

SiliaBond[®]

Chromatographic and
Ion Exchange Phases



SILICYCLE^{Inc.} 
UltraPure **SILICA GELS**

SiliaBond Chromatographic and Ion Exchange Phases

SiliCycle offers a large range of silica-based chromatographic and ion exchange phases:

- Non Polar SiliaBond Phases: C1 to C18
- Polar SiliaBond Phases: Amine, Cyano and Diol
- Ion Exchange SiliaBond Phases: SCX, SCX-2, WCX, SAX, SAX-2 and WAX



SiliaBond Chromatographic Phases

Silica is the most widely used matrix in chromatography. These bare and grafted supports possess great properties for use as stationary phases and are particularly appreciated for their high mechanical resistance. In chromatography, there are two phases: the stationary phase that is packed in a column and the mobile phase that will be eluted through the stationary phase. If the analyte is strongly soluble in the mobile

phase, there will be no retention. If the analyte interacts strongly with the stationary phase, there will be no or low migration. In a mixture, the interactions between the two phases will generate the separation. So, depending on the analyte polarity, the appropriate stationary phase has to be chosen, and the mobile phase polarity has to be tuned.

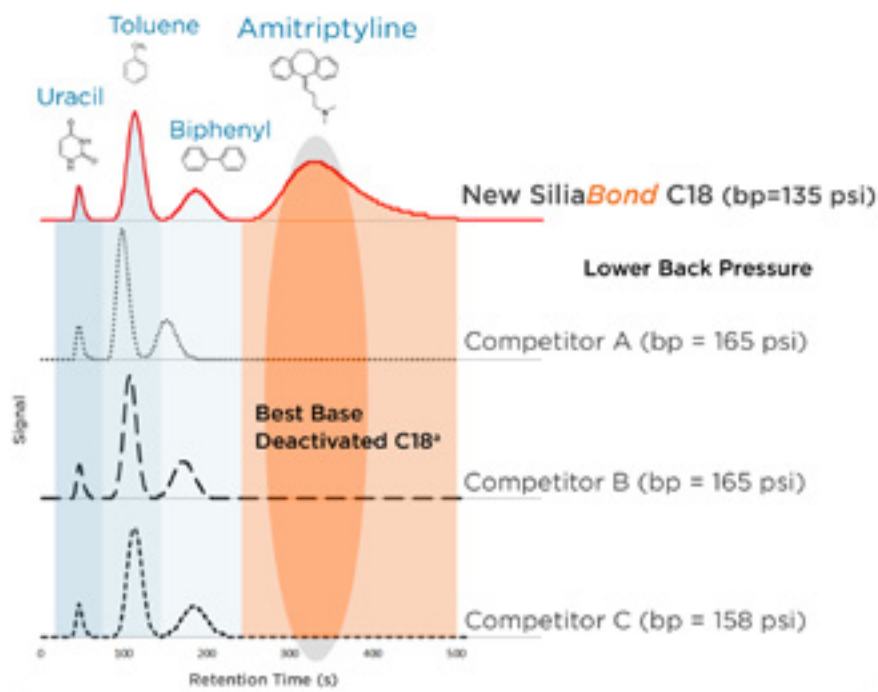
SiliaBond Reversed Phases

In reversed phase chromatography, the packing material is always non-polar (*hydrophobic*) while the mobile phase is polar to non-polar. An important parameter affecting chromatographic efficiency is the hydrophobicity of the sorbent. As a general rule, stationary phase hydrophobicity increases with the alkyl chain length.

Last year, SiliCycle developed a new and innovative C18 chromatographic phase characterized by a homogeneous coverage of the alkyl chains on the surface. Consequently, the endcapping step is more controlled, which leads to much improved separations and also to inhibition of the non-specific interactions with silanol (*highly deactivated silanol phase*). This chromatographic phase is available on

irregular (*R332-*) and spherical (*S032-*) high quality supports. This grafting process will be available soon for all other reversed phases.

Compare to competitive products, this endcapped 17% C18 exhibits high hydrophobicity and base deactivated properties. We have compared this new chromatographic phase to comparable 20% C18 phases on the market. The comparison was done on a mixture of compounds to evaluate the dead volume (*uracil*), the hydrophobicity (*toluene and biphenyl*) and the silanol activity (*amitriptyline*). The test was done in isocratic conditions, with a mobile phase composed of 8/20 methanol/buffer (20 nM potassium phosphate pH = 7). The results are presented below:



The basic product, amitriptyline, interacts with residual silanol groups and stays immobilized on all the competitor phases, but not on the new SiliaBond C18. This new C18 phase presents a better separation property with a better end capped surface. Also, the SiliaBond C18 presents lower back pressure compared to the competition.

SiliaBond Reversed Phases Portfolio

The table below presents all the reversed phases available from SiliCycle:

SiliaBond Reversed Phases					
Sorbent Phase	Functional Group	Endcapping	%C Loading ^a	Density (g/mL)	SiliCycle P/N
C18	Monofunctional C18	Yes	17.0	0.639	R33230B
C18 <i>nec</i>	Monofunctional C18	No	15.5	0.640	R33330B
C18 Low Loading	Monofunctional C18	Yes	11.0	0.619	R33530B
C18 High Loading	Trifunctional C18	Yes	23.0	0.864	R00030B
C18 High Loading <i>nec</i>	Trifunctional C18	No	23.0	0.867	R00130B
C18 Moderate Loading	Trifunctional C18	Yes	17.0	0.735	R02130B
C18 Low Loading	Trifunctional C18	Yes	11.0	0.705	R00430B
C12	Trifunctional Adamantyl	Yes	16.0	0.705	R53030B
C8	Monofunctional C8	Yes	11.0	N/A	R30830B
C8	Trifunctional C8	Yes	12.0	0.759	R31030B
C8 <i>nec</i>	Trifunctional C8	No	11.0	0.703	R31130B
C6	Trifunctional Cyclohexyl	Yes	10.0	0.662	R61530B
C4	Monofunctional C4	Yes	7-8.0	N/A	R32730B
C4	Trifunctional C4	Yes	8.0	0.656	R32030B
C4 <i>nec</i>	Trifunctional C4	No	8.0	0.692	R32130B
C1	Methyl	Yes	5.0	0.599	R33030B
CN	Trifunctional Cyano	Yes	7.0	0.703	R38030B
PHE	Monofunctional Phenyl	Yes	9.0	N/A	R33830B
PHE	Trifunctional Phenyl	Yes	9.0	0.637	R34030B
PHE <i>nec</i>	Trifunctional Phenyl	No	9.0	0.607	R34130B
PFP	Pentafluorophenyl	Yes	9.0	N/A	R67530B

Also available on all irregular SiliaFlash Silica. Example: the 300 Å, 40-63 µm (Rxxx30M) ^aBased on our Standard SiliaFlash Silica matrix R10030B, 40-63 µm, 60 Å

Typical applications using SiliaBond Reversed Phases

Sorbent Phase	Typical Applications
C18	Peptides, pesticides, PCBs, PAHs, toxins, drugs & their metabolites in physiological fluids
C8	Highly hydrophobic pesticides, peptides, heavy drugs and their metabolites in physiological fluids
C6 (cyclohexyl)	Phenols, chloroanilines and anthelmintics from tissues and water
C4	Molecules with large hydrophilic region such as peptides, proteins and zwitterions (300 Å)
C1	Polar and non-polar pharmaceutical natural products, highly hydrophobic molecules and biomolecules
CN	Cyclosporine and carbohydrates
PHE	Aflatoxins, caffeine, and phenols from water
PFP	Conjugated compounds or for a new selectivity approach

SiliaBond Normal Chromatographic Phases

Normal-phase chromatography is used to separate polar compounds through polar interactions with the support. The interactions take place on the highly polar silanols of the silica gel surface, but there are also moderately polar interactions with the hydrogen bonds on amino or diol functions. The non-encapped cyano phase can be used in applications in normal-phase chromatography as a less polar alternative to silica. The AgNO₃ phase is particularly useful to separate isomers that present unsaturated groups.

SiliaBond Normal Chromatographic Phases

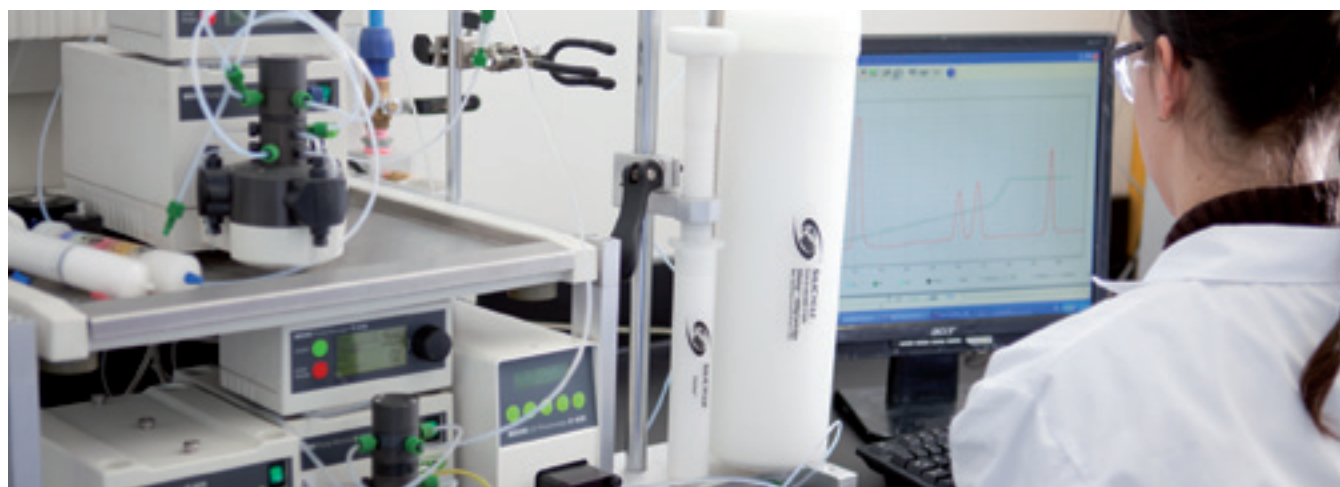
Sorbent Phase	Functional Group	Endcapping	Loading ^a	Density (g/mL)	SiliCycle P/N
SiO ₂	Bare silica gel	No	N/A		R10030B
NH ₂ <i>nec</i>	Amine	No	1.6	0.687	R52130B
CN <i>nec</i>	Cyano	No	1.0		R38130B
Diol <i>nec</i>	Diol	No	1.0	0.687	R35030B
AgNO ₃	Silver Nitrate	No	10% w/w	0.604	R23530B

Also available on all irregular SiliaFlash Silica. Example: the 300 Å, 40-63 µm (Rxxx30M)

^a Based on our Standard SiliaFlash Silica matrix R10030B, 40-63 µm, 60 Å

Typical applications using SiliaBond Normal Chromatographic Phases

Sorbent Phase	Typical Applications
NH ₂ <i>nec</i>	Sugars, nucleotides and water-soluble vitamins
CN <i>nec</i>	Polar organic compounds such as basic drugs and molecules containing π electron systems
Diol <i>nec</i>	Peptides, proteins and malto-oligosaccharides
AgNO ₃	Cis/trans isomers of unsaturated compounds such as alkenes, lipids, steroids and terpenes



SiliaBond Ion Exchange Phases

In an ion exchange process, the silica support is modified by a function carrying a charge with its counter ion. This counter ion is exchangeable with other ions in solution. If the immobilized phase is carrying an anion, the exchangeable species is a cation. Inversely, if the immobilized phase carries a cation, the ion exchangeable species will be an anion. Ion exchange phases are widely used in separation, purification and decontamination.

The stationary phase can be a cation exchanger of varying strength:

- Strong cation exchanger such as our SiliaBond Tonic Acid (SCX) and SiliaBond Propylsulfonic Acid (SCX-2)
- Weak cation exchanger such as our SiliaBond Carboxylic Acid (WCX)

The stationary phase can also be an anion exchanger of varying strength:

- Strong anion exchanger such as our SiliaBond TMA Chloride *nec* (SAX), SiliaBond TMA Acetate *nec* (SAX-2) and SiliaBond TBA Chloride
- Weak Anion exchanger such as our SiliaBond Amine *nec* (WAX) and SiliaBond Diethylamine *nec* (WAX-2)

SiliCycle has recently developed SiliaBond TMA Acetate, which has been particularly effective in customers' anionic exchange applications.

SiliaBond Ion Exchange Phases					
Sorbent Phase	Functional Group	Endcapping	Loading (mmol/g) ^a	Density (g/mL)	SiliCycle P/N
WAX	Amine	No	1.60	0.687	R52130B
WAX-2	Diethylamine	No	1.20	0.761	R76630B
SAX	Trimethylammonium Chloride	No	1.10	-	R66230B
SAX-2	Trimethylammonium Acetate	No	0.70	0.707	R66430B
TBA Chloride	Tributylammonium Chloride	No	0.50	0.656	R65530B
SCX	Tonic Acid	No	0.80	-	R60430B
SCX-2	Propylsulfonic Acid	No	1.00	0.642	R51430B
WCX	Carboxylic Acid	No	1.40	6.682	R70130B

Also available on all irregular SiliaFlash Silica. Example: the 300 Å, 40-63 µm (Rxxx30M)

^a Based on our Standard SiliaFlash Silica matrix R10030B, 40-63 µm, 60 Å

SiliaBond Ion Exchange Phases (con't)

Typical applications for using SiliaBond Ion Exchange Phases

Sorbent Phase	Typical Applications
SiliaBond Amine (WAX)	A weak anion exchanger with pKa of 9.8. At pH 7.8 or below, the functional groups are positively charged. It facilitates the rapid release of very strong anions such as sulfonic acids that may be retained irreversibly on SAX.
SiliaBond Diethylamine (WAX-2)	With a pKa of 10.5, this phase is preferred over the SiliaBond TMA Chloride (SAX) when performing catch and release purification of compounds bearing a permanent negative charge such as salts of sulfonic acids. Using SAX in this case could make the release of the compounds of interest difficult (<i>but not necessarily impossible</i>), not to say irreversible, due to the strong interaction between the two strong ions.
SiliaBond TMA Chloride (SAX)	The quaternary amine is permanently charged (<i>pH independent</i>). It is commonly used for the extraction of weak cations (<i>such as carboxylic acids</i>) that may not bind strongly enough to weaker anion exchangers.
SiliaBond TMA Acetate (SAX-2)	The acetate counter ion is easily exchangeable (<i>so than the chloride ion</i>) for compounds with pKa < 5, such as carboxylic acids. This phase can be used in organic chemistry applications to selectively purify acidic compounds or remove acidic impurities from reaction mixtures.
SiliaBond TBA Chloride	SiliaBond TBA Chloride may be used in the same applications as SiliaBond TMA Chloride. This phase is more sterically hindered, which offers a different selectivity than other anion exchangers.
SiliaBond Tosic Acid (SCX)	Due to the very low pKa (< 1) these functions are strong cation exchangers since they maintain a negative charge throughout the pH scale. The most common use is likely for catch and release purification.
SiliaBond Propylsulfonic Acid (SCX-2)	
SiliaBond Carboxylic Acid (WCX)	At a pH of 6.8 or above, this weak cation exchanger carries a negative charge. A pH of 2.8 or below is needed for easier elution of strong cationic analytes that are neutralized only at extreme basic conditions. This phase is commonly used for the extraction of strong cationic species, which would be irreversibly retained on strong cation exchangers.

