

# 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

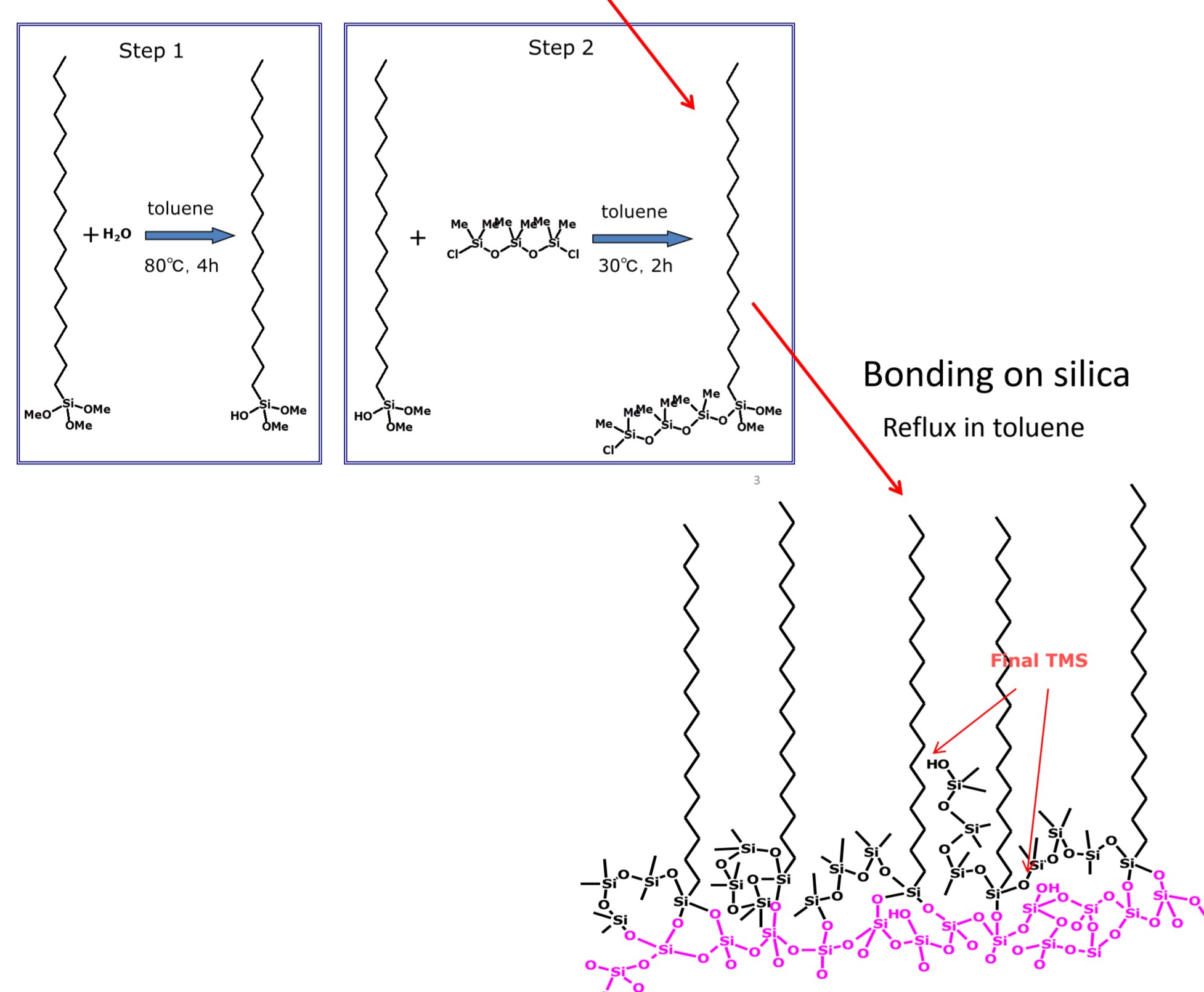


A column packed with core shell particles has been widely used on HPLC and UHPLC because it showed not only excellent column efficiency but also lower back pressure than sub-2 μm column. More than 20 kinds of core shell columns are available in the market. Recently high stability under a basic pH condition 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.

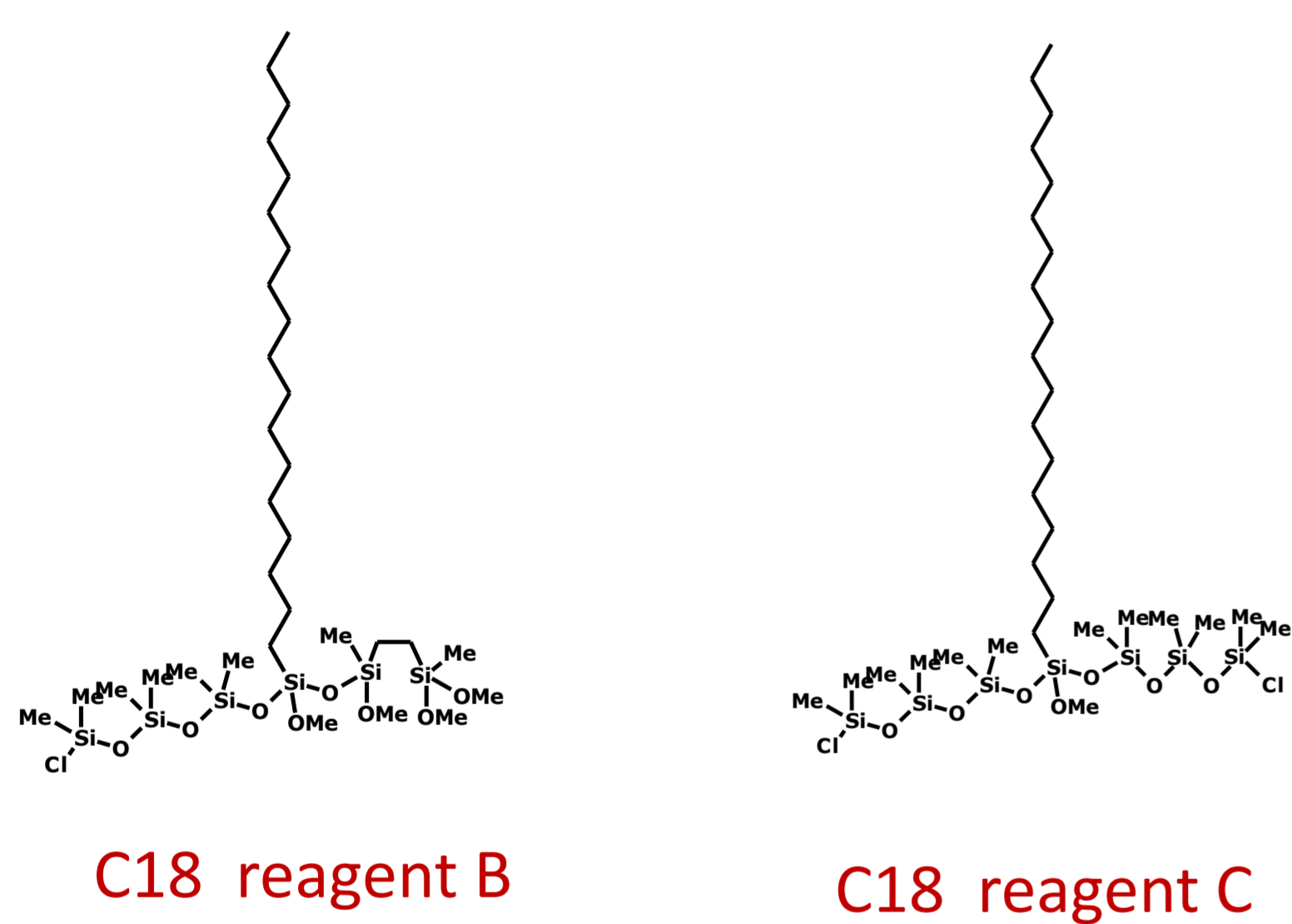
## Present Bonding Technique

Sunniest bonding technique including end-capping

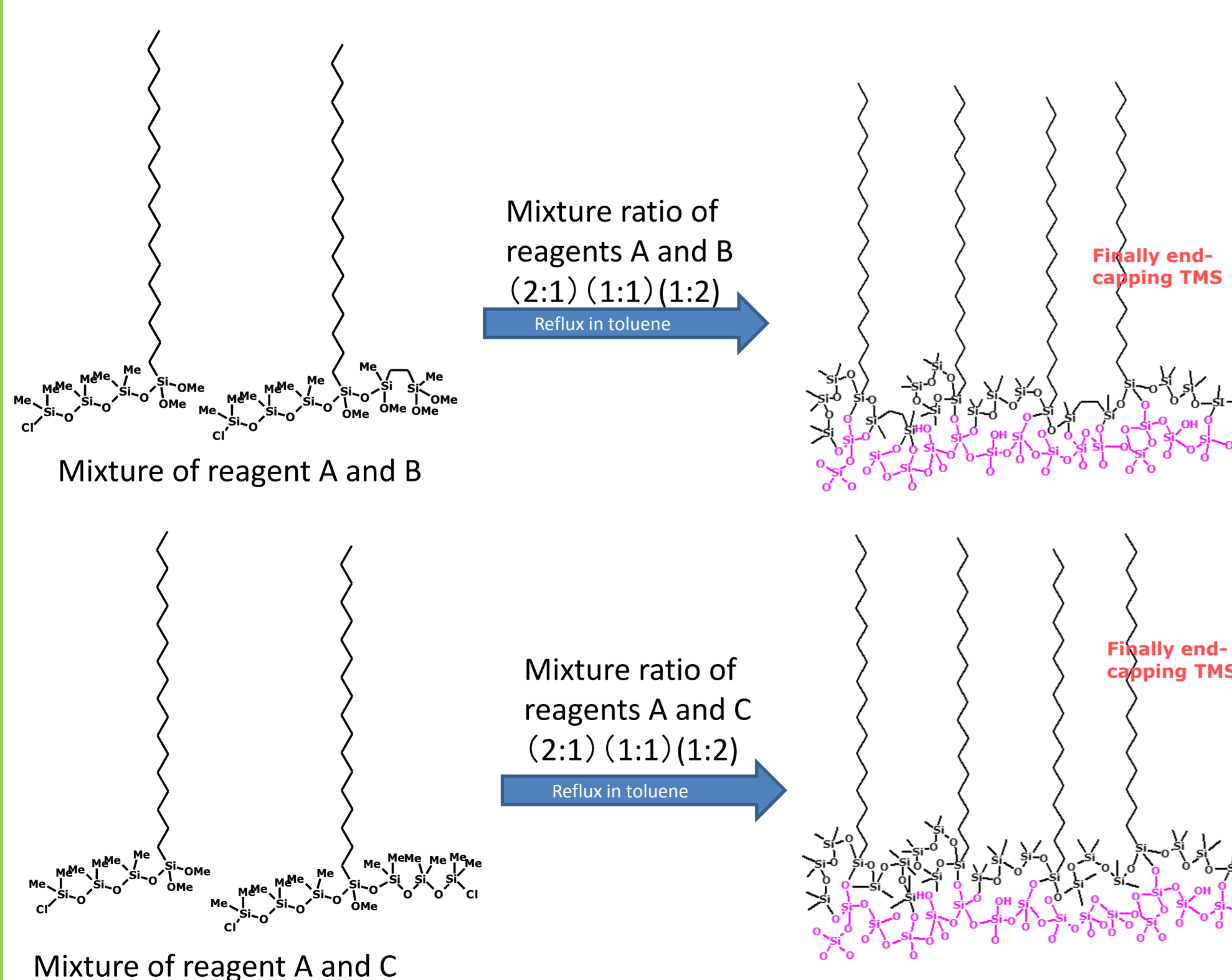
Synthesis of hexamethyloctadecyltetrasilane (C18 reagent A)



## Novel Silyl-Reagents



## Bonding on Silica



## 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 led a poor peak shape for formic acid.

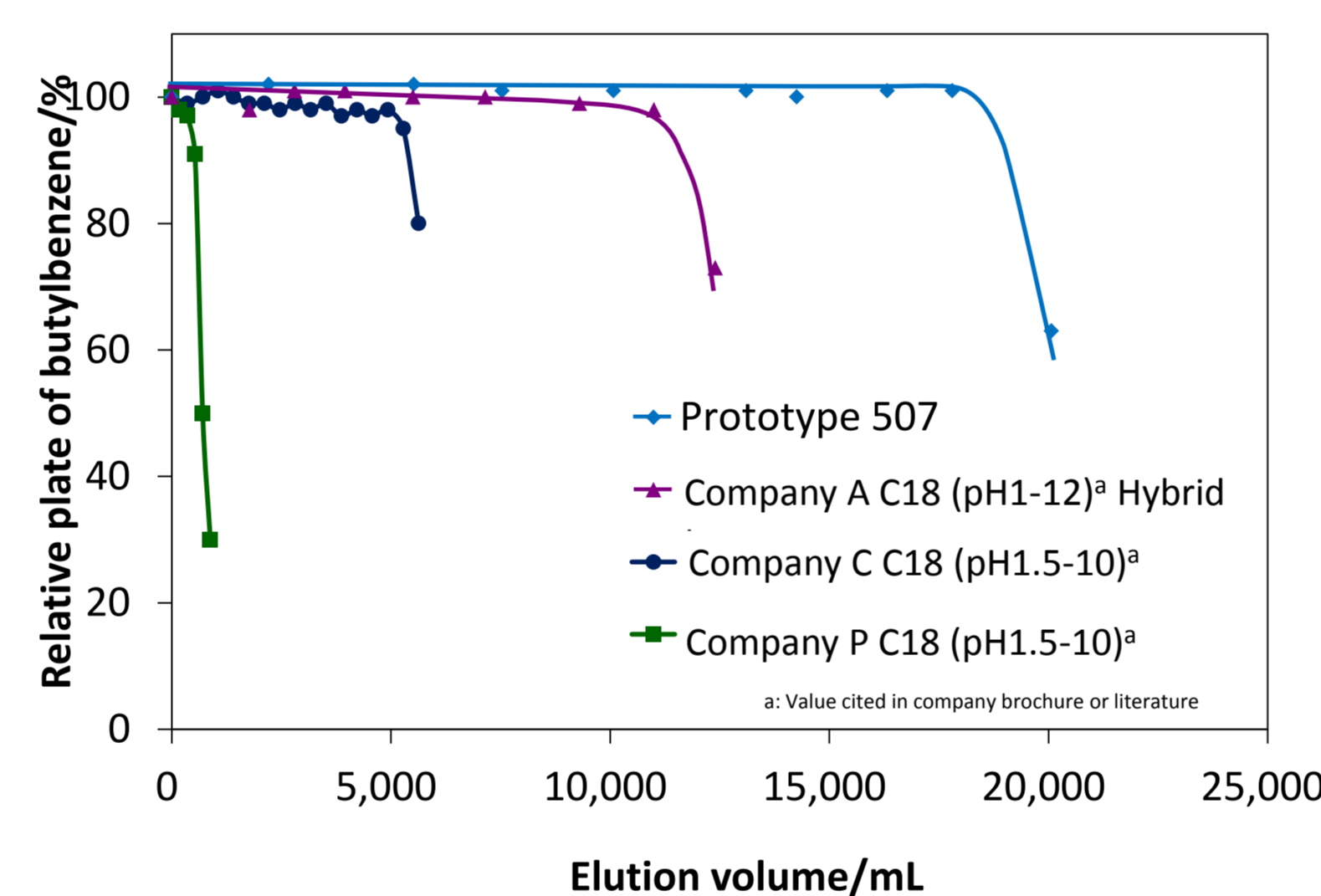
## Evaluation of Stability under pH11.5 condition

|               | 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<sub>3</sub>OH/50mM Sodium phosphate buffer = 10 / 90 (pH11.5)  
Flow rate: 1 mL/min  
Temperature: 40 °C

**Measurement condition**  
Mobile phase:  
CH<sub>3</sub>CN/H<sub>2</sub>O=70/30  
Flow rate: 1 mL/min  
Temperature: 40 °C  
Sample: 1 = Butylbenzene

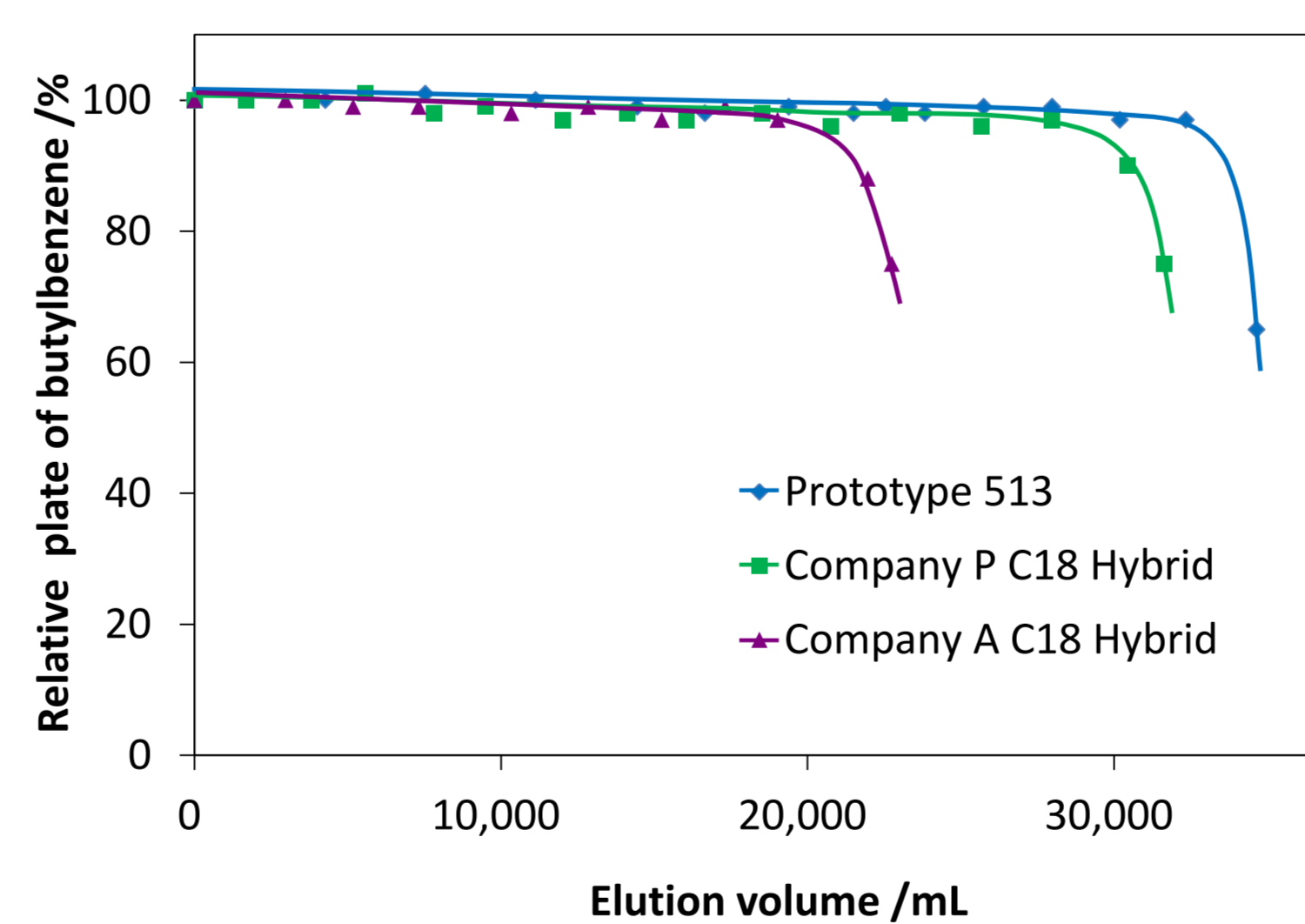
## Stability under pH10 and at 50 °C



**Durable test condition**  
Column dimension: 50 x 2.1 mm  
Mobile phase:  
CH<sub>3</sub>OH/20mM Sodium borate/10mM NaOH=30/21/49 (pH10)  
Flow rate: 0.4 mL/min  
Temperature: 50 °C

**Measurement condition**  
Column dimension: 50 x 2.1 mm  
Mobile phase:  
CH<sub>3</sub>CN/H<sub>2</sub>O=60/40  
Flow rate: 0.2 mL/min  
Temperature: 40 °C  
Sample: 1 = Butylbenzene

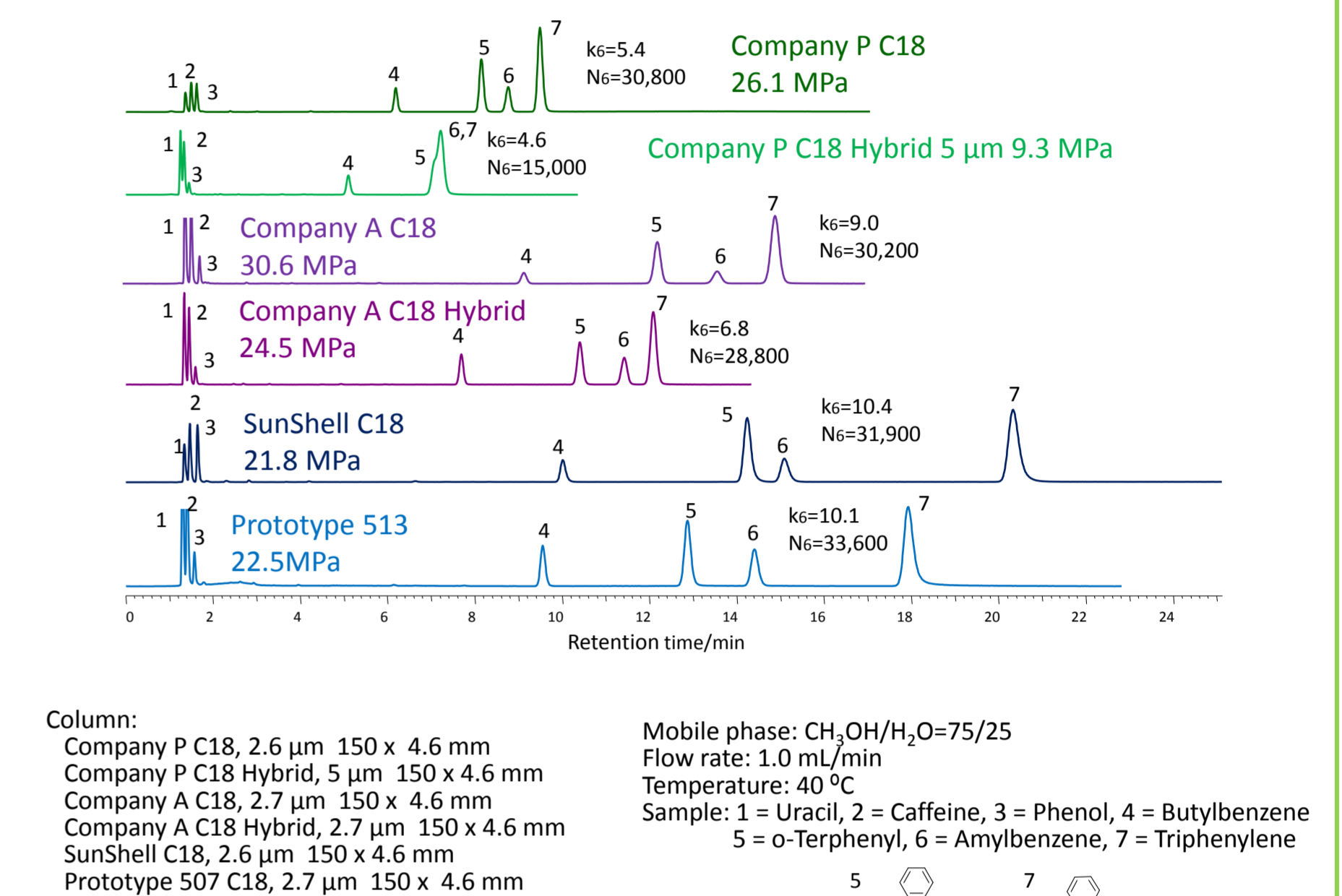
## Stability under pH10.5 and at 60 °C



**Durable test condition**  
Column dimension: 50 x 2.1 mm  
Mobile phase:  
CH<sub>3</sub>OH/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<sub>3</sub>CN/H<sub>2</sub>O=60/40  
Flow rate: 0.2 mL/min  
Temperature: 40 °C  
Sample: 1 = Butylbenzene

## Standard Sample

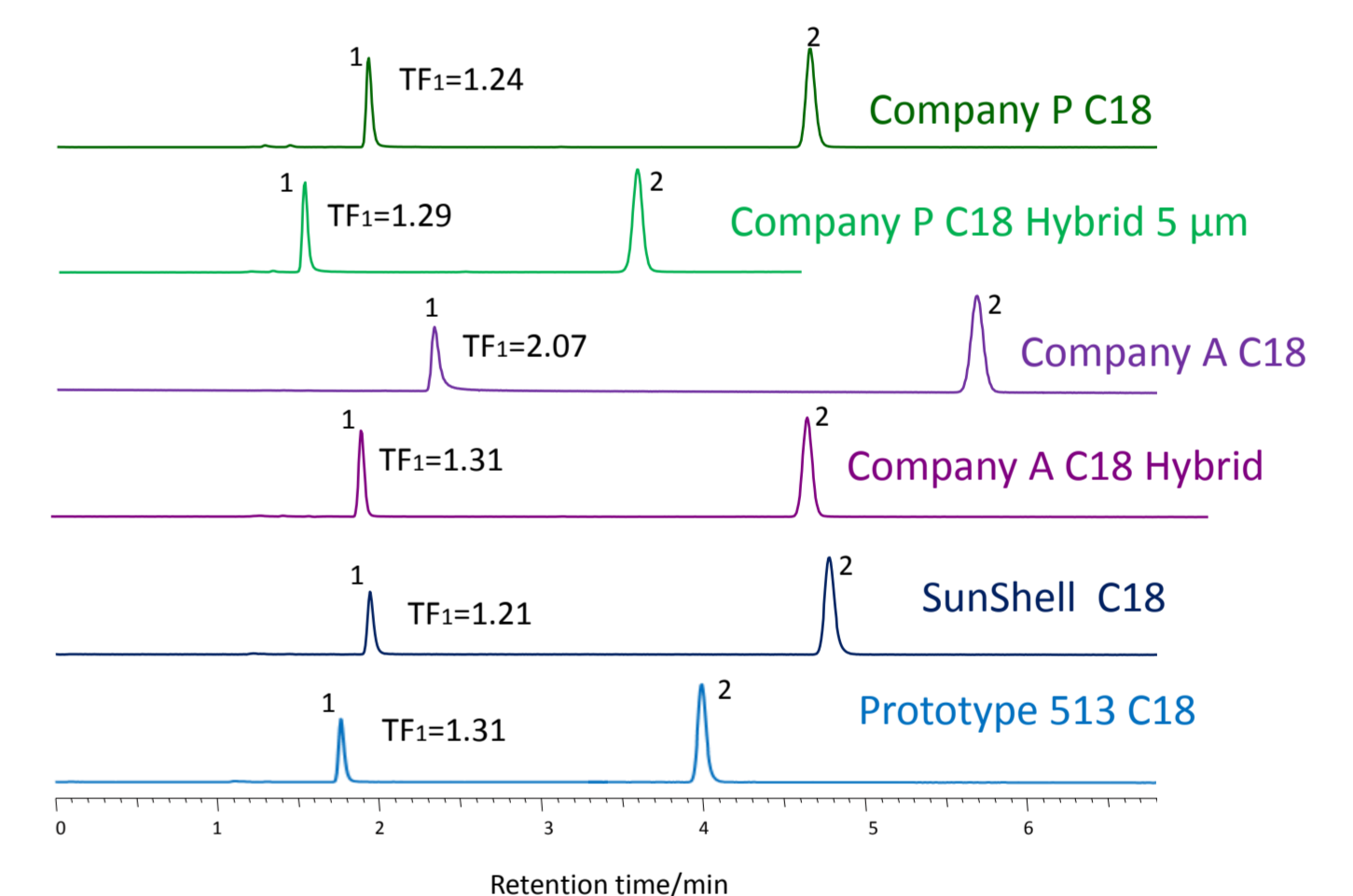


Column:  
Company P C18, 2.6 μm 150 x 4.6 mm  
Company P C18 Hybrid, 5 μm 150 x 4.6 mm  
Company A C18, 2.7 μm 150 x 4.6 mm  
Company A C18 Hybrid, 2.7 μm 150 x 4.6 mm  
SunShell C18, 2.6 μm 150 x 4.6 mm  
Prototype 507 C18, 2.7 μm 150 x 4.6 mm

Mobile phase: CH<sub>3</sub>OH/H<sub>2</sub>O=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

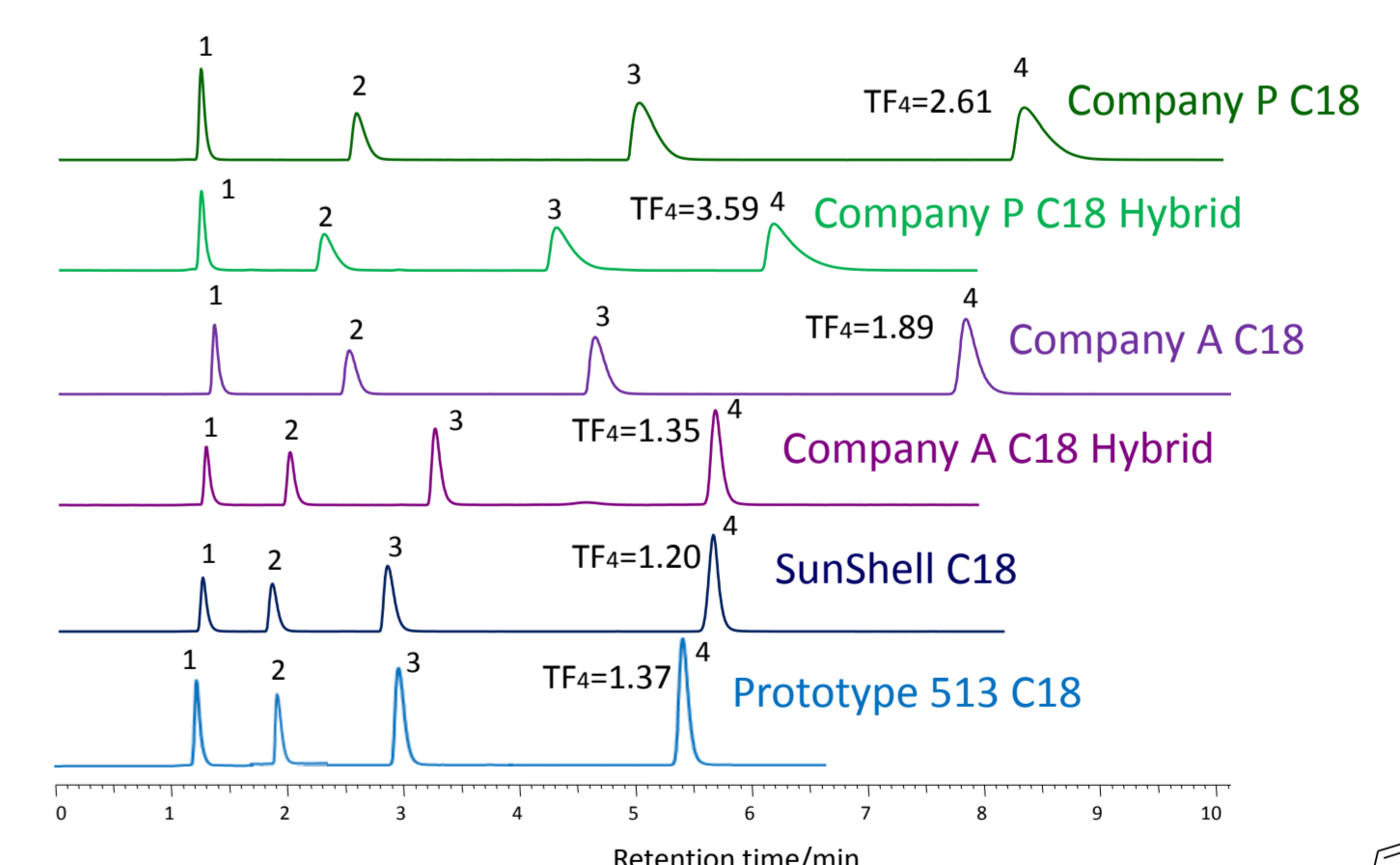
|                              | Hydrogen Bonding Capacity (Caffeine/Phenol) | Hydrophobicity (Amylbenzene/Butylbenzene) | Steric Selectivity (Triphenylene/o-Terphenyl) |
|------------------------------|---------------------------------------------|-------------------------------------------|-----------------------------------------------|
| 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    | 0.37                                        | 1.59                                      | 1.44                                          |

## Metal Chelating Compound

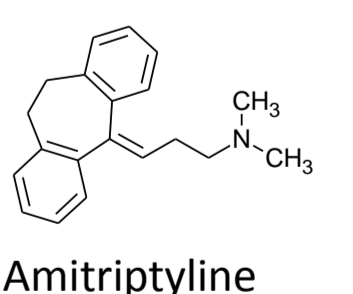


Mobile phase: CH<sub>3</sub>CN/20mM H<sub>3</sub>PO<sub>4</sub>=10/90  
Flow rate: 1.0 mL/min, Temperature: 40 °C  
Detection: UV@250nm  
Sample: 1 = 8-Quinololin (Oxine), 2 = Caffeine

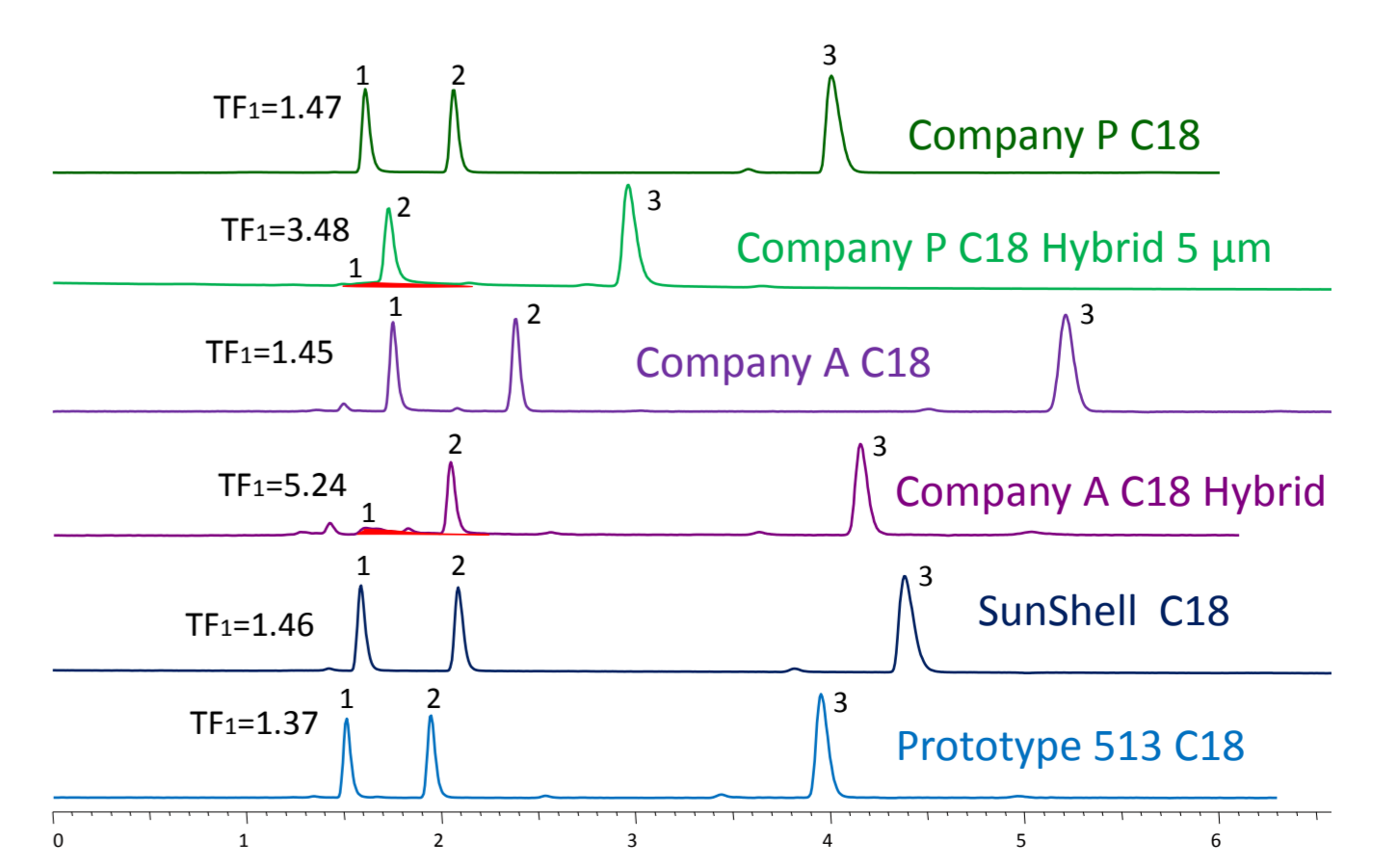
## Basic Compound



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



## Acidic Compound



Mobile phase: CH<sub>3</sub>CN/0.1% H<sub>3</sub>PO<sub>4</sub>=2/98  
Flow rate: 1.0 mL/min, Temperature: 40 °C  
Detection: UV@210nm  
Sample: 1 = Formic acid, 2 = Acetic acid, 3 = Propionic acid