Stability Evaluation of Core Shell C18 with Encapsulated Type End-Capping

Norikazu Nagae* and Tomoyasu Tsukamoto
ChromaNik Technologies Inc. Namiyoke, Minato-ku, Osaka Japan 552-0001
*Corresponding author email: nagae@chromanik.co.jp

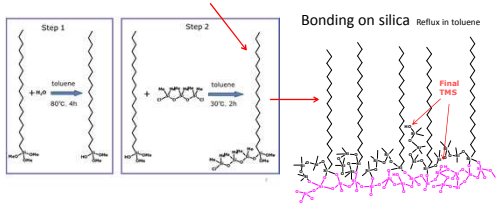
ChromaNik Technologies Inc.
www.chromanik.co.jp

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 20 kinds of core shell columns are available in the market. Recently high stability under a basic pH condition has been requested for a core shell reversed-phase as well as a fully porous C18. In this study, dense end-capping using a difunctional silyl-reagent was evaluated as an encapsulated type end-capping.

Present Bonding Technique

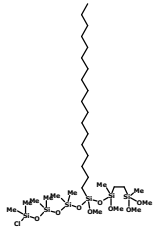
Sunniest bonding technique including end-capping

Synthesis of hexamethyloctadecyltetrasilane (C18 reagent A)

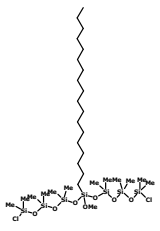


Bonding on silica Reflux in toluene

Novel Silyl-Reagents

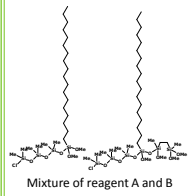


C18 reagent B



C18 reagent C

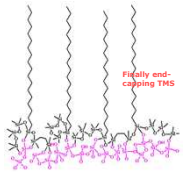
Bonding on Silica

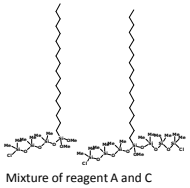


Mixture of reagent A and B

Mixture ratio of reagents A and B (2:1) (1:1) (1:2)

Reflux in toluene





Mixture of reagent A and C

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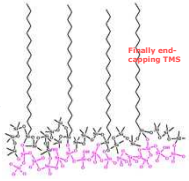
Reflux in toluene



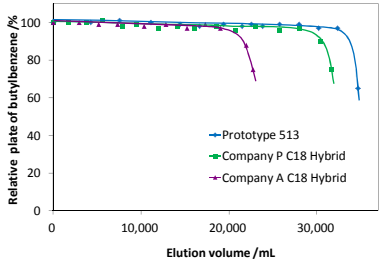
Evaluation of Stability under pH11.5 condition

	Ratio of reagents	Carbon loading	Elution time	Void in column	Relative plate
Present C18	A	7.2%	14 hours	1.3 mm	90%
Prototype 501	A:B=2:1	7.3%	34 hours	2.7 mm	83%
Prototype 502	A:B=1:1	7.1%	34 hours	2.2 mm	90%
Prototype 504	A:B=1:2	6.9%	34 hours	4.3 mm	62%
Prototype 505	A:C=2:1	7.7%	34 hours	3.0 mm	85%
Prototype 507	A:C=1:1	7.9%	34 hours	2.0 mm	91%
Prototype 508	A:C=1:2	7.1%	20 hours	3.3 mm	82%
Prototype 513	A:D=1:1	7.8%	50 hours	1.0 mm	90%

Durable test condition
Column dimension: 150 x 4.6 mm
Mobile phase:
CH_3OH/50mM Sodium phosphate buffer =10 / 90 (pH11.5)
Flow rate: 1 mL/min
Temperature: 40 °C

Measurement condition
Mobile phase:
CH_3CN/H_2O=70/30
Flow rate: 1 mL/min
Temperature: 40 °C
Sample: 1 = Butylbenzene

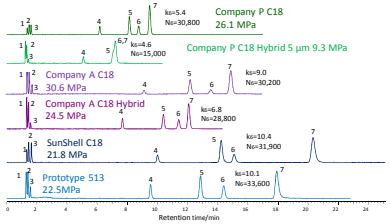
Stability under pH10.5 and at 60 °C



Durable test condition
Column dimension: 50 x 2.1 mm
Mobile phase:
CH_3OH/10mM Ammonium bicarbonate (pH 10.5)=30/70
Flow rate: 0.8 mL/min
Temperature: 60 °C

Measurement condition
Column dimension: 50 x 2.1 mm
Mobile phase:
CH_3CN/H_2O=60/40
Flow rate: 0.2 mL/min
Temperature: 40 °C
Sample: 1 = Butylbenzene

Standard Sample



Column:
Company P C18, 2.6 µm 150 x 4.6 mm
Company P C18 Hybrid, 5 µm 150 x 4.6 mm
Company A C18, 2.7 µm 150 x 4.6 mm
Company A C18 Hybrid, 2.7 µm 150 x 4.6 mm
SunShell C18, 2.6 µm 150 x 4.6 mm
Prototype 507 C18, 2.7 µm 150 x 4.6 mm

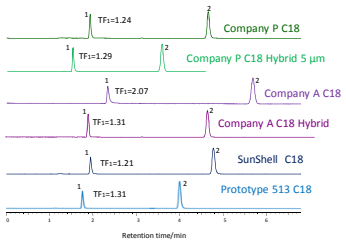
Mobile phase: CH_3OH/H_2O=75/25
Flow rate: 1.0 mL/min
Temperature: 40 °C
Sample: 1 = Uracil, 2 = Caffeine, 3 = Phenol, 4 = Butylbenzene 5 = o-Terphenyl, 6 = Amylbenzene, 7 = Triphenylene

	Hydrogen Bonding Capacity (Caffeine/Phenol)	Hydrophobicity (Amylbenzene/Butylbenzene)	Steric Selectivity (Triphenylene/o-Terphenyl)
Company A C18 2.6 µm	0.48	1.54	1.20
Company P C18 Hybrid 5 µm	0.40	1.45	1.02
Company A C18, 2.7 µm	0.42	1.57	1.25
Company A C18 Hybrid, 2.7 µm	0.42	1.58	1.19
SunShell C18, 2.6 µm	0.39	1.60	1.46
Prototype 513 C18, 2.6 µm	0.39	1.59	1.44

Conclusion

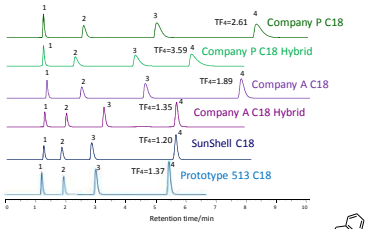
* The novel C18 with encapsulated type end-capping showed almost same stability under basic pH conditions to compare with a hybrid type C18.
* The novel C18 with encapsulated type end-capping showed a good peak shape for a metal chelating compound, acidic compounds and basic compounds although the other hybrid type C18 showed a poor peak shape for formic acid.
* It was guessed that an amine remained on the silica surface like a by-product of a silyl-reagent leaded a poor peak shape for formic acid.

Metal Chelating Compound



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

Basic Compound

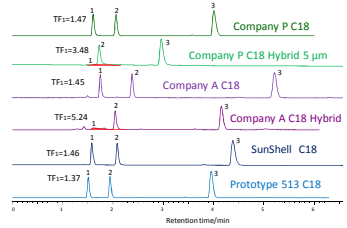


Mobile phase: Acetonitrile/10mM ammonium acetate pH6.8=40/60
Flow rate: 1.0 mL/min, Temp.: 40°C

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

Amitriptyline

Acidic Compound



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

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