APPLICATION NOTE
#SB006-0

SiliCycle

SiliaBond®

Working with fluorinated silica phases

Fluorinated sorbents are particularly useful for the separation of fluorine containing molecules from one another as well as from non-fluorous ones. They can also be used in chromatographic separations of non-fluorinated molecules, similarly to a regular reversed-phase just with a different selectivity. SiliCycle offers three different phases with various selectivities: SiliaBond® Pentafluorophenyl (*PFP*), SiliaBond® Tridecafluoro (*TDF*) and SiliaBond® Fluorochrom (*FCM*) nec. Available in bulk (*SiliaBond®*), in SPE cartridges (*SiliaPrep™*), in pre-packed flash cartridges (*either irregular SiliaSep™ or as spherical SiliaSep™ PREMIUM*) and in HPLC columns (*SiliaChrom® Plus PFP*) to help our customers achieve their separation goals.

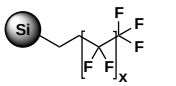
LEARN MORE about our chromatographic phases in our brochure "*Solutions for Purification and Chromatography*".

APPLICATION NOTE
#SB006-0

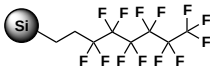
Working with fluorinated silica phases

Available phases

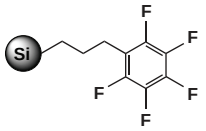
SiliCycle offers three fluorinated products:



Silia*Bond*® Fluorochrom (*Si-FCM*) nec (*R63730B*) is a fluorinated sorbent particularly useful for the separation of fluorine containing molecules from non-fluorous ones. In the left structure x > 5, but the exact number is proprietary.



Silia*Bond*® tridecafluoro (*Si-TDF*) (*R63530B*) is a sorbent primarily used to separate fluorinated molecules. It can also be used in fluorous solid-phase extraction (*FSPE*) or fluorous-tag compounds as reported for the synthesis of oligosaccharide.



Silia*Bond*® Pentafluorophenyl (*Si-PFP*) (*R67530B*) is a sorbent primarily used in the separation of molecules bearing fluorine atoms but may also be used in the separation of non-fluorous compounds such as Taxol® and its derivatives. Si-PFP has a higher selectivity for aromatics containing molecules compared to the other fluorinated sorbents due to its phenyl ring.

The main difference between these three phases is the fluorine content, with Silia*Bond*® Pentafluorophenyl (*PFP*) having the least and Silia*Bond*® Fluorochrom (*FCM*) nec having the most. By increasing the fluorine content, the more selective the sorbent becomes towards fluorine containing molecules.

The use of fluorinated phases in chromatography

For the chromatographic methods using fluorinated phases, the retention of compounds depends mainly on two factors: the hydrophobic character of the analyte and its percentage of fluorine atoms. Thus, the more hydrophobic and fluorinated a molecule is, the better it will be retained in the column. Keep in mind that in some cases, some molecules might not be fluorinated enough to show good retention.

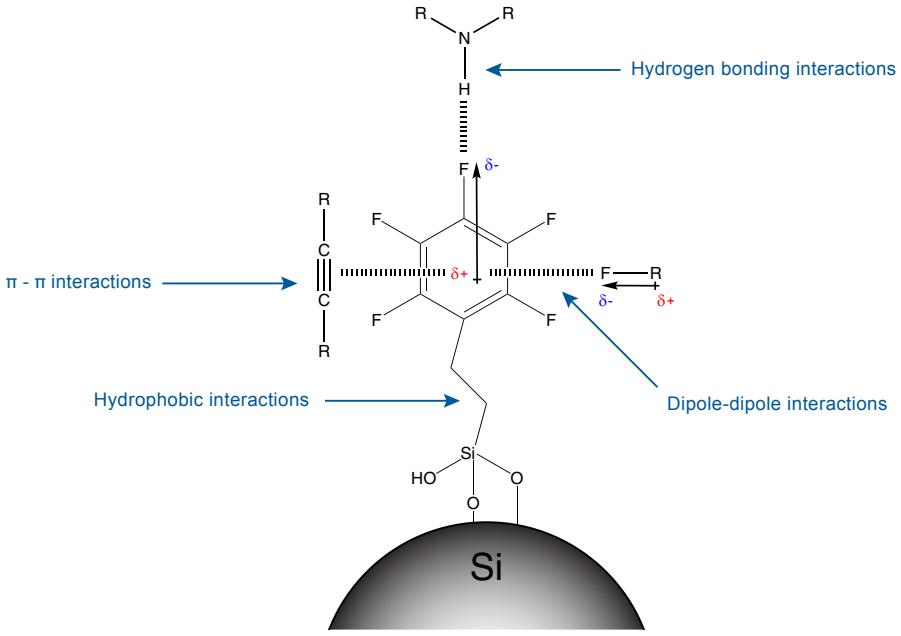
Fluorinated phases are reversed-phase silica gels with a different selectivity than a traditional C18 phase, therefore the same solvent systems applied with traditional reversed-phases can also be used. With these phases, the separation of the fluorinated compounds will be dictated by the stationary phase. Thus, to increase the selectivity or increase the interaction between the fluorinated analyte and the stationary phase, the residence time in the column must be increased. That is why, a mobile phase containing a high percentage of water must be used with a hydrophilic analyte and a mobile phase with a high percentage of organic solvent is preferred for a hydrophobic analyte.

Silia*Bond*® fluorinated phases behave the same as reversed-phase silica gels in terms of packing into columns. One thing to note is that a slight foaming may be observed when adding the solvent (*more precisely with a MeOH/water mixture*) which is completely normal. For an easier use of this product line, Silia*Sep*™ and Silia*Sep*™ PREMIUM flash cartridges pre-packed with fluorinated silica gels could be good alternatives.

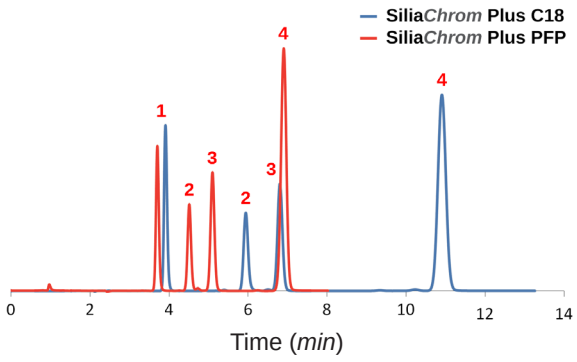
Similar to traditional reversed-phases, it is more difficult to develop a chromatographic method for these types of phases using TLC plates. Therefore, it is recommended to use an HPLC system to determine the best elution conditions for the analytes. To this end, SiliCycle offers Silia*Chrom*® Plus PFP HPLC columns.

PFP phase SiliaChrom® Plus HPLC columns

SiliaChrom Plus PFP offers a different selectivity than the SiliaChrom Plus C18. Indeed, separation on a C18 column is only driven by hydrophobic interactions, whereas a PFP phase brings 4 different types of interactions:



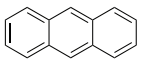
Therefore, the same analysis can lead to different chromatograms (*and sometimes even inversions in peak elution order*) depending on the column selectivity chosen. The chromatogram below underlines the selectivity change of SiliaChrom Plus PFP, compared to SiliaChrom Plus C18. A difference in retention times can be observed, reflecting the decrease of the stationary phase's hydrophobicity. The PFP phase was able to perfectly separate the 4 compounds (*Acetophenone, Toluene, Naphtalene and Anthracene*), a lot faster.



1. Acetophenone

2. Toluene

3. Naphthalene



4. Anthracene

APPLICATION NOTE
#SB006-0

SiliCycle

Fluorinated phases in SiliaPrep™ SPE cartridges

SiliaPrep™ SPE cartridges containing fluorinated silica gel will selectively retain fluorous compounds while non-fluorous ones will be directly eluted out of the cartridge, regardless of the polarity. By using these cartridges, you will be able to get a quick and easy separation of fluorous compounds from different reaction mixtures. This separation method can be applied either for scavengers, protecting groups, tags as well as reagents with fluorine content. This method utilizes an easy two-step elution process based on solvent selectivity (*fluorophobic and fluorophilic solvents*).

The following example shows an experiment done using SiliaPrep™ SPE cartridges packed with SiliaBond® Fluorochrom (*Si-FCM*) nec (*R63730B*). It is a standard demonstration using dyes to show the procedure with SiliaPrep™ SPE Cartridges (*in blue is an organic dye and in yellow a fluorous dye*). The typical loading capacity for these cartridges is 5-15 % of the silica bed weight.

EXTRACTION PROCEDURE:

Sample pre-treatment: Dissolve the sample in the minimum volume required of DMF.

Conditioning step: Wash the cartridge with a small volume of DMF (< *1 column volume*) to remove impurities (*oil*) coming from the cartridge moulding step. Discard collected fraction.

Equilibration step: Wash the cartridge with a "fluorophobic" solvent for better reproducibility.

- Solvent: 80/20 MeOH/H_2O
- Volume: 1-2 column volumes
- Flow rate: 2-4 mL/min

Loading step: Load the prepared sample directly on the top of the cartridge under vacuum (*if using an SPE vacuum manifold or either an SPE vacuum adaptor*) or positive pressure.

Flow rate: same rate as the cartridge volume:

- 1 mL SPE cartridge: 1 mL/min
- 3 mL SPE cartridge: 3 mL/min
- 6 mL SPE cartridge: 6 mL/min, etc.

Washing step: Elute non-fluorous compounds using a "fluorophobic" solvent.

- Solvent: 80/20 MeOH/H_2O
- Volume: 2-3 column volumes
- Flow rate: 1-2 mL/min

Note: water content is very important to prevent the chance of fluorous breakthrough. Acetonitrile and ethanol mixed with at least 20 % of water can also be used as "fluorophobic".

Elution step: Fluorinated compounds are eluted using methanol.

- Solvent: MeOH
- Volume: 2-3 column volumes
- Flow rate: 1 mL/min

Note: acetonitrile can also be used as "fluorophilic" solvent for the elution. More "fluorophilic" solvents like THF and acetone can be employed.

Regeneration step: Wash the stationary phase with appropriate solvent, then dry the cartridge (*air dry sufficient*).

- Solvent: THF or acetone
- Volume: 1-2 column volumes

SILICYCLE
CERTIFIÉE ISO 9001:2015 CERTIFIED

High Value Silica-based and Specialty Products for Chromatography, Analytical and Organic Chemistry, and Purification

DISCOVER OUR BROCHURES

METAL & ORGANIC SCAVENGING

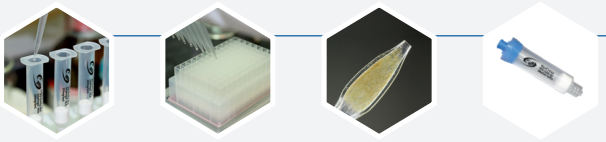
Silia*MetS*® – Metal Scavengers
Silia*Bond*® – Organic Scavengers
***E-PAK*®** – Fixed Bed Flow-Through Purification Cartridges

CHROMATOGRAPHY & PURIFICATION

Silia*Flash*® – Irregular Silica Gels
Silia*Sphere*™ PC – Spherical Silica Gels
Silia*Bond*® – Chromatographic Phases
Silia*Sep*™ – Flash Cartridges
Silia*Plate*™ – TLC Plates

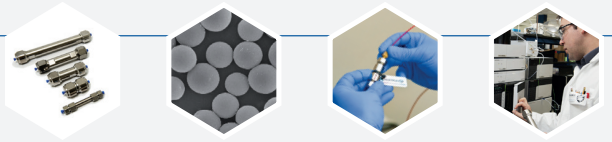
SAMPLE PREPARATION

Silia*Prep*™ – Silica-based SPE Cartridges & Well Plates
Silia*PrepX*™ – Polymeric SPE Cartridges & Well Plates
Silia*Prep*™ – Micro-SPE Tips
Silia*Fast*™ – FaPEx® Cartridges for Pesticide Residue Analysis



ANALYTICAL & PREPARATIVE CHROMATOGRAPHY

Silia*Sphere*™ – Spherical Silica Gels
Silia*Chrom*® – HPLC Columns



ORGANIC SYNTHESIS

Silia*Bond*® – Reagents & Oxidants

R&D SERVICES

Metal & Organic Scavenging Screening
Organic Synthesis
Extraction & Purification
Custom Column Packing
Material Science

SILICYCLE

SiliCycle Inc.
2500, Parc-Technologique Blvd,
Quebec City (Quebec) G1P 4S6
CANADA

Phone: **+1 418.874.0054**
Toll Free **+1 877.745.4292** (*North America only*)
Fax: **+1 418.874.0355**

Email: **info@silicycle.com**
Website: **www.silicycle.com**

Overseas Offices

SiliCycle Europe	europe@silicycle.com
SiliCycle India	india@silicycle.com
SiliCycle Shanghai	contact@silicyclechina.com

Follow us: