

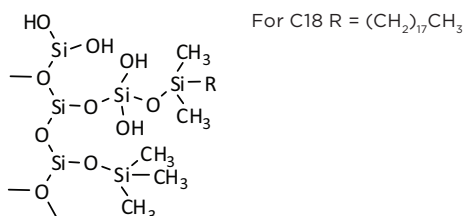
SiliaChrom dt C18 SiliaChrom AQ C8 and C18

Description

Universal 100% aqueous compatible HPLC columns **SiliaChrom dt** adsorbent presents an optimum ratio of C18 short TMS chains and some free silanol groups. This new technology shows good peak shapes for any type of molecule (*acid, neutral and base*). The silica framework is exempt of any metal permitting a high sensitivity for LC-MS applications.

SiliaChrom AQ presents the same modified surface chemistry as dt but the silica framework contain low level of metal. C8 and C18 functions are available.

Structure



SiliaChrom dt Purity: 99.9999% SiO₂ (*no metal content*)

SiliaChrom AQ Purity: 99.9999% SiO₂

Sorbent Characteristics

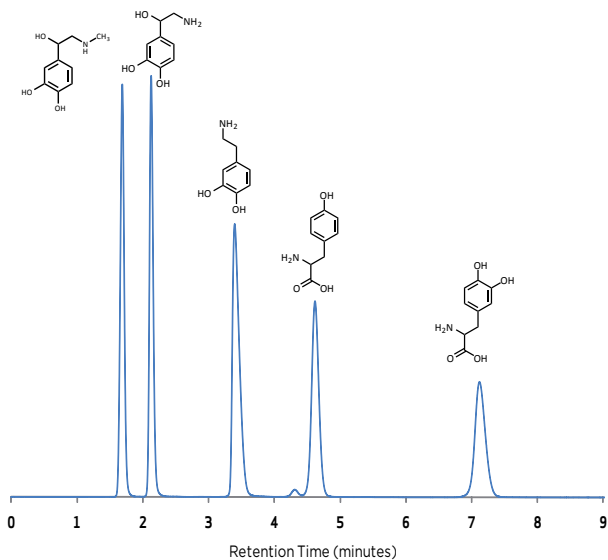
- **Pore Size:** 100 Å
- **Specific Surface Area:** SiliaChrom dt C18 410 - 440 m²/g
SiliaChrom AQ C8 & C18 380 m²/g
- **Particle Sizes Available:** 2.5, 3, 5 and 10 µm
- **USP Code:** L1
- **Typical Carbon Loading:** SiliaChrom dt & AQ C18: 18%
SiliaChrom AQ C8 14%

SiliaChrom dt and AQ Main Characteristics

- Enhanced retention of hydrophilic molecules
- Inertness for acidic and basic analytes
- Compatible from 100% aqueous mobile phase to 100% organic
- Exceptional stability from pH 1.5 to 9.0
- Good tolerance to direct injection of biological matrix (*dirty samples*)
- Reduces the need for mobile phase modifiers
- Low bleeding and high sensitivity for LC-MS
- Partially endcapped

Separation of Catecholamines in Acidic Mobile Phase

Catecholamines are hydrophilic compounds with acidic functions. The mobile phase needs to be acidic to have the catecholamines under the molecular configuration and use the sorbent hydrophilic character to drive the separation.



Chromatographic conditions

- **Column:** SiliaChrom dt C18, 5 µm
- **Column size:** 4.6 x 150 mm
- **SiliCycle P/N:** H141805E-N150
- **Mobile phase:** 1% Acetic Acid in water
- **Temperature:** 23°C
- **Temperature:** 23°C
- **Flow rate:** 1.000 mL/min
- **Detector:** UV at 265 nm
- **Injection volume:** 5 µL

« Polar metabolites separation is very challenging. Using SiliaChrom, dt C18 in normal phase solved the problem. »

Huns Nejad from BASF, Research Triangle Park, NC, USA

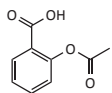


Forensic

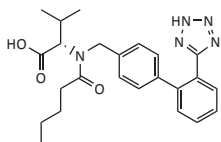


Assay for QC Testing of Blood Pressure and Cholesterol Medication

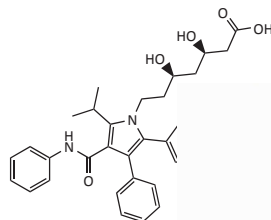
The SiliaChrom dt C18 presents a high lot-to-lot reproducibility, which makes it an excellent choice for quality control analysis in pharmaceutical laboratories.



A: Aspirine



B: Valsartan



C: Atorvastatin

Chromatographic conditions

- **Column:** SiliaChrom dt C18, 5 μm
- **Column size:** 4.6 x 150 mm
- **SiliCycle P/N:** H141805E-N150
- **Mobile phase:** Methanol/H₂O (70/30), 0.1% (v/v) formic acid
- **Temperature:** 30°C
- **Flow rate:** 0.800 mL/min
- **Detector:** UV at 280 nm
- **Injection volume:** 10 μL

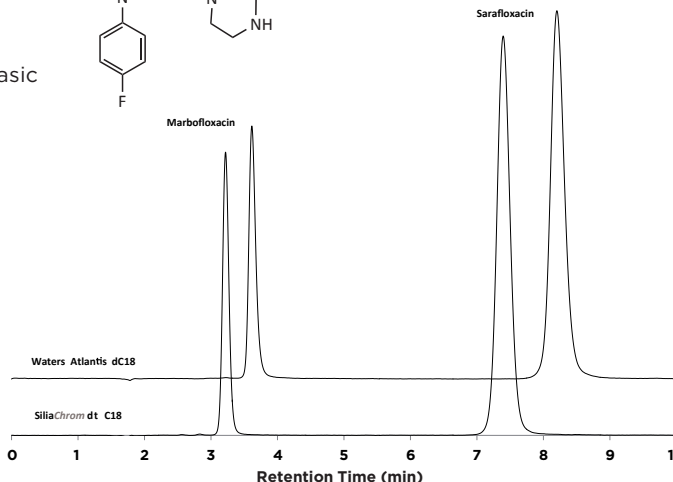
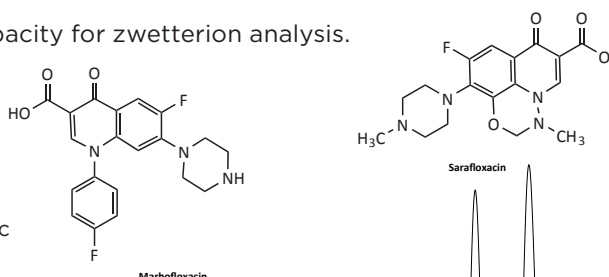


Peak Shape Evaluation for Zwitterion Fluoroquinolones

The SiliaChrom dt C18 presents a high separation capacity for zwitterion analysis.

Chromatographic conditions

- **Column:** SiliaChrom dt C18, 5 μm
- **Column size:** 4.6 x 150 mm
- **SiliCycle P/N:** H141805E-N150
- **Mobile phase:** 2.5 mM potassium phosphate monobasic (adjust to pH 2.5 with H₃PO₄)/ethanol (68/32)
- **Temperature:** 23°C
- **Flow rate:** 1.000 mL/min
- **Detector:** UV at 275 nm
- **Injection volume:** 10 μL



Peak Shape Results		
Product	Asymmetry (USP) SiliaChrom dt C18	Asymmetry (USP) Atlantis dC18
Marbofloxacin	1.11	1.29
Sarafloxacin	1.08	1.14



Ropinirole and Amitriptyline Detection in Human Plasma

SiliaChrom dt C18 presents low bleeding and is excellent for dirty samples. Partial endcapping allows for some interactions with free silanol groups. The use of SiliaPrep CleanDRUG prior to injection onto the column will insure a very clean sample which results in very low ionic suppression when using in LC-MS/MS analysis. Another big advantage is the high selectivity of SiliaChrom dt C18 for all concentration levels.

Chromatographic conditions

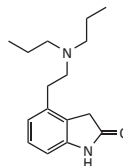
- **Column:** SiliaChrom dt C18, 2.5 μm
- **Column size:** 3.0 x 30 mm
- **SiliCycle P/N:** H141802E-H030
Sample preparation by SPE
SiliaPrep CleanDRUG 3 mL/500 mg
PN: SPEC-R651230B-03G
- **Mobile phase:**
MPA: 1 mM ammonium formate in (ACN/water, 10/90), 0.1% formic acid (v/v)
MPB: 1 mM ammonium formate in (ACN/water, 90/10), 0.1% formic acid (v/v)

Gradient		
Time (min)	MPA (%)	MPB (%)
0.00 - 0.20	85	15
0.21 - 1.20	50	50
1.21 - 1.60	0	100
1.61 - 3.50	85	15

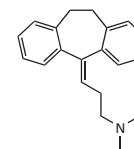
- **Temperature:** 23°C
- **Flow rate:** 1.000 mL/min
- **MS splitting flow:** 0.30 mL/min
- **Injection volume:** 5 μL

Tandem mass spectroscopy conditions

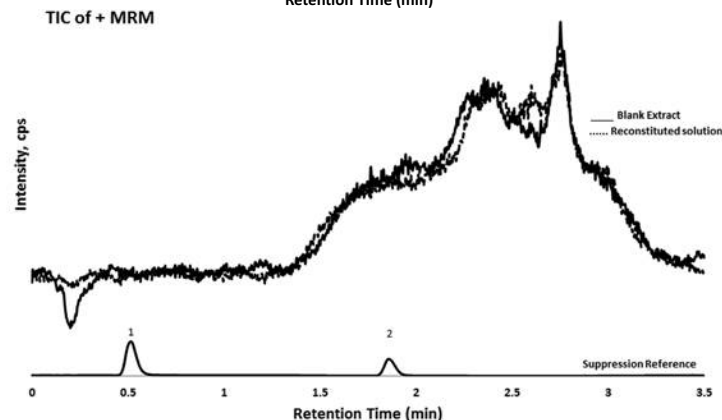
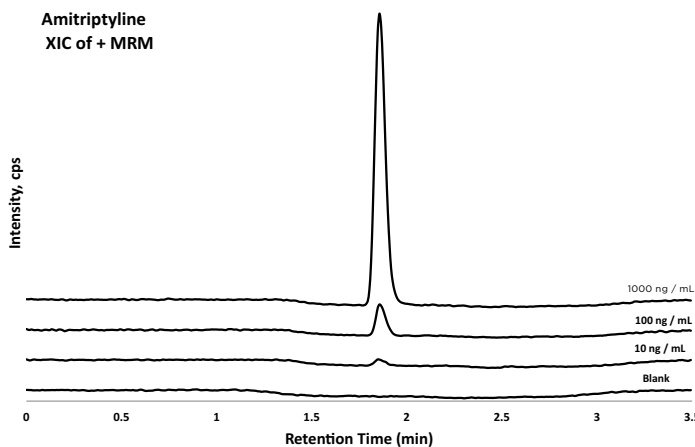
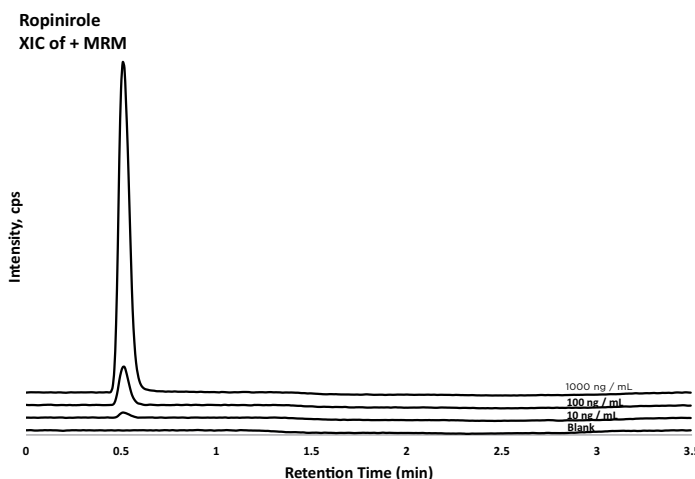
- **Detector:** Sciex API 3000, Applied Biosystem
- **Ion Source:** Positive Electrospray (ESI+)
- **Turbolon Ion Spray heater gas flow:** 8000 cc/min
- **Turbolon Ion Spray heater temperature:** 375°C
- **MRM Transition:** Ropinirole: m/z (261.2 \rightarrow 114.2)
Amitriptyline: m/z (278.4 \rightarrow 233.1)



Ropinirole

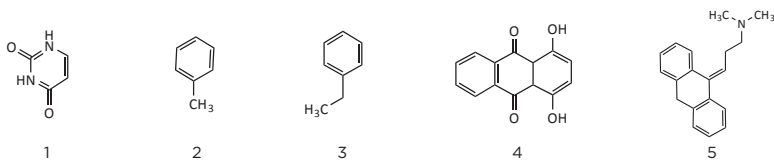


Amitriptyline



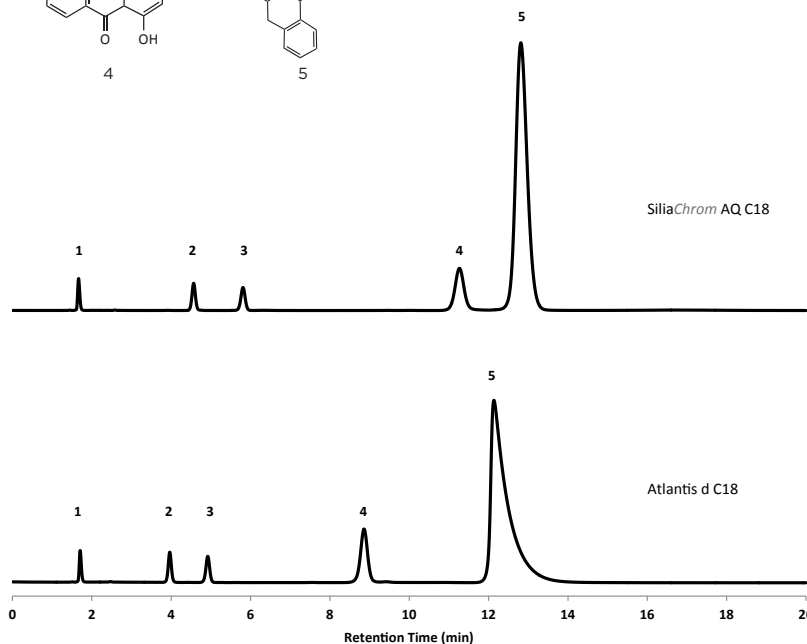
SiliaChrom AQ C18 is Highly Efficient for Basic Compounds

Amitriptyline, a strong basic compound, can be adsorbed on residual silanols on the surface of the packing material. With the traditional endcapping technique, this results in poor peak shapes. SiliCycle has developed a new method of silanol deactivation to eliminate the peak tailing from adsorption of compounds on residual silanol groups. This enables highly qualitative and quantitative analysis of strong basic compounds.



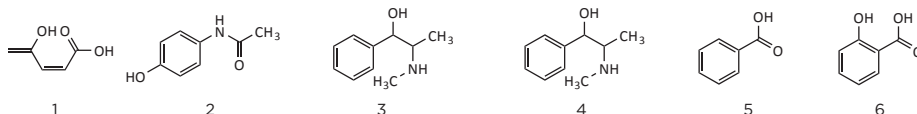
Chromatographic conditions

- **Column:** SiliaChrom AQ C18, 5 μ m
- **Column size:** 4.6 x 150 mm
SiliCycle P/N: H151805E-N150
- **Mobile phase:** 80/20 methanol/
20 mM potassium phosphate pH 7.00
- **Temperature:** 23°C
- **Flow rate:** 1.000 mL/min
- **Detector:** UV at 254 nm
- **Injection volume:** 1 μ L



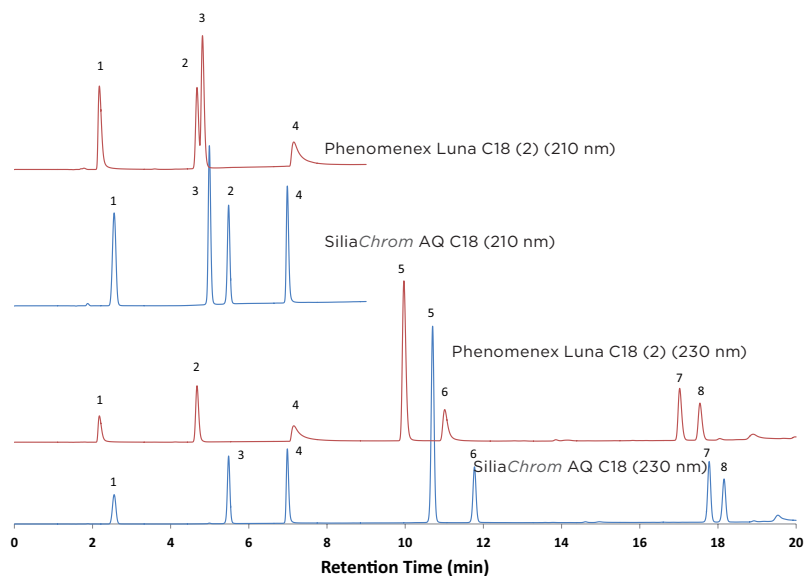
Evaluation of Resolution and Peak Shape

The SiliaChrom AQ C18 column is universal, efficient even for mixtures of basic and acidic compounds.



Chromatographic conditions

- **Column:**
SiliaChrom AQ C18, 5 μ L
Phenomenex Luna, C18 (2) 5 μ L
- **Column size:** 4.6 x 150 mm
- **SiliCycle P/N:** H151805E-N150
- **Mobile phase:**
MPA: 5 mM potassium phosphate monobasic
(adjust to pH 2.5 with H_3PO_4)/ACN (90/10)
MPB: 5 mM potassium phosphate monobasic
(adjust to pH 2.5 with H_3PO_4)/ACN (10/90)
- **Temperature:** 23°C
- **Flow rate:** 1.000 mL/min
- **Detector:** UV at 254 nm
- **Injection volume:** 5 μ L





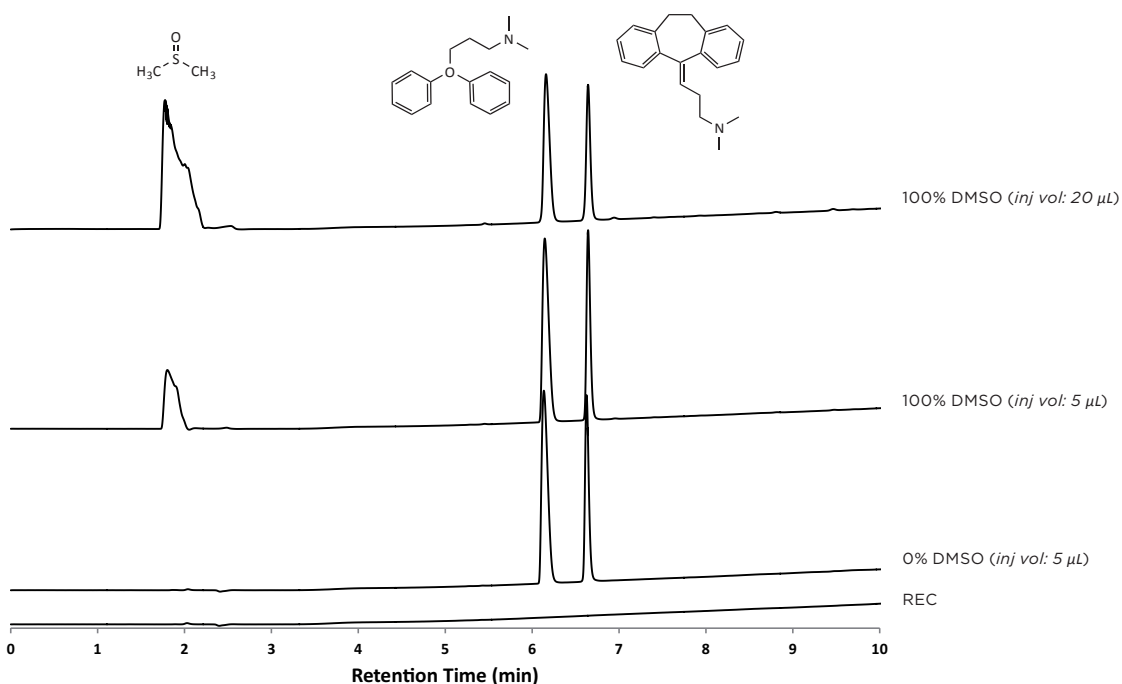
Retention Capacity of DMSO on SiliaChrom AQ C18

DMSO (*Dimethylsulfoxide*) is an excellent solvent to solubilize most compounds. Unfortunately, this solvent is not volatile and with some C18 columns, the DMSO can interact with the stationary phase and decrease the selectivity. In this case, the only way to inhibit this effect is to use preparative chromatography. In this study, we show that DMSO does not interact with our SiliaChrom AQ C18. A linear gradient has been used from a highly aqueous mobile phase to a highly organic phase.

Chromatographic conditions

- **Column:** SiliaChrom AQ C18, 5 μm
- **Column size:** 4.6 x 150 mm
- **SiliCycle P/N:** H151805E-N150
- **Mobile phase:** MPA 0.1% formic acid in water
MPB 0.1% formic acid in ACN
- **Temperature:** 23°C
- **Flow rate:** 1.000 mL/min
- **Detector:** UV at 254 nm
- **Reconstitution solution (REC):** DMSO

Gradient		
Time (min)	% MPA	% MPB
0	90	10
9	10	90
10	10	90
11	90	10



Statistic Analysis Results

Conditions	A_{DMSO}	$T_{\text{r DMSO}}$ (min)	K'_{DMSO}	W_{DMSO}	$T_{\text{r diphenhydramine}}$ (min)	$T_{\text{r amitriptyline}}$ (min)
0% DMSO 5 μL	-	-	-	-	6.14	6.63
100% DMSO 5 μL	2.29	1.80	0.09	0.3	6.15	6.64
100% DMSO 20 μL	4.10	1.78	0.08	0.5	6.16	6.64

Conclusion: The study shows that DMSO does not interact with the SiliaChrom AQ C18. No specific retention is observed. The SiliaChrom AQ C18 is an excellent choice to purify components contaminated with DMSO.



Pharmaceutical



Forensic

Dewetting Phenomena

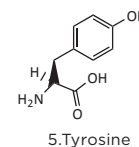
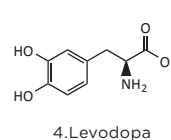
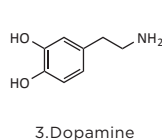
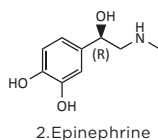
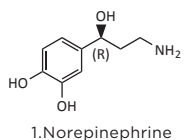
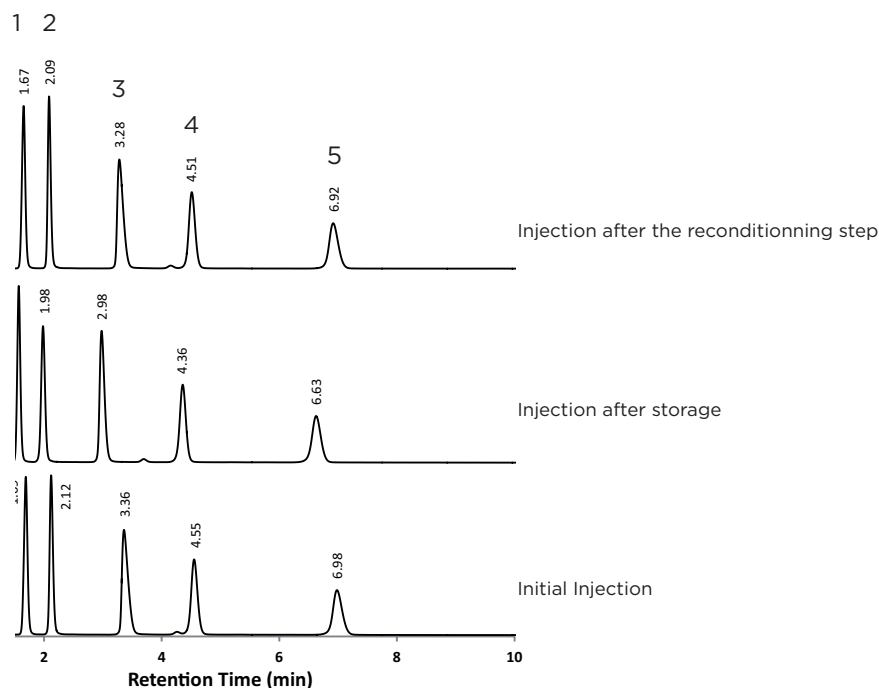
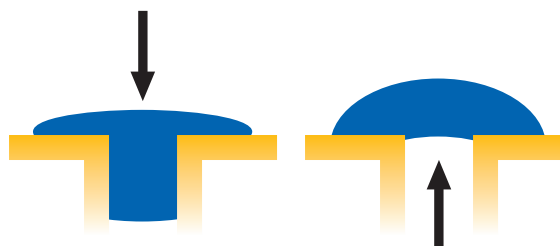
The dewetting phenomena is the formation of drops on the solid surface caused by hydrophobic repulsions of highly hydrophobic sorbents. This phenomena is illustrated by the following scheme.

General procedure

- The mixture of catecholamines is eluted on the column
- The flow is then stopped
- The column is stored in this condition during 18 h
- The mixture is then re-injected after a reconditioning step

Chromatographic conditions

- **Column:** SiliaChrom AQ C18, 5 μm
- **Column size:** 4.6 x 150 mm
- **SiliCycle P/N:** H151805E-N150
- **Mobile phase:** 1% acetic acid in water
- **Temperature:** 23°C
- **Flow rate:** 1.000 mL/min
- **Detector:** UV at 265 nm
- **Injection volume:** 5 μL



Conclusion: A small decrease in retention time is observed, but is not significant. The displacement has been resolved after the reconditioning step. The SiliaChrom AQ C18 does not present the dewetting phenomena.