antibody (IgG), not only core shell C4 with 30 nm pore showed the better separation than totally porous C4, but also 100 nm of pore leaded the best separation. Nano column showed almost the same separation of IgG as semi-micro column.

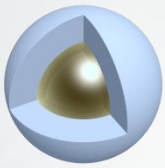
SunShell 2-EP, 2.6 µm

For Supercritical fluid Chromatography

2.6 µm core shell column shows only one third of back pressure to compare with 1.7 µm fully porous column although both show almost same efficiency. By such low back pressure, a difference of density of supercritical fluid between an inlet and an outlet of the column is reduced. Consequently, . 2.6 µm core shell column performs a superior separation for SFC.

Characteristics of SunShell 2-EP

	Core shell silica							
	Particle size	Pore diameter	Specific surface area	Carbon content	Bonded phase	End-capping	Maximum operating pressure	Available pH range
SunShell 2-EP	2.6 µm	9 nm	150 m^2/g	2.5%	2-Ethylpyridine	no	60 MPa or 8,570 psi	2 – 7.5



Comparison between SunShell 2-EP and 1.7 µm fully porous 2-EP

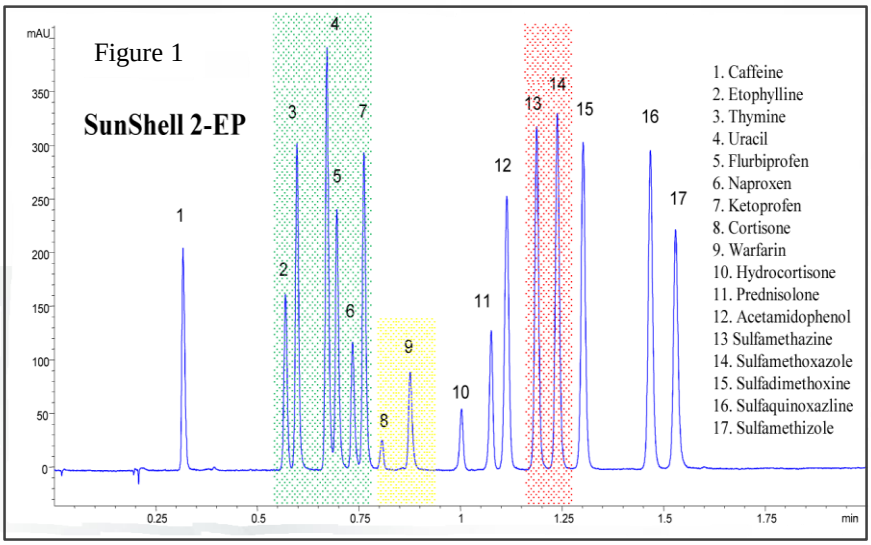


Figure 1: Chromatogram of the separation for he 17-component mix using the Sun Shell 2-EP 150 x 3.0 mm column. A methanol gradient of < 2 minutes was used on the Agilent 1260 Infinity SFC system. SFC conditions: flow rate: 4.0mL/min; outlet pressure 160 bar; column temperature 55ºC. Gradient program: 5.0-7.5% in 0.20 min, then 7.5-20% in 1.3 min and held at 20% for 0.2 min.

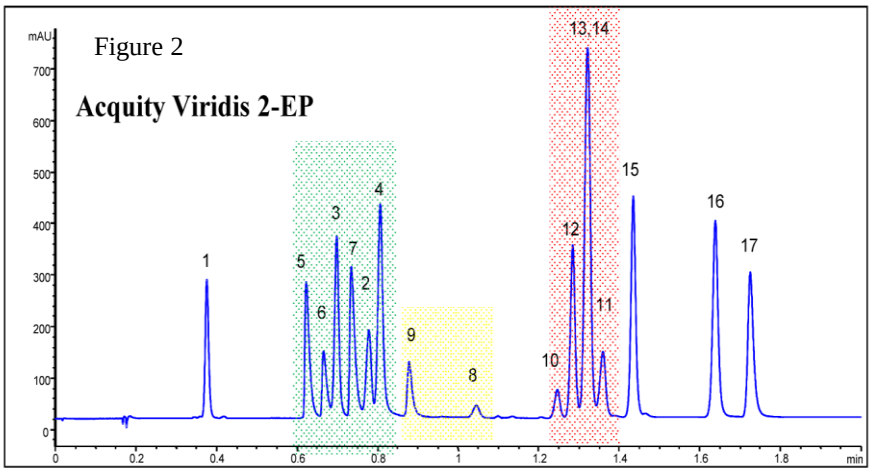
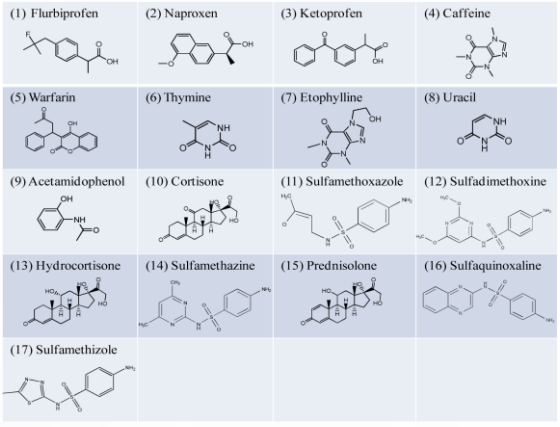


Figure 2: Chromatogram of the separation for the 17-component mix using Acquity UPC² Viridis 2-EP 100 x 3.0 mm column. 16 of the 17 components were resolved. A methanol gradient of < 2 minutes was used on the Agilent 1260 Infinity SFC system. SFC conditions: flow rate 3.5 mL/min; outlet pressure 160 bar; and column temperature 70ºC. Gradient program: 5.0-12.5% in 1.0 min, 12.5% for 0.25 min, then 12.5-20% in 0.75 min.



Courtesy of Pfizer Inc.

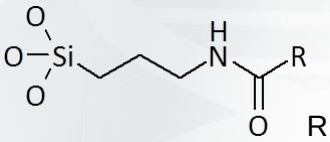
SunShell HILIC-Amide, HILIC-S, 2.6 µm

For Hydrophilic Interaction Chromatography

Characteristics of SunShell HILIC-Amide

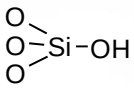
		Core shell silica			Amide (USP L68)					
	Particle size	Core size	Pore diameter	Specific surface area	Carbon content	Bonded phase	End-capping	Maximum operating pressure	USP category	Available pH range
SunShell HILIC-Amide	2.6 µm	1.6 µm	9 nm	150 m^2/g	3%	Amide	no	60 MPa or 8,570 psi	L63	2 - 8
SunShell HILIC-S	2.6 µm	1.6 µm	9 nm	150 m^2/g	0%	Bare silica	no	60 MPa or 8,570 psi	L3	1 - 5

Stationary phase of HILIC-Amide

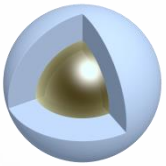


R: Hydrophilic group

Stationary phase of HILIC-S

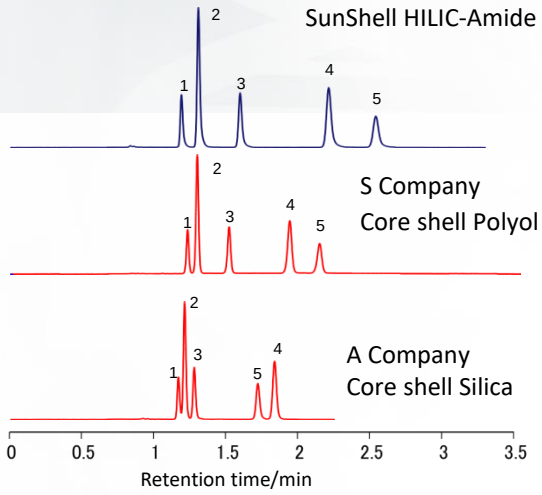


Bare silica



Stationary phase of SunShell HILIC-Amide consists of AMIDE and HYDROPHILIC GROUP, so that this stationary phase is more polar than an individual group. High speed separation is leaded by core shell structure that derives high efficiency and fast equilibration. HILIC-S is recommended for separation using LC/MS.

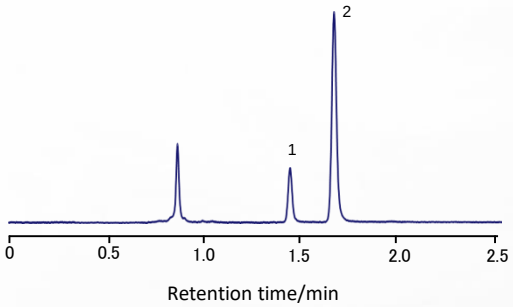
Separation of Nucleic acid bases: Comparison of the other core shell hilic columns



Column:
SunShell HILIC-Amide, 2.6 µm 100 x 4.6 mm,
Coreshell polyol, 2.7 µm 100 x 4.6 mm,
Core shell Silica, 2.7 µm 100 x 4.6 mm
Mobile phase:
Acetonitrile/20 mM ammonium acetate(pH4.7) = 8/2
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@250 nm
Sample: 1 = Thymine, 2 = Uracil, 3 = Uridine, 4 = Cytosine, 5 = Cytidine

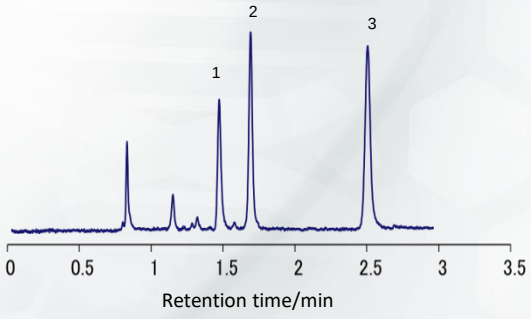
Regarding retention of cytidine, SunShell HILIC-Amide showed 30% higher retention factor than S core shell polyol.

Separation of Cyanuric acid and Melamine



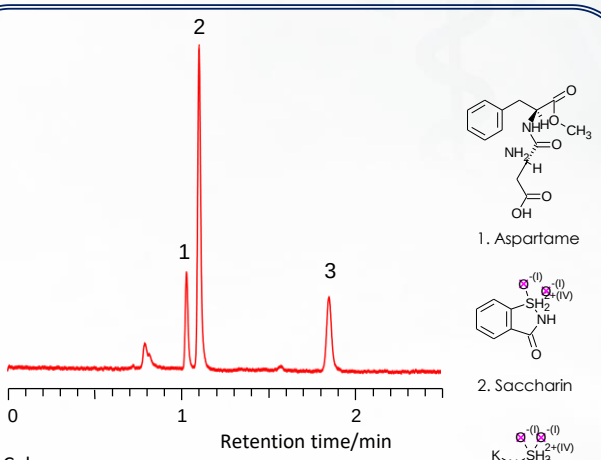
Column: SunShell HILIC-Amide, 2.6 µm 100 x 4.6 mm
Mobile phase:
Acetonitrile/5 mM phosphate Buffer (pH6.9) =75/25
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@220 nm,
Sample: 1 = Cyanuric acid, 2 = Melamine

Separation of water- soluble vitamins



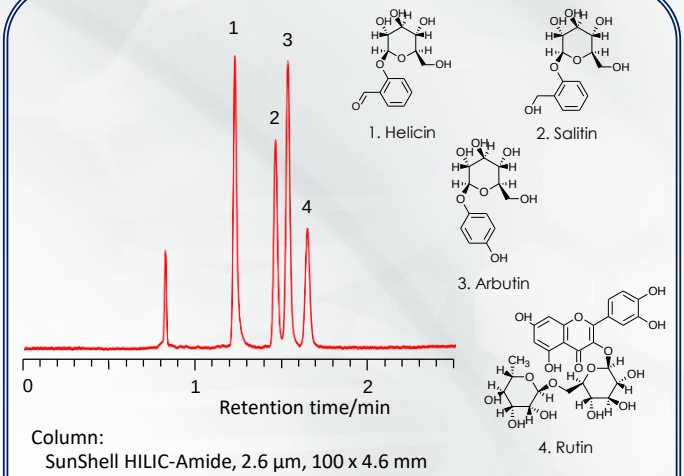
Column: SunShell HILIC-Amide, 2.6 µm 100 x 4.6 mm
Mobile phase:
Acetonitrile/25 mM phosphate buffer (pH2.5) =8/2
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@250 nm,
Sample: 1 = Nicotinic acid, 2 = Ascorbic acid, 3 = Pyridoxine

Artificial sweeteners



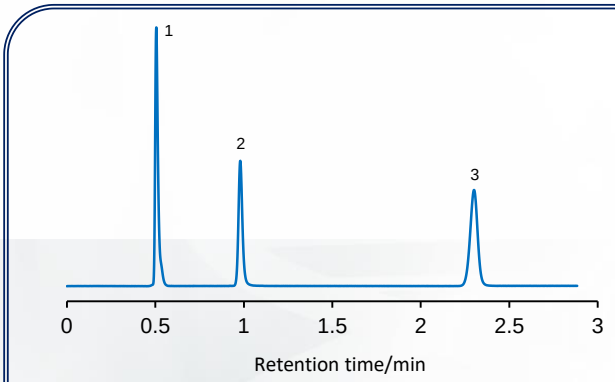
Column:
SunShell HILIC-Amide, 2.6 µm, 100 x 4.6 mm
Mobile phase:
Acetonitrile: 25 mM phosphate buffer (pH2.5) =8:2
Flow rate: 1.0 mL/min ,
Temperature: Ambient
Detection: UV@215 nm
Sample: 1 = Aspartame, 2 = Saccharin, 3 = Acesulfame K

Glycoside



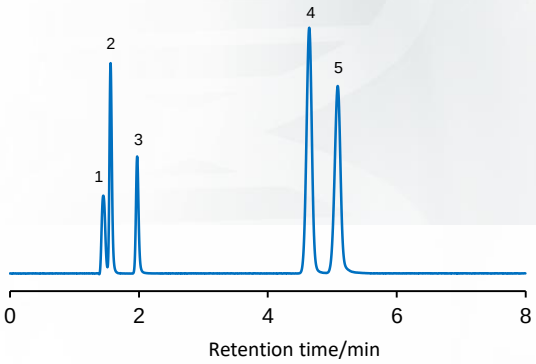
Column:
SunShell HILIC-Amide, 2.6 µm, 100 x 4.6 mm
Mobile phase:
Acetonitrile:25 mM phosphate Ammonium (pH4.9) =8:2
Flow rate: 1.0 mL/min
Temperature: Ambient
Detection: UV@215 nm
Sample: 1 = Helicin, 2 = Salicin, 3,= Arbutin, 4 = Rutin

Nucleic acid base



Column: SunShell HILIC-S, 2.6 µm 100 x 2.1 mm
Mobile phase: 100 mM ammonium acetate (pH3.0) /acetonitrile = 1/9
Flow rate: 0.4 mL/min
Temperature: 40 °C
Detection: UV@250 nm
Sample: 1 = Acenaphthene, 2 = Uridine, 3 = Cytosine

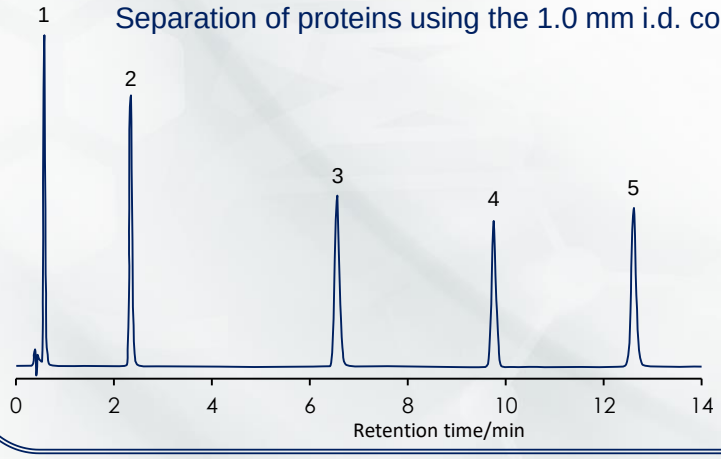
Nucleic acid bases



Column: SunShell HILIC-S, 2.6 µm 100 x 2.1 mm
Mobile phase: 100 mM ammonium acetate (pH3.0) /acetonitrile = 1/9
Flow rate: 0.2 mL/min
Temperature: 40 °C
Detection: UV@250 nm
Sample: 1 = Thymine, 2 = Uracil, 3 = Uridine, 4 = Cytosine, 5 = Cytidine

Core shell column with 1.0 mm i.d.

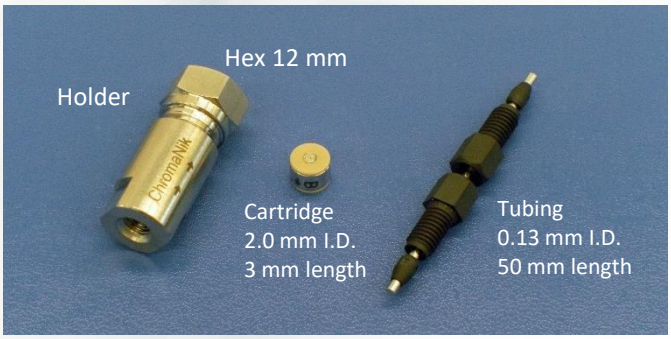
Separation of proteins using the 1.0 mm i.d. column



Column: SunShell RP-AQUA, 2.6 µm 100 x 1.0 mm
Mobile phase: A) 0.1 % trifluoroacetic acid (TFA) in water
B) 0.08 % trifluoroacetic acid (TFA) in acetonitrile
%B 10% to 30% in 25 min
Flow rate: 0.15 mL / min
Temperature: 60 °C
Detection: UV@214 nm
Sample: 1 = Gly-Tyr, 2 = Val-Tyr-Val, 3 = Met enkephalin,
4 = Leu enkephalin, 5 = Angiotensin II
(HPLC peptide standard mixture by Sigma-Aldrich)

SunShell Guard Cartridge Column

RP & S GUARD CARTRIDGE COLUMN



- The cartridge column is packed with SunShell C18 (RP) and Core shell silica (S) into a cartridge sized 3 x 2 mm i.d.
- RP guard cartridge is used for all reversed phases and S guard cartridge for hilic phases.
- Low dead volume structure
- Upper pressure limit is more than 60 Mpa
- Availablr for 2.1 mm i.d. to 4.6 mm i.d. columns

SunShell C18, 2.6 µm 50 x 2.1 mm

Without Guard Cartridge column

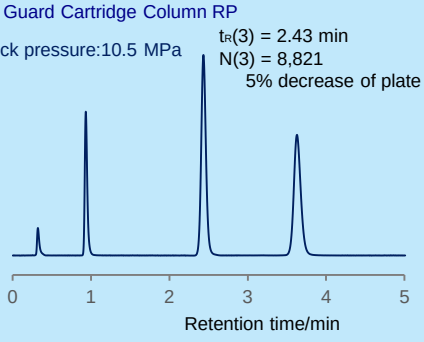
Back pressure:10.2 MPa

t_R(3)= 2.37 min
N(3) = 9,256

With Guard Cartridge Column RP

Back pressure:10.5 MPa

t_R(3) = 2.43 min
N(3) = 8,821
5% decrease of plate



Mobile phase:
CH_3CN/H_2O=60/40 for 2.1 mm i.d.
CH_3CN/H_2O=70/30 for 4.6 mm i.d.
Flow rate:
0.3 mL/min for 2.1 mm i.d.
1.8 mL/min for 4.6 mm i.d.
Temperature: 25 °C
Detection: UV@250nm
Sample: 1 = Uracil
2 = Ethylbenzoate
3 = Acenaphthene
4 = Butylbenzene

SunShell C18, 2.6 µm 150 x 4.6 mm

Without Guard Cartridge column

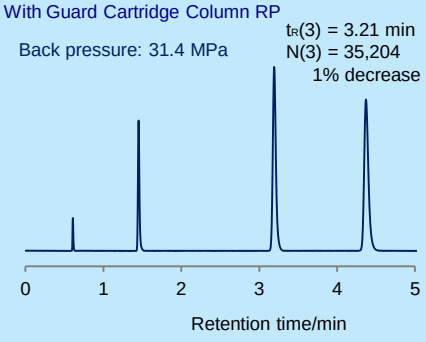
Back pressure: 28.8 MPa

t_R(3)= 3.20 min
N(3) = 35,632

With Guard Cartridge Column RP

Back pressure: 31.4 MPa

t_R(3) = 3.21 min
N(3) = 35,204
1% decrease of plate



Ordering Information of SunShell Guard Cartridge Column

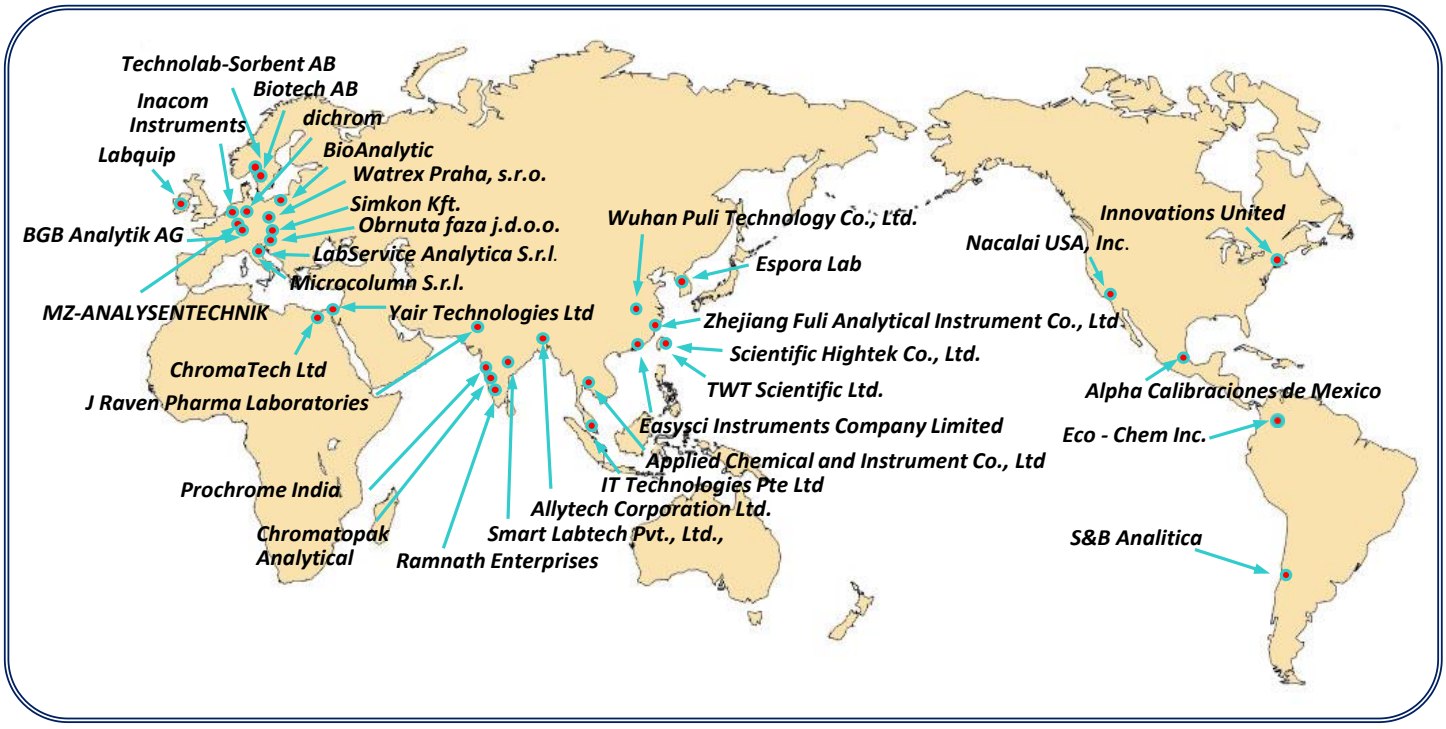
Description	Part number
SunShell Guard Cartridge RP Starter Kit (holder, cartridge, tubing)	CB32CK
SunShell Guard Cartridge RP for exchange (2 PCS)	CB32CC
SunShell Guard Cartridge S Starter Kit (holder, cartridge, tubing)	CS32CK
SunShell Guard Cartridge S for exchange (2 PCS)	CS32CC
SunShell Guard Cartridge holder	HOL2CC

Ordering information of SunShell

	Inner diameter (mm)	1.0	2.1	3.0	4.6	USP category
	Length (mm)	Catalog number	Catalog number	Catalog number	Catalog number	
SunShell C18, 2 μm	50	------	CB1941	------	------	
	100	------	CB1961	------	------	
	150	------	CB1971	------	------	
SunShell C18, 2.6 μm	30	------	CB6931	CB6331	CB6431	
	50	CB6141	CB6941	CB6341	CB6441	
	75	------	CB6951	CB6351	CB6451	L1
	100	CB6161	CB6961	CB6361	CB6461	
	150	CB6171	CB6971	CB6371	CB6471	
	250	------	------	CB6381	CB6481	
SunShell C18, 5 μm	150	------	------	CB3371	CB3471	
	250	------	------	CB3381	CB3481	
SunShell C8, 2.6 μm	30	------	CC6931	CC6331	CC6431	
	50	------	CC6941	CC6341	CC6441	
	75	------	CC6951	CC6351	CC6451	L7
	100	------	CC6961	CC6361	CC6461	
	150	------	CC6971	CC6371	CC6471	
SunShell PFP, 2.6 μm	30	------	CF6931	CF6331	CF6431	
	50	------	CF6941	CF6341	CF6441	
	75	------	CF6951	CF6351	CF6451	L43
	100	------	CF6961	CF6361	CF6461	
	150	------	CF6971	CF6371	CF6471	
SunShell C18-WP, 2.6 μm	30	------	CW6931	CW6331	CW6431	
	50	------	CW6941	CW6341	CW6441	
	75	------	CW6951	CW6351	CW6451	L1
	100	------	CW6961	CW6361	CW6461	
	150	------	CW6971	CW6371	CW6471	
SunShell RP-AQUA, 2.6 μm	30	------	CR6931	CR6331	CR6431	
	50	CR6141	CR6941	CR6341	CR6441	
	75	------	CR6951	CR6351	CR6451	Equivalent to L62
	100	CR6161	CR6961	CR6361	CR6461	
	150	CR6171	CR6971	CR6371	CR6471	
SunShell Phenyl, 2.6 μm	30	------	CP6931	CP6331	CP6431	
	50	------	CP6941	CP6341	CP6441	
	75	------	CP6951	CP6351	CP6451	L11
	100	------	CP6961	CP6361	CP6461	
	150	------	CP6971	CP6371	CP6471	
SunShell C30, 2.6 μm	30	------	CT6931	CT6331	------	
	50	------	CT6941	CT6341	------	
	75	------	CT6951	CT6351	------	L62
	100	------	CT6961	CT6361	------	
	150	------	CT6971	CT6371	------	
SunShell 2-EP, 2.6 μm	30	------	CE6931	CE6331	CE6431	
	50	------	CE6941	CE6341	CE6441	
	75	------	CE6951	CE6351	CE6451	
	100	------	CE6961	CE6361	CE6461	
	150	------	CE6971	CE6371	CE6471	
SunShell HILIC-Amide, 2.6 μm	30	------	CH6931	CH6331	CH6431	
	50	------	CH6941	CH6341	CH6441	
	75	------	CH6951	CH6351	CH6451	L68
	100	------	CH6961	CH6361	CH6461	
	150	------	CH6971	CH6371	CH6471	
SunShell HILIC-S, 2.6 μm	50	------	CU6941	------	------	
	100	------	CU6961	------	------	L3
	150	------	CU6971	------	------	

	Inner diameter (mm)	1.0	2.1	3.0	4.6	USP category
	Length (mm)	Catalog number	Catalog number	Catalog number	Catalog number	
SunShell HFC18-16, 2.6 µm	50	------	CG6941	CG6341	CG6441	L1
	100	------	CG6961	CG6361	CG6461	
	150	------	CG6971	CG6371	CG6471	
SunShell HFC18-30, 2.6 µm	50	------	C46941	C46341	C46441	L1
	100	------	C46961	C46361	C46461	
	150	------	C46971	C46371	C46471	
SunShell C8-30, 2.6 µm	50	------	C36941	C36341	C36441	L7
	100	------	C36961	C36361	C36461	
	150	------	C36971	C36371	C36471	
SunShell C8-30HT, 3.4 µm	50	------	C56941	------	------	L7
	100	------	C56961	------	------	
	150	------	C56971	------	------	
SunShell C4-30, 2.6 µm	50	------	C26941	C26341	C26441	L26
	100	------	C26961	C26361	C26461	
	150	------	C26971	C26371	C26471	
SunShell C4-100, 2.6 µm	50	------	C66941	------	------	L26
	100	------	C66961	------	------	
	150	------	C66971	------	------	

*Distributor



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