Evaluation of Five Core Shell Columns Based on Both Separation Behavior and Physical Property

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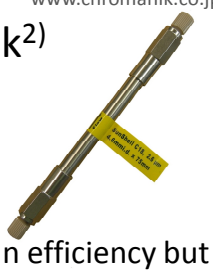
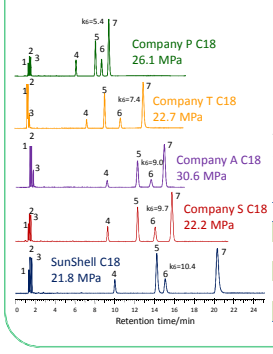


Abstract

A column packed with core shell particles has been widely used on HPLC and UHPLC, because it showed not only excellent column efficiency but also lower back pressure than sub-2 um column. More than 10 kinds of core shell column are available in the market. It is said that two types of core shell silica particle are used. One is a mono-layer structure as a porous silica layer like a core shell silica manufactured by Advanced Materials Technology and another is a multi-layer structure like that manufactured by Phenomenex.
In this study, a separation behavior and physical property of Kinetex C18, Accucore C18, PoroShell C18 EC, Ascentis Express C18 and SunShell C18 were evaluated. Retention factor, hydrogen bonding capacity, hydrophobicity and steric selectivity were measured using Tanaka method. Not only peak shape of neutral, acidic and basic compounds but also loading capacity of amitriptyline under neutral and acidic conditions were also measured. Furthermore stability under acidic pH1 and basic pH10 conditions was evaluated. Regarding physical property, carbon loading of each C18 packing material, and specific surface area, pore volume, pore diameter of each core shell silica which was deleted alkyl chains by sintering at 600 degree Celsius for 8 hours were measured. As a result, the big difference was recognized among 5 kinds of core shell C18 for separation behavior, stability and physical property. This difference is considered to be due to each manufacturing method and bonding technique as well as fully porous silica C18s. SunShell C18 showed the largest retention factor and the highest stability though its carbon loading is not the highest, while Kinetex C18 showed the lowest retention factor, the lowest carbon loading and the lowest specific surface area.

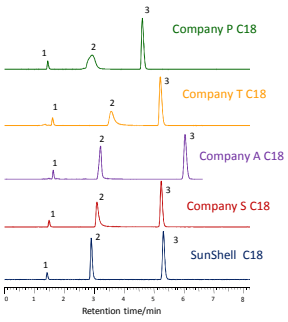
Comparison of Standard Samples



Column:
Company P C18, 2.6 µm 150 x 4.6 mm (26.1 MPa)
Company T C18, 2.6 µm 150 x 4.6 mm (22.7 MPa)
Company A C18, 2.7 µm 150 x 4.6 mm (30.6 MPa)
Company S C18, 2.7 µm 150 x 4.6 mm (22.2 MPa)
SunShell C18, 2.6 µm 150 x 4.6 mm (21.8 MPa)
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

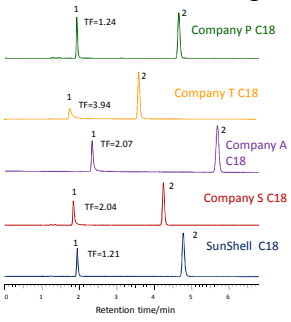
	Hydrogen bonding (Caffeine/Phenol)	Hydrophobicity (Amylbenzene/Butylbenzene)	Steric selectivity (Triphenylene/o-Terphenyl)
Company P C18	0.48	1.54	1.20
Company T C18	0.35	1.56	1.50
Company A C18	0.42	1.57	1.25
Company S C18	0.44	1.60	1.31
SunShell C18	0.39	1.60	1.46

Comparison of Pyridine



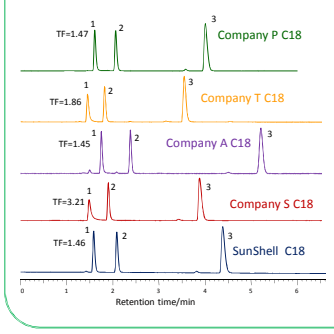
Column:
Company P C18, 2.6 µm 150 x 4.6 mm
Company T C18, 2.6 µm 150 x 4.6 mm
Company A C18, 2.7 µm 150 x 4.6 mm
Company S C18, 2.7 µm 150 x 4.6 mm
SunShell C18, 2.6 µm 150 x 4.6 mm
Mobile phase: CH_3OH/H_2O=30/70
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@250nm
Sample: 1 = Uracil, 2 = Pyridine, 3 = Phenol

Comparison of Oxine, Metal Chelating Compound



Column:
Company P C18, 2.6 µm 150 x 4.6 mm
Company T C18, 2.6 µm 150 x 4.6 mm
Company A C18, 2.7 µm 150 x 4.6 mm
Company S C18, 2.7 µm 150 x 4.6 mm
SunShell C18, 2.6 µm 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

Comparison of Formic Acid



Column:
Company P C18, 2.6 µm 150 x 4.6 mm
Company T C18, 2.6 µm 150 x 4.6 mm
Company A C18, 2.7 µm 150 x 4.6 mm
Company S C18, 2.7 µm 150 x 4.6 mm
SunShell C18, 2.6 µm 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

Summary

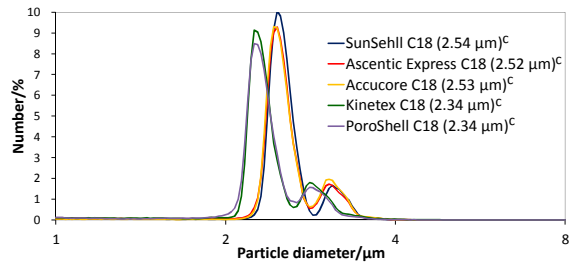
	Pressure[a]	Retention[b]	Pyridine	Oxine	Formic acid	Point
SunShell C18	◎21.8	10.4	◎	◎	◎	12
Ascentis Express C18	◎22.2	9.7	△	△	×	5
PoroShell C18 EC	×30.6	9.0	◎	△	◎	7
Accucore C18	◎22.7	7.4	×	×	△	4
Kinetex C18	△26.1	5.4	×	◎	◎	7

a. Mobile phase, methanol:water=75:25, 40 °C, 1mL/min 150 x 4.6mm
b. Retention factor of amylbenzene
◎: 3 point, ○: 2 point, △: 1 point, ×: 0 point

Physical Property

	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]
Kinetex C18	4.9 (12 effective)[b]	102 (200 effective)[b]	0.237	9.25 (10)[b]

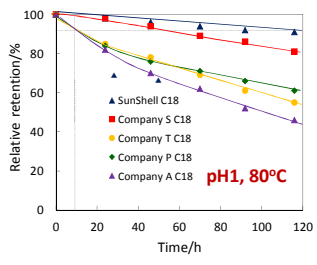
a. Measured after C18 materials were sintered at 600 degree Celsius for 8 hours. The measured value of each sintered core shell silica is considered to be smaller than that of the original core shell silica.
b. Value written in each brochure or literature



*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.

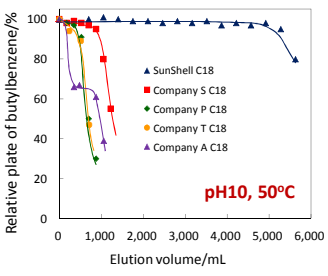
c. Median particle size

Stability under pH1 and 10 Conditions



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 °C

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 °C
Sample: 1 = Uracil, 2 = Butylbenzene



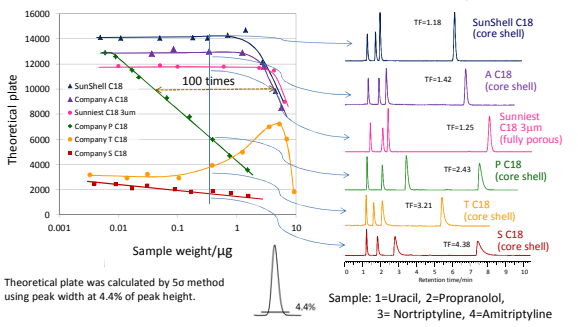
Durable test condition
Column size: 50 x 2.1 mm
Mobile phase: CH_3OH/20mM Sodium borate/10mM NaOH=30/21/49 (pH10)
Flow rate: 0.4 mL/min
Temperature: 50 °C

Measurement condition
Column size: 50 x 2.1 mm
Mobile phase: CH_3OH/H_2O=70/30
Flow rate: 0.4 mL/min
Temperature: 40 °C
Sample: 1 = Butylbenzene

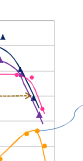
	Acidic condition pH 1	Basic condition pH 10	pH range written in each brochure
SunShell C18	◎	◎	1.5 - 10
Ascentis Express C18	○	○	2 - 9
PoroShell C18 EC	△	△	2 - 9
Accucore C18	△	△	1 - 11
Kinetex C18	△	△	1.5 - 10

Loading Capacity of Amitriptyline

Mobile phase: Acetonitrile/**20mM phosphate buffer pH7.0**=(60:40)
Column dimension: 150 x 4.6 mm, Flow rate: 1.0 mL/min, Temp.: 40°C

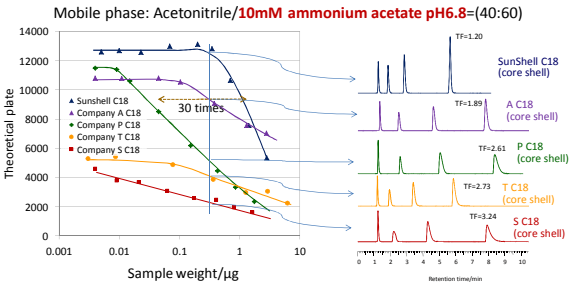


Theoretical plate was calculated by 5σ method using peak width at 4.4% of peak height.



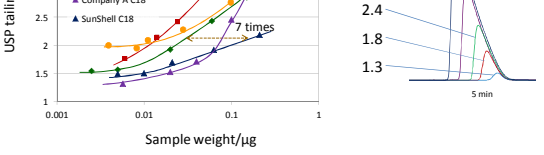
Sample: 1=Uracil, 2=Propranolol, 3= Nortriptyline, 4=Amitriptyline

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



Sample: 1=Uracil, 2=Propranolol, 3= Nortriptyline, 4=Amitriptyline

Mobile phase: Acetonitrile/**0.1% formic acid**=(30:70)



In the case of using acetonitrile /0.1% formic acid as a mobile phase, amitriptyline peak shows more tailing because a loading capacity decreases in an acidic, low-ionic-strength mobile phase.

Column name

1. Company P C18, 2.6 µm: Kinetex c18
2. Company T C18, 2.6 µm: Accucore C18
3. Company A C18, 2.7 µm: PoroShell C18 EC
4. Company S C18, 2.7 µm: Ascentis Express C18
5. SunShell C18, 2.6 µm

Conclusion

- ✓ A big difference concerning retention, residual silanol groups, metal impurity, loading capacity and stability among core shell C18s was confirmed as well as fully porous C18s
- ✓ SunShell showed the longest retention, the lowest pressure, the most symmetrical peak and the highest stability.
- ✓ Measurements of physical property are not same as a value to be shown in a brochure and a literature in some cases.