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SILICYCLE
UltraPure SILICA GELS

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2008 catalog Si SILICYCLE

2008 catalog

Purification and Synthesis
Silica gels, Cartridges, SPE, TLC,
Scavengers and Reagents

SILICYCLE
UltraPure SILICA GELS

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Word from the President

Dear valued Customers and Partners,

You have in your hands our new 2008 catalog. It is not the only thing that is new at SiliCycle®. Indeed since the last edition of our catalog, SiliCycle® has gone through remarkable growth. We have experienced marked increases in sales and expanded our customer base and product portfolio, and we have solidified our distribution network by teaming up with partners worldwide that have great knowledge and experience in chromatography and in organic synthesis.

This trend shows no sign of slowing down. In 2008, SiliCycle® will move into its new state of the art facility to be able to sustain its growth. Our production capacity will quadruple. Also we are looking to establish production facilities, warehouses and technical support centers in the USA, Europe and Asia. All of this to better serve our customers and partners. It also means that we are looking to hire exceptional people with great knowledge in chromatography, organic synthesis, and sales. Please have a look in the corporate section of our web site to see our job openings (www.silicycle.com/html/english/corporate).

Quality, as always, continues to be of paramount importance. SiliCycle® will obtain the ISO 9001:2000 certification before the end of 2007. This is important for us because it shows our determination to continuously improve ourselves and our products.

Silia*Bond*® supported scavengers and reagents, **Silia*Flash*®** silica gels, **Silia*Sphere*™** spherical silica gels, **Silia*Sep*™** flash cartridges, **Silia*Prep*™** SPE, **Silia*Plate*™** TLC, and IMPAQ® preparative silica gels; all silica based-products that makes SiliCycle® one of major player in chromatography and the world leader in functionalized silica gels for organic synthesis.

The environment being a corporate priority, SiliCycle® is striving towards reducing the impact of its production on the environment and constantly improves the company's environmental management by investing a part of its operating expenses in environmental projects.

To conclude I'd like to join all of SiliCycle®'s team in thanking you for your support and your continuous trust. You're the reason behind SiliCycle®'s success.

Thank you for doing business with us!

Sincerely,

Hugo St-Laurent
President, CEO

OPPORTUNITIES FOR DISTRIBUTORS

SiliCycle® has established many partners-distributors in different countries. However there are still market sectors where we are actively looking for partners. Also SiliCycle® is interested in working with you to supply custom products to support your vision and to serve your customers. Contact us and we will be happy to explore the possibilities with you.

Abbreviations

BEMP ····· 2-*tert*-Butylimino-2-diethylamino-1,3-dimethyl-perhydro-1,3,2- diazaphosphorine
^{13}C NMR ···· Carbon Nuclear Magnetic Resonance
DAC ····· Dynamic Axial Compression
DCC ····· Dicyclocarbodiimide
DCM ····· Dichloromethane
DCU ····· 1,3-Dicyclohexylurea
DIPEA ····· *N, N*-Diisopropylethylamine
DMAP ····· 4-Dimethylaminopyridine
DMF ····· *N, N*-Dimethylformamide
ec ····· endcapped
GC-FID ··· Gas Chromatography –Flame Ionization Detector
GC-MS ····· Gas Chromatography – Mass Spectra
^{1}H NMR ····· Proton Nuclear Magnetic Resonance
HP ····· High Performance
HOBt ····· 1-Hydroxybenzotriazole
HPLC ··· High Performance Liquid Chromatography
IEC ····· Ion Exchange Chromatography
IMPAQ® ····· Preparative Angular Silica
MeCN ····· Acetonitrile
MeOH ····· Methanol
nec ····· non-endcapped
PC ····· Preparative Chromatography
PCC ····· Pyridinium Chlorochromate
PDC ····· Pyridinium Dichlorochromate
SAX ····· Strong Anion Exchanger
SCX ····· Strong Cation Exchanger
SEM ····· Scanning Electron Microscopy
SFC ····· Supercritical Fluid Chromatography
Silia*Bond*® ····· Functionalized Silica Gels
Silia*Cat*® ····· Heterogeneous Catalysts
Silia*Flash*® ····· Irregular Silica Gels
Silia*Plate*™ ····· Thin Layer Chromatography Plates
Silia*Prep*™ ····· SPE Cartridges and 96 Well Plates
Silia*Sep*™ ····· Flash Purification Cartridges
Silia*Sphere*™ ····· Analytical Spherical Silica Gels
SPE ····· Solid Phase Extraction
TAAcOH ····· Triaminetetraacetic acid
TAAcONa ····· Triaminetetraacetate sodium salt
TBACl ····· Tetrabutylammonium chloride
TBD ····· 1,5,7-Triazabicyclo[4.4.0]dec-5-ene
TEA ····· Triethylamine
TEMPO ····· 2,2,6,6-Tetramethylpiperidine 1-oxyl
THF ····· Tetrahydrofuran
TMACl ····· Tetramethylammonium chloride
TLC ····· Thin Layer Chromatography
UPLC ··· Ultra Performance Liquid Chromatography
WAX ····· Weak Anion Exchanger
WCX ····· Weak Cation Exchanger

SiliaBond® and SiliaCat® category listing

ION EXCHANGERS

METAL SCAVENGERS

NUCLEOPHILE SCAVENGERS

OXIDANTS

REAGENTS

OTHERS

SiliaBond® and SiliaCat® alphabetical listing (FUNCTIONALIZED SILICA GELS)

Application notes index

SILIAFLASH®

2008 catalog

UltraPure Silia*Flash*® silica gel

TIGHT PARTICLE SIZE DISTRIBUTION

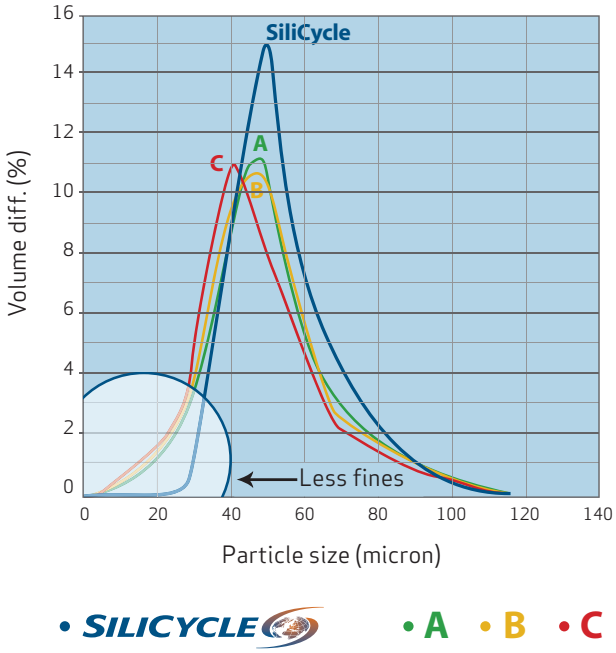
Silia*Flash*® is a very high purity acid washed silica gel with tight particle size distribution. The particle size of the standard 40-63 microns flash chromatography product *(Silia*Flash*® F60)* compares favorably with the overall industry average of 35-75 microns. This ensures more uniform flash column or cartridge packing, as well as better resolutions and separations.

The importance of the particle size distribution varies depending on the type of chromatography being done. For instance, it is very important for HPLC that the particle size distribution of the spherical particles being used be very narrow.

When selecting a silica gel, chemists need to remember that not all 40-63 µm gels are the same. The figure on the right shows the distribution curves of SiliCycle®'s Silia*Flash*® gel compared to three other manufacturers of flash silica gels. All products were sold as 40-63 µm gels.

The two key points of the graph are the height of the volume differential (diff) and percentage of particles below 40 µm. The SiliCycle® curve has a much higher percentage of particles between 40-63 µm and a very low level of small particles below 40 µm (or "fines"). Fines increase backpressure that can result in clogging which is particularly dangerous when using glass columns. Fines can also pass through filters and contaminate final products. No fines give a more regular, stable, and reproducible chromatography bed that gives a faster and more even flow rate for better separation. SiliCycle® has the lowest level of fines on the market.

Comparison of particle size distribution between SiliCycle® and three competitors



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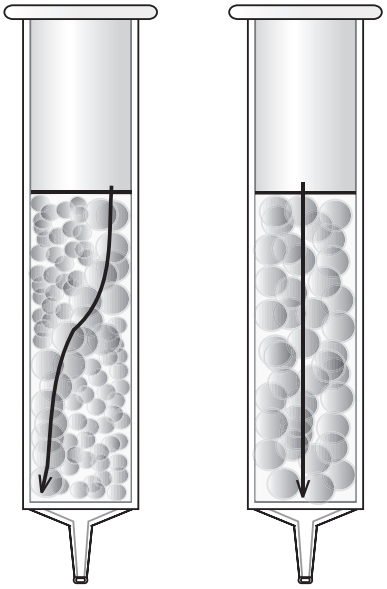
Today almost all silica gel manufacturers sell a form of 40-63 µm gel but not all gels are equal. SiliCycle®'s **Silia***Flash*® gels have a mean of 90% of the particles in the nominal range in comparison with 80% for most of the competitors. The connection between particle size distribution and column performance is very simple. When the range is broad the column packing is uneven. Some parts are composed of only large particles where the solvent will flow fast and meet little resistance, and there are sections composed of small particles where the solvent flows slowly and meets great resistance. As a result, the solvent will take the path of least resistance through the column and flow around the pockets of small particles instead of straight through the column. This uneven flow greatly affects the separation because the compounds will have different retention times depending on their flow path through the column when they merge. As they exit the column, the compounds will give broad and poorly separated peaks.

The figure below visually illustrates the effect of a wide versus a narrow particle size distribution. A narrower particle size distribution will give a more homogenous packing that will help in collecting more concentrated fractions and in reducing solvent consumption, which will ultimately be more cost-efficient.

Effect of fines (left) on elution compared with silica gel that has none (right)



LOW TRACE METAL CONTENT

Irregular silica, dependent on its method of manufacturing, normally contains trace quantities of a variety of metals, which in turn can affect the separation. Sodium, iron and lead are particularly problematic and cause peak tailing. SiliCycle® proprietary technology generates a silica gel with the lowest trace metal content on the market today. As shown in the table presented on the next page, trace metal concentration in SiliCycle®'s silica gel is significantly lower when compared to flash silica gels from other manufacturers. Our low trace metal content ensures you get optimal performance from your chromatography. Tight control of trace metals on every batch also improves your reproducibility and reduces risks of interaction between metals and desired compounds.

The narrow particle size distribution, the low level of fines and the reduced concentration of trace metals offered by SiliCycle® optimize your separating power, saving you time and money.

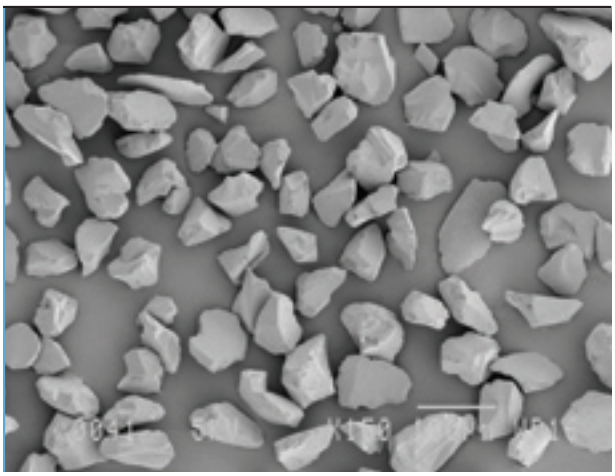
Silia*Flash*® silica gels also offer these advantages over competitors' products:

Neutral pH: The pH of our **Silia***Flash*® silica gels is kept between 6.5 and 7.5. This compares advantageously with the pH of 6.0 to 7.0 often seen in the market. A neutral pH is needed to separate pH sensitive compounds. SiliCycle® can adjust the pH upon request to help protect pH sensitive products that request a lower pH.

Stable water level content: Water level of silica gel is affecting the selectivity of the silica. SiliCycle® **Silia***Flash*® has a water content of 4.0 to 6.0%. This compares advantageously with the other products available that have water variation of 2% to 9% depending on the manufacturer. SiliCycle® can adjust the water level upon request.

High surface area: Higher surface area provides higher separation power.

Available in pre-packed SPE and flash cartridges: We are using **Silia***Flash*® as a standard sorbent in the **Silia***Sep*™ flash cartridges and **Silia***Prep*™ SPE. It is also our standard starting material for the **Silia***Bond*® functionalized silica gels.



Typical trace metal concentration

Metals	SiliCycle®	Manufacturer A	Manufacturer B
Aluminum (Al)	33	262	280
Barium (Ba)	9.4	59.7	32.5
Calcium (Ca)	336	1150	502
Chromium (Cr)	0.5	0.6	0.4
Copper (Cu)	0.2	0.2	0.2
Iron (Fe)	32	75	41
Lead (Pb)	0.41	5.24	0.95
Magnesium (Mg)	61	149	104
Nickel (Ni)	0.4	0.5	0.5
Silver (Ag)	0.09	0.29	0.19
Sodium (Na)	466	945	585
Tin (Sn)	0.2	0.2	0.1
Titanium (Ti)	147	250	179
Zinc (Zn)	1	1	2
Zirconium (Zr)	32	75	56

All numbers are in ppm.

SiliaFlash® Products (FORMATS: 1kg, 5kg, 10kg, 25kg*)

Product Number	Name	Particle size distribution (µm)	Pore size (Å)	Specific surface area (m^2/g)
R10015A	SiliaFlash® S40	15 – 35	40	600
R10030A	SiliaFlash® F40	40 – 63	40	600
R10040A	SiliaFlash® G40	60 – 200	40	600
R10070A	SiliaFlash® B40	200 – 500	40	600
R10080A	SiliaFlash® N40	500 – 1000	40	600
R10010B	SiliaFlash® C60	0 – 20	60	500
R10014B	SiliaFlash® T60	5 – 20	60	500
R10019B	SiliaFlash® U60	10 – 30	60	500
R10013B	SiliaFlash® I60	15 – 25	60	500
R10015B	SiliaFlash® S60	15 – 35	60	500
R10017B	SiliaFlash® E60	15 – 40	60	500
R10023B	SiliaFlash® H60	20 – 45	60	500
R10030B	SiliaFlash® F60	40 – 63	60	500
R12030B	SiliaFlash® P60	40 – 63 (academic grade)	60	500
R10050B	SiliaFlash® M60	60 – 120	60	500
R10040B	SiliaFlash® G60	60 – 200	60	500
R10060B	SiliaFlash® L60	120 – 200	60	500
R10070B	SiliaFlash® B60	200 – 500	60	500
R10080B	SiliaFlash® N60	500 – 1000	60	500
R10015D	SiliaFlash® S90	15 – 35	90	400
R10030D	SiliaFlash® F90	40 – 63	90	400
R10040D	SiliaFlash® G90	60 – 200	90	400
R10080D	SiliaFlash® N90	500 – 1000	90	400
R10040H	SiliaFlash® G150	60 – 200	150	300
R10050H	SiliaFlash® M150	60 – 120	150	300
R10060H	SiliaFlash® L150	120 – 200	150	300
R10072H	SiliaFlash® B150	250 – 500	150	300
R10030M	SiliaFlash® F300	40 – 63	300	100
R10040M	SiliaFlash® G300	60 – 200	300	100

All these products are available in different sizes from lab scale to multi-ton scale.

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SILIACARTRIDGER™

2008 catalog

The **Silia***Cartridger*™ is a small equipment that allows the packing of flash cartridges quickly and effectively. These cartridges can then be used on any commercial equipment with the use of simple adapters. Each cartridge can be packed in less than 30 seconds. The packed cartridges give very good separations on par with commercially available cartridges at a fraction of the price. You can choose between a large fluidization vessel for standard flash silica gel and a small one for packing reversed-phase materials.

Silia*Cartridger*™ *with* ***Small*** *fluidization vessel*

Silia*Cartridger*™ *with* ***Large*** *fluidization vessel*

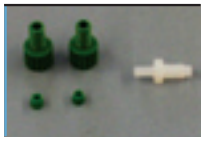
Fluidization vessel size	Small	Large
PN (order)	HDW200	HDW202
Dimensions (w × h × d) mm	220 × 315 × 450	
Empty weight	7.5kg	
Cartridge size	All four sizes available	
Requirements	• Oil free pressurized gas/air ~ 1 bar (15 psi), flow: ~ 200 L/h • Vacuum source (flow: ≥ 1 m³/h) ≤ 20 mbar (15 mm Hg)	
Hose connections	ID 6 mm for gas/air and vacuum	

Type	# 1	# 2	# 3	#4
PN (order)	HDW212	HDW210	HDW216	HDW214
Dimensions (ID × L) mm	12 × 150	12 × 75	40 × 150	40 × 75
Cartridge	High transparent and chemical resistant Polypropylene cartridges			
Frit	Porous High Density Polyethylene (20 µm)			
Silia*Flash*® packing weight	~ 8.0g	~ 4.0g	~ 90.0g	~ 40.0g
Sample range purification	≤ 400mg	≤ 200mg	≤ 5.0g	≤ 1.0g

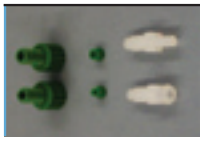
12 mm Adapter Set

40 mm Adapter Set

Biotage™ Adapter



FlashMaster™ Adapter



Isco™ Adapter

Adapter Set	12 mm	40 mm
PN (order)	HDW230	HDW232
Use	For fast installation of cartridges into flash chromatography systems	

Compatibility	Biotage™	FlashMaster™	Isco™
PN (order)	HDW240	HDW242	HDW244
Use	Enables the easy integration of the SiliCycle® cartridge adapter set into existing technologies		

SILIAPLATE™

2008 catalog

UltraPure SiliaPlate™ TLC

Optimize your separation conditions by using the same silica as in your flash column or cartridge. Maximize your benefit by using our ***UltraPure*** **Silia*Plate***™ Thin Layer Chromatography (TLC) plates with an extra hard layer of silica. For your convenience we offer different sizes and choice of backings. We also offer reversed and specialty phases plates.

All our **Silia*Plate***™ TLC have F254 indicator.

SiliaPlate™ Al (aluminum)

Product Number	Size (cm)	Thickness (µm)	Quantity per box
TLA-R10011B-323	20 × 20	250	25

SiliaPlate™ G (glass)

Product Number	Size (cm)	Thickness (µm)	Quantity per box
TLG-R10011B-323	20 × 20	250	25
TLG-R10011B-124	2.5 × 7.5	250	100
TLG-R10011B-424	5 × 20	250	100
TLG-R10011B-624	2.5 × 10	250	100
TLGSR10011B-723	10 × 20 (scored)	250	25

SiliaPlate™ Prep (glass preparative)

Product Number	Size (cm)	Thickness (µm)	Quantity per box
TLG-R10011B-341	20 × 20	1000	25

SiliaPlate™ C18

Product Number	Size (cm)	Thickness (µm)	Quantity per box
TLG-R30411B-213	10 × 10	200	25

SiliaPlate™ CN (cyano)

Product Number	Size (cm)	Thickness (µm)	Quantity per box
TLG-R38011B-203	10 × 10	150	25

SiliaPlate™ NH_2 (amine)

Product Number	Size (cm)	Thickness (µm)	Quantity per box
TLG-R52011B-203	10 × 10	150	25

SiliaPlate™ Ag (10% silver nitrate impregnated)

Product Number	Size (cm)	Thickness (µm)	Quantity per box
TLG-R23511B-423	5 × 20	250	25

SiliaSep™

2008 catalog

SiliaSep™ Cartridges

SiliaSep™ cartridges are compatible with every commercially available system.

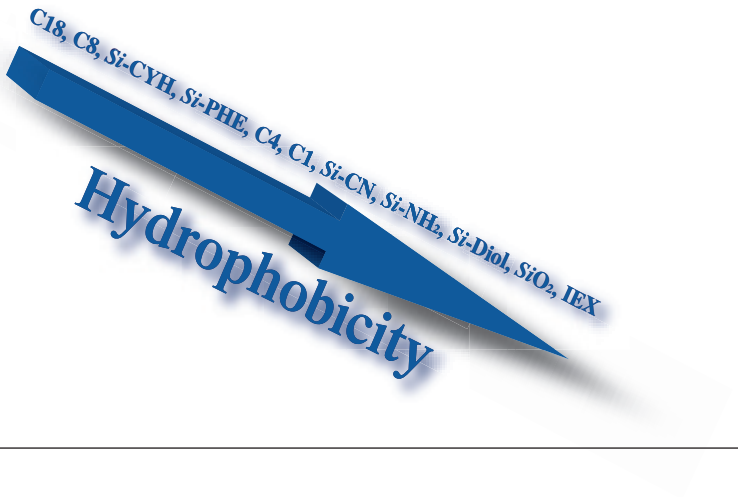
Use the superior performance of our *UltraPure* SiliaFlash® and SiliaBond® silica gels in all your separations with our pre-packed SiliaSep™ cartridges. You can increase the reproducibility of your separations by using the same silica gel for all your flash systems.

SiliCycle® offers a full line of cartridges compatible with existing technologies: Isco™ (SiliaSep™), Biotage™ (SiliaSep™ BT), FlashMaster™ (SiliaSep™ OT) and Büchi™ (SiliaSep™ BU) systems.

By using our SiliaSep™ cartridges, you will not only save time and money but you will also generate higher purity samples and reduce the number of false positives in your screening, resulting in higher quality data. All our flash cartridges are packed with sorbents based on our fines-free *UltraPure* SiliaFlash® silica gel which has the highest purity on the market. When using our SiliaSep™cartridges, we guarantee you the following benefits:

- Excellent packing and storage qualities
- Very good separation and tighter peaks
- Less time and solvent spent conditioning the cartridge
- No silica contamination in your final product
- Higher recoveries
- Highly controlled pH, water and metal content from lot-to-lot
- Reproducible and faster flow rate
- Wide variety of sorbents available (SiliaBond® phases)

To better suit your needs, SiliCycle® SiliaSep™ cartridges are available in a wide variety of sorbents: normal phase, reversed-phase, ion exchanger, and fluorous phase. The hydrophobicity of the sorbent is an important parameter when selecting the sorbent. As a general rule, the stationary phase hydrophobicity decreases as follow:



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SiliaSep™ CARTRIDGES CHARACTERISTICS

Our cartridges present a high purity alternative to the other cartridges available on the market. SiliCycle®'s cartridges are 100% compatible with commercially available systems. All our SiliaSep™ flash cartridges are packed with SiliaFlash® F60 (40-63µm, 60Å) as standard sorbent. You will have great value because our cartridges present the following advantages:

Characteristics of SiliaSep™ Cartridges

- Very high purity and quality silica gels
- Clean, pre-packed, disposable polypropylene (or high density polyethylene) cartridges
- Leak-free guaranteed
- Choice of many types of packing
  - Choice of pore sizes and particle size diameters
- Choice of bonded phases (SiliaBond® phases)
  - C18, Amine, Cyano, Diol, Fluorochrom and SCX-2
  - scavengers and other phases available upon request
- Faster separations with improved band definition
- Safe
  - No glass handling or washing
  - No breakage
- Excellent pricing and volume discounts available
- Readily available from inventory for many volumes

SiliaSep™
(UNIVERSAL CLOSED TOP LUER-LOCK CARTRIDGES)
Isco™ Compatible

SiliaSep™ available silica weights:

- 4g
- 12g
- 25 g
- 40g
- 80g
- 120 g
- 220 g
- 330 g
- 750 g
- 1500 g

NEW

Also available: SiliaSep™ HP, *packed with our* ***UltraPure*** *small particle* SiliaFlash®.

SiliaSep™ BT
Biotage™ i Compatible

SiliaSep™ BT available sizes:

- 12S and 12M
- 40S, 40M and 40L
- 65
- 75S, 75M and 75L

SiliaSep™ OT
FlashMaster™ Compatible

SiliaSep™ OT available silica weights:

- 2, 5, 10, 15, 20, 25, 50, 70, 100 and 200g
- Other sorbent weights are available upon request

SiliaSep™ OT column volumes:

- 12, 25, 70, 150, 276 and 377mL

SiliaSep™ BU
Büchi™ Compatible

SiliaSep™ BU available sizes:

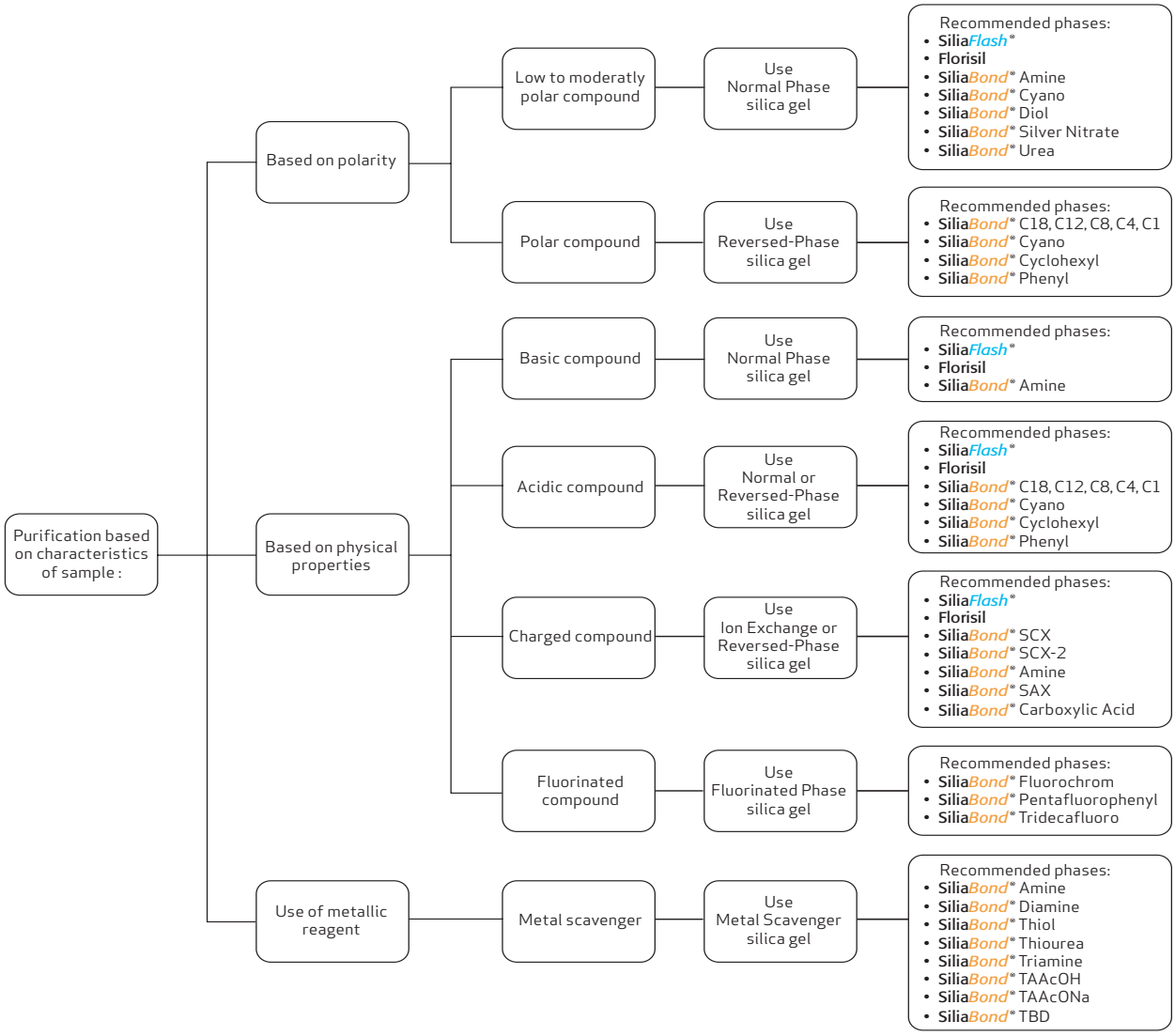
- 12S and 12M
- 40S and 40M

Visit our website for new metal scavengers and application notes.

Our flash chromatography products offer high levels of performance and reproducibility. The flash media consistently has a tight particle size distribution with low fines and extremely low metal ion content, for more predictable and easier separations.

Using SiliCycle® **Silia***Sep*™ cartridges, you can purify your sample based on different mechanisms depending on the properties of your compound. Because we can provide cartridges packed with our **Silia***Bond*® functionalized silica gel, you can choose exactly the cartridge that will fit your need. The chart below is designed to serve as a guide in sorbent selection by considering the characteristics of your sample.

Silia*Bond*® selection chart for flash chromatography and metal scavenging



SiliaSep™ Cartridges

Silica Mass	4 g	12 g	25 g	40 g	80 g
flash cartridge max pressure	Closed-Top 4g 60 psi	Closed-Top 12g 60 psi	Closed-Top 25g 100 psi	Closed-Top 40g 100 psi	Closed-Top 80g 100 psi
SiliaSep™	FLH-R10030B-ISO04	FLH-R10030B-ISO12	FLH-R10030B-ISO25	FLH-R10030B-ISO40	FLH-R10030B-ISO80
NEW SiliaSep™ HP	FLH-R10017B-ISO04	FLH-R10017B-ISO12	FLH-R10017B-ISO25	FLH-R10017B-ISO40	FLH-R10017B-ISO80
	20/box	20/box	15/box	15/box	12/box
Dimensions ID x L (mm)	**12.3x60**	**21.1x77**	**21.0x129**	**26.7x127**	**31.0x194**
SiliaSep™ Cartridges	2/box	1/box	1/box	1/box	1/box
SiliaSep™ C18 nec (23%) (see p.33 for phase description)	FLH-R30130B-ISO04	FLH-R30130B-ISO12	FLH-R30130B-ISO25	FLH-R30130B-ISO40	FLH-R30130B-ISO80
SiliaSep™ C18 (17%) (see p.33 for phase description)	FLH-R30230B-ISO04	FLH-R30230B-ISO12	FLH-R30230B-ISO25	FLH-R30230B-ISO40	FLH-R30230B-ISO80
SiliaSep™ Amine (see p.35 for phase description)	FLH-R52030B-ISO04	FLH-R52030B-ISO12	FLH-R52030B-ISO25	FLH-R52030B-ISO40	FLH-R52030B-ISO80
SiliaSep™ Cyano (see p.38 for phase description)	FLH-R38030B-ISO04	FLH-R38030B-ISO12	FLH-R38030B-ISO25	FLH-R38030B-ISO40	FLH-R38030B-ISO80
SiliaSep™ Diol (see p.42 for phase description)	FLH-R35030B-ISO04	FLH-R35030B-ISO12	FLH-R35030B-ISO25	FLH-R35030B-ISO40	FLH-R35030B-ISO80
SiliaSep™ Fluorochrom (see p.43 for phase description)	FLH-R63730B-ISO04	FLH-R63730B-ISO12	FLH-R63730B-ISO25	FLH-R63730B-ISO40	FLH-R63730B-ISO80
SiliaSep™ SCX-2 (see p.49 for phase description)	FLH-R51230B-ISO04	FLH-R51230B-ISO12	FLH-R51230B-ISO25	FLH-R51230B-ISO40	FLH-R51230B-ISO80

For solid load cartridges, see SiliaSep™ accessories on page 21.

Our new high performance SiliaSep™ HP flash cartridges are packed with small particle silica gel. These cartridges give extremely high resolution and increased loading compared to our regular cartridges. These are perfect when some compounds elute close to one another and for your most difficult separations.

SiliaSep™ Cartridges (continued)

Silica Mass	120 g	220 g	330 g	750 g	1500 g
flash cartridge max pressure	Closed-Top 120g 100 psi	Closed-Top 220g 100 psi	Closed-Top 330g 100 psi	Closed-Top 750g 60 psi	Closed-Top 1500g 60 psi
SiliaSep™	FLH-R10030B-IS120	FLH-R10030B-IS220	FLH-R10030B-IS330	FLH-R10030B-IS750	FLH-R10030B-I1500
SiliaSep™ HP	FLH-R10017B-IS120				
	10/box	4/box	4/box	2/box	2/box
Dimensions ID x L (mm)	**36.1 x 211**	**60.2 x 141**	**60.2 x 211**	**77.2 x 337**	**103 x 372**
SiliaSep™ Cartridges	1/box	1/box	1/box	1/box	1/box
SiliaSep™ C18 nec (23%) (see p.33 for phase description)	FLH-R30130B-IS120	FLH-R30130B-IS220	FLH-R30130B-IS330	FLH-R30130B-IS750	FLH-R30130B-I1500
SiliaSep™ C18 (17%) (see p.33 for phase description)	FLH-R30230B-IS120	FLH-R30230B-IS220	FLH-R30230B-IS330	FLH-R30230B-IS750	FLH-R30230B-I1500
SiliaSep™ Amine (see p.35 for phase description)	FLH-R52030B-IS120	FLH-R52030B-IS220	FLH-R52030B-IS330	FLH-R52030B-IS750	FLH-R52030B-I1500
SiliaSep™ Cyano (see p.38 for phase description)	FLH-R38030B-IS120	FLH-R38030B-IS220	FLH-R38030B-IS330	FLH-R38030B-IS750	FLH-R38030B-I1500
SiliaSep™ Diol (see p.42 for phase description)	FLH-R35030B-IS120	FLH-R35030B-IS220	FLH-R35030B-IS330	FLH-R35030B-IS750	FLH-R35030B-I1500
SiliaSep™ Fluorochrom (see p.43 for phase description)	FLH-R63730B-IS120	FLH-R63730B-IS220	FLH-R63730B-IS330	FLH-R63730B-IS750	FLH-R63730B-I1500
SiliaSep™ SCX-2 (see p.49 for phase description)	FLH-R51230B-IS120	FLH-R51230B-IS220	FLH-R51230B-IS330	FLH-R51230B-IS750	FLH-R51230B-I1500

Recommended flow rate and loading capacity.

SiliaSep™ Cartridges	Flow rate mL/min	Loading capacity (g)
4 g	15 – 25	0.005 – 0.40
12 g	20 – 40	0.015 – 1.20
25 g	20 - 45	0.025 - 2.50
40 g	25 – 50	0.05 – 4.0
80 g	40 – 80	0.10 – 8.0
120 g	60 – 120	0.15 – 12
220 g	60 – 180	0.25 – 22
330 g	80 – 180	0.50 – 33
750 g	200 - 300	0.75 - 75
1500g	350 - 450	1.50 - 150

Loading capacity depends strongly on sample mixture and elution protocol.

SiliaSep™ BT Cartridges

	12S	12M	40S	40M	40L
Dimensions ID × L (mm)	12S Flash Cartridge 12 × 75	12M Flash Cartridge 12 × 150	40S Flash Cartridge 40 × 75	40M Flash Cartridge 40 × 150	40L Flash Cartridge 40 × 200
SiliaSep™ BT	FLH-R10030B-12iS	FLH-R10030B-12iM	FLH-R10030B-40iS	FLH-R10030B-40iM	FLH-R10030B-40iL
	20 / box	20 /box	12 / box	12 / box	12 / box
Phase	**12S**	**12M**	**40S**	**40M**	**40L**
SiliaSep™ BT Cartridges	2 / box	2 / box	1 / box	1 / box	1 / box
SiliaSep™ BT C18 nec (23%) (SEE P.33 FOR PHASE DESCRIPTION)	FLH-R30130B-12iS	FLH-R30130B-12iM	FLH-R30130B-40iS	FLH-R30130B-40iM	FLH-R30130B-40iL
SiliaSep™ BT C18 (17%) (SEE P33 FOR PHASE DESCRIPTION)	FLH-R30230B-12iS	FLH-R30230B-12iM	FLH-R30230B-40iS	FLH-R30230B-40iM	FLH-R30230B-40iL
SiliaSep™ BT Amine (SEE P.35 FOR PHASE DESCRIPTION)	FLH-R52030B-12iS	FLH-R52030B-12iM	FLH-R52030B-40iS	FLH-R52030B-40iM	FLH-R52030B-40iL
SiliaSep™ BT Cyano (SEE P.38 FOR PHASE DESCRIPTION)	FLH-R38030B-12iS	FLH-R38030B-12iM	FLH-R38030B-40iS	FLH-R38030B-40iM	FLH-R38030B-40iL
SiliaSep™ BT Diol (SEE P.42 FOR PHASE DESCRIPTION)	FLH-R35030B-12iS	FLH-R35030B-12iM	FLH-R35030B-40iS	FLH-R35030B-40iM	FLH-R35030B-40iL
SiliaSep™ BT Fluorochrom (SEE P.43 FOR PHASE DESCRIPTION)	FLH-R63730B-12iS	FLH-R63730B-12iM	FLH-R63730B-40iS	FLH-R63730B-40iM	FLH-R63730B-40iL
SiliaSep™ BT SCX-2 (SEE P.49 FOR PHASE DESCRIPTION)	FLH-R51230B-12iS	FLH-R51230B-12iM	FLH-R51230B-40iS	FLH-R51230B-40iM	FLH-R51230B-40iL

SiliaSep™ BT Cartridges (continued)

	65	75S	75M	75L
Dimensions ID × L (mm)	65 Flash Cartridge 65 × 200	75S Flash Cartridge 75 × 90	75M Flash Cartridge 75 × 150	75L Flash Cartridge 75 × 200
SiliaSep™ BT	FLH-R10030B-65i	FLH-R10030B-75iS	FLH-R10030B-75iM	FLH-R10030B-75iL
	6 / box	2 / box*	2 / box*	2 / box*

Phase	65	75S	75M	75L
SiliaSep™ BT Cartridges	1 / box	1 / box	1 / box	1 / box
SiliaSep™ BT C18 nec (23%) (see p.33 for phase description)	FLH-R30130B-65i	FLH-R30130B-75iS	FLH-R30130B-75iM	FLH-R30130B-75iL
SiliaSep™ BT C18 (17%) (see p.33 for phase description)	FLH-R30230B-65i	FLH-R30230B-75iS	FLH-R30230B-75iM	FLH-R30230B-75iL
SiliaSep™ BT Amine (see p.35 for phase description)	FLH-R52030B-65i	FLH-R52030B-75iS	FLH-R52030B-75iM	FLH-R52030B-75iL
SiliaSep™ BT Cyano (see p.38 for phase description)	FLH-R38030B-65i	FLH-R38030B-75iS	FLH-R38030B-75iM	FLH-R38030B-75iL
SiliaSep™ BT Diol (see p.42 for phase description)	FLH-R35030B-65i	FLH-R35030B-75iS	FLH-R35030B-75iM	FLH-R35030B-75iL
SiliaSep™ BT Fluorochrom (see p.43 for phase description)	FLH-R63730B-65i	FLH-R63730B-75iS	FLH-R63730B-75iM	FLH-R63730B-75iL
SiliaSep™ BT SCX-2 (see p.49 for phase description)	FLH-R51230B-65i	FLH-R51230B-75iS	FLH-R51230B-75iM	FLH-R51230B-75iL

** Box of 10 also available.*

SiliaSep™ OT Cartridges

	2g	5g	10g	15g	20g
Dimensions ID × L (mm)	Open-Top 2g Flash Cartridge 15.8 × 90	Open-Top 5g Flash Cartridge 20.5 × 100	Open-Top 10g Flash Cartridge 26.8 × 154	Open-Top 15g Flash Cartridge 26.8 × 154	Open-Top 20g Flash Cartridge 26.8 × 154
Volume	12mL	25mL	70mL	70mL	70mL
SiliaSep™ OT	FLH-R10030B-15U	FLH-R10030B-25X	FLH-R10030B-70Y	FLH-R10030B-70i	FLH-R10030B-70Z
	20 / box	20 / box	16 / box	16 / box	16 / box

Phase	2g	5g	10g	15g	20g
SiliaSep™ OT Cartridges	20 / box	20 / box	16 / box	16 / box	16 / box
SiliaSep™ OT C18 nec (23%) (SEE P.33 FOR PHASE DESCRIPTION)	SPE-R30130B-12U	SPE-R30130B-20X	FLH-R30130B-70Y	FLH-R30130B-70i	FLH-R30130B-70Z
SiliaSep™ OT C18 (17%) (SEE P.33 FOR PHASE DESCRIPTION)	SPE-R30230B-12U	SPE-R30230B-20X	FLH-R30230B-70Y	FLH-R30230B-70i	FLH-R30230B-70Z
SiliaSep™ OT Amine (SEE P.35 FOR PHASE DESCRIPTION)	SPE-R52030B-12U	SPE-R52030B-20X	FLH-R52030B-70Y	FLH-R52030B-70i	FLH-R52030B-70Z
SiliaSep™ OT Cyano (SEE P.38 FOR PHASE DESCRIPTION)	SPE-R38030B-12U	SPE-R38030B-20X	FLH-R38030B-70Y	FLH-R38030B-70i	FLH-R38030B-70Z
SiliaSep™ OT Diol (SEE P.42 FOR PHASE DESCRIPTION)	SPE-R35030B-12U	SPE-R35030B-20X	FLH-R35030B-70Y	FLH-R35030B-70i	FLH-R35030B-70Z
SiliaSep™ OT Fluorochrom (SEE P.43 FOR PHASE DESCRIPTION)	SPE-R63730B-12U	SPE-R63730B-20X	FLH-R63730B-70Y	FLH-R63730B-70i	FLH-R63730B-70Z
SiliaSep™ OT SCX-2 (SEE P.49 FOR PHASE DESCRIPTION)	SPE-R51230B-12U	SPE-R51230B-20X	FLH-R51230B-70Y	FLH-R51230B-70i	FLH-R51230B-70Z

SiliaSep™ OT Cartridges (continued)

	25 g	50 g	70 g	100 g	200 g
	Open-Top 25g Flash Cartridge	Open-Top 50g Flash Cartridge	Open-Top 70g Flash Cartridge	Open-Top 100g Flash Cartridge	Open-Top 200g Flash Cartridge
Dimensions ID × L (mm)	38.2 × 170	38.2 × 170	38.2 × 170	40.0 × 220	40.0 × 300
Volume	150mL	150mL	150mL	276mL	377mL
SiliaSep™ OT	FLH-R10030B-95K	FLH-R10030B-95M	FLH-R10030B-95N	FLH-R10030B-276F	FLH-R10030B-377D
	10 / box	10 / box	10 / box	12 / box	12 / box

Phase	25 g	50 g	70 g	100 g	200 g
SiliaSep™ OT Cartridges	10 / box	10 / box	10 / box	12 / box	12 / box
SiliaSep™ OT C18 nec (23%) (see p.33 for phase description)	FLH-R30130B-95K	FLH-R30130B-95M	FLH-R30130B-95N	FLH-R30130B-276F	FLH-R30130B-377D
SiliaSep™ OT C18 (17%) (see p.33 for phase description)	FLH-R30230B-95K	FLH-R30230B-95M	FLH-R30230B-95N	FLH-R30230B-276F	FLH-R30230B-377D
SiliaSep™ OT Amine (see p.35 for phase description)	FLH-R52030B-95K	FLH-R52030B-95M	FLH-R52030B-95N	FLH-R52030B-276F	FLH-R52030B-377D
SiliaSep™ OT Cyano (see p.38 for phase description)	FLH-R38030B-95K	FLH-R38030B-95M	FLH-R38030B-95N	FLH-R38030B-276F	FLH-R38030B-377D
SiliaSep™ OT Diol (see p.42 for phase description)	FLH-R35030B-95K	FLH-R35030B-95M	FLH-R35030B-95N	FLH-R35030B-276F	FLH-R35030B-377D
SiliaSep™ OT Fluorochrom (see p.43 for phase description)	FLH-R63730B-95K	FLH-R63730B-95M	FLH-R63730B-95N	FLH-R63730B-276F	FLH-R63730B-377D
SiliaSep™ OT SCX-2 (see p.49 for phase description)	FLH-R51230B-95K	FLH-R51230B-95M	FLH-R51230B-95N	FLH-R51230B-276F	FLH-R51230B-377D

SiliaSep™ BU Cartridges

Dimensions ID × L (mm)	12S Flash Cartridge 12 x 75	12M Flash Cartridge 12 x 150	40S Flash Cartridge 40 x 70	40M Flash Cartridge 40 x 150
SiliaSep™ OT	FLB-R10030B-12S	FLB-R10030B-12M	FLB-R10030B-40S	FLB-R10030B-40M
	20 / box	20 / box	10 / box	10 / box
Phase	**12S**	**12M**	**40S**	**40M**
SiliaSep™ BU Cartridges	2 / box	2 / box	1 / box	1 / box
SiliaSep™ BU C18 nec (23%) (see p.33 for phase description)	FLB-R30130B-12S	SPE-R30130B-12M	FLH-R30130B-40S	FLH-R30130B-40M
SiliaSep™ BU C18 (17%) (see p.33 for phase description)	FLB-R30230B-12S	SPE-R30230B-12M	FLH-R30230B-40S	FLH-R30230B-40M
SiliaSep™ BU Amine (see p.35 for phase description)	FLB-R52030B-12S	SPE-R52030B-12M	FLH-R52030B-40S	FLH-R52030B-40M
SiliaSep™ BU Cyano (see p.38 for phase description)	FLB-R38030B-12S	SPE-R38030B-12M	FLH-R38030B-40S	FLH-R38030B-40M
SiliaSep™ BU Diol (see p.42 for phase description)	FLB-R35030B-12S	SPE-R35030B-12M	FLH-R35030B-40S	FLH-R35030B-40M
SiliaSep™ BU Fluorochrom (see p.43 for phase description)	FLB-R63730B-12S	SPE-R63730B-12M	FLH-R63730B-40S	FLH-R63730B-40M
SiliaSep™ BU SCX-2 (see p.49 for phase description)	FLB-R51230B-12S	SPE-R51230B-12M	FLH-R51230B-40S	FLH-R51230B-40M

See Büchi™ adapters for Isco™, Biotage™, and FlashMaster™ in section SiliaCartridger™ (page 7)

SiliaSep™ accessories

SiliaSep™ ADAPTERS

Easily attach luer fitting adapters to your Biotage™ or FlashMaster™ instrument solvent lines to use SiliaSep™ (Isco™ compatible cartridges)

KAD-1006	Isco™ – Biotage™ Adapter Kit (2 pieces)
KAD-1016	Isco™ – FlashMaster™ Adapter Kit (2 pieces)

KAD-1006

SiliaSep™ SUPPORT RINGS

Support rings allow you to support SiliaSep™ (Isco™ compatible cartridges) on your Biotage™ system (Flash+ serie).

AUT-0068-004	Support ring-4 (16 mm)	(5/box)
AUT-0068-012	Support ring-12 (24 mm)	(5/box)
AUT-0068-040	Support ring-40 (32 mm)	(5/box)
AUT-0068-080	Support ring-80 (36 mm)	(5/box)
AUT-0068-120	Support ring-120 (42 mm)	(5/box)
KAD-1008	Support ring kit (5 of different sizes/box)	

KAD-1008

Plungers

KAD-1014

SiliaSep™ PLUNGERS

AUT-0060-010	Plunger for 10 mL SPE (16 mm)
AUT-0060-060	Plunger for 60 mL SPE (27 mm)

SiliaSep™ LINE

Simply replace the solvent lines on your Biotage™ Horizon, SP1 or SP4 with our luer fitting solvent lines.

KAD-1010	Luer extention line for Isco™ instruments (1 line/box)
KAD-1012	Luer fitting line replacement kit for Biotage™ Horizon instruments (2 lines/box)
KAD-1014	Luer fitting line replacement kit for Biotage™ SP1 and SP4 instruments (2 lines/box)

SiliaSep™ SOLID-LOAD CARTRIDGES

SPL-R10030B-10X	Pre-packed Luer-Lock Cartridges, 5 g, 10 mL (20/box)
SPL-R10030B-60K	Pre-packed Luer-Lock Cartridges, 25 g, 60 mL (16/box)
SPL-R10030B-065	Pre-packed Cartridges 65 g, 150 mL (12/box)
SPL-R10030B-270	Pre-packed Cartridges 270 g, 700 mL (6/box)
SPL-0009-010	Empty Luer-Lock Cartridges, 10 mL (100/box)
SPL-0012-060	Empty Luer-Lock Cartridges, 60 mL (100/box)
AUT-0090-150	Empty Cartridges, 150 mL (12/box)
AUT-0090-700	Empty Cartridges 700 mL (6/box)

SILIABOND®

2008 catalog

SiliaBond® product line introduction

SiliCycle® is the leader in the development of functionalized silica gels, gathered under the name **SiliaBond®**, for use in organic synthesis. The backbone of all **SiliaBond®** products is SiliCycle®'s **SiliaFlash®** which provides superior performance for all types of applications due to the narrow particle size distribution and high purity. All the products in this section are made of standard flash silica gel, namely 40-63 µm, 60 Å. **We are able to provide all of our functionalized products on any silica of this catalog (IMPAQ®, spherical, large beads, etc) in any format (SPE and flash cartridges, 96 well plates, etc) on a custom basis.**

The use of solid supported reagents and scavengers is an excellent way to expedite organic synthesis by simplifying the purification process. Standard purification procedures including chromatography, liquid-liquid extraction, and crystallization can be time-consuming and difficult to scale up. In many cases, supported reagents have distinct advantages over their solution-phase counterparts, the biggest one being the ability to do multiple transformations in a single pot, the immobilization of toxic reagents, and increased selectivity. They are suitable for use in batch reactions and in flow through applications.

ADVANTAGES OF FUNCTIONALIZED SILICA OVER POLYMER

- **Fast Kinetics:** Since the silica is surface functionalized, the rate of reaction is not controlled by the diffusion in and out of the polymer. Most of our scavengers work under an hour.
- **Solvent Independent:** Silica neither shrinks nor swells in any solvent and because it is endcapped, it will not dissolve in any solvent. The **SiliaBond®** does not dictate the solvent of your reaction.
- **Easy to Use:** Easy to weigh and handle since silica does not carry a static charge and is always free flowing. Its high density makes it suitable for small volume work. It does not require extensive washing for high recoveries and will not stick to glassware. It is amenable to automation.
- **Mechanically Stable:** Works well with overhead stirring and can be used with a bottom coupled spin bar for up to four hours.
- **Thermally Stable:** Most **SiliaBond®** can withstand temperatures of over 200°C and are suitable for use in microwave synthesizers.
- **Easily Scalable:** Works on the microgram to the multi-ton scale.
- **Flexible Formats:** Because it does not swell, silica gel can be packed into a variety of flow through formats such as HPLC columns, flash and SPE cartridges, and 96 well plates.
- **Controlled Loading:** Consistent and accurate loading (lot-to-lot).

Tel.: 1 418.874.0054 Fax: 1 418.874.0355 Toll-free: 1 877.SILICYCLE (North America only) info@silicycle.com
www.SiliCycle.com

The application domains of the **Silia*Bond*®** are mainly divided in two:

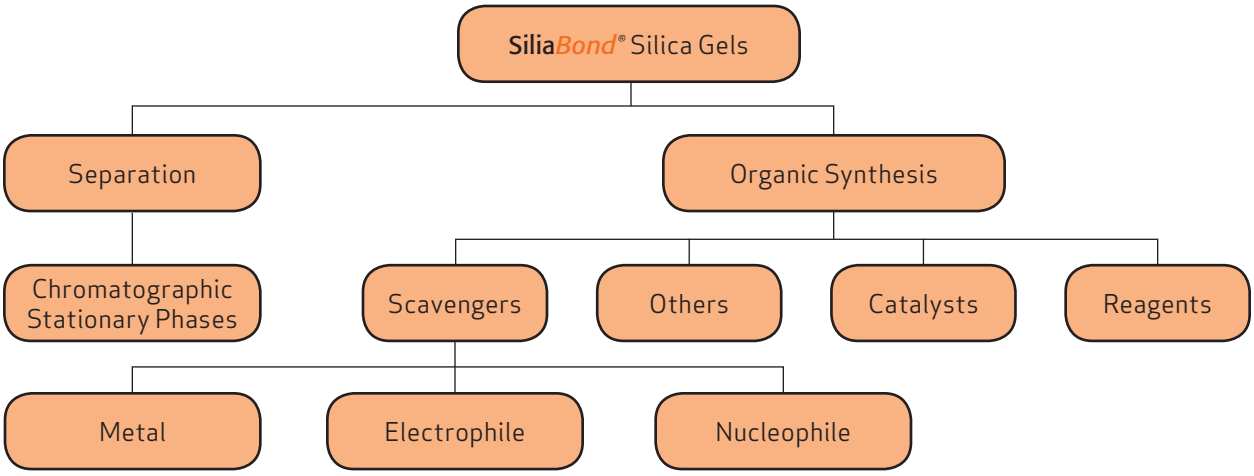
1. They are either for the separation of mixtures in their individual components (chromatography) ***or***

2. As tools in organic synthesis.

This last category breaks down into four subcategories:

- Scavengers
- Reagents
- Catalysts
- Functions having other uses.

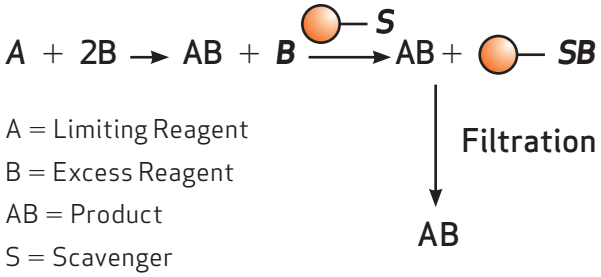
Classification of functionalized silica gels



SCAVENGERS

SiliCycle® offers a wide range of scavengers to simplify your purifications. Scavengers are functionalized silica gels designed to react and bind excess reagents,metal complexes and/or by-products. The process for using scavengers is outlined in the scheme shown on next page. In the case of batch processing, the scavenger is added to the reaction mixture after completion. The process relies on chemically driven reactions where the excess reagents and reaction by-products react with and bind to the scavenger. The solution, which now only contains the product of interest, is separated from the silica bound impurities by filtration. For flow through processing, the scavenger is packed into a column and the reaction mixture is pass through the column until complete scavenging is obtained.

What is a Scavenger?



METAL SCAVENGERS

Removing residual transition metals from post reactions is a major issue for many chemists, particularly process chemists in the pharmaceutical industry where the toxic nature of transition metals means their concentration has to be reduced to the single digit ppm concentration level for the material to be used *in vivo* and as active pharmaceutical ingredient (API). The low metal concentration can also be very important in other cases, such as in polymeric materials where very low metal concentration has to be reached in order to improve the optoelectronic.[1] SiliCycle® has a wide range of scavengers to remove a variety of transition metals.

When selecting a metal scavenger for your reaction, every component in the reaction must be considered. The metal complex and the solvent used are important but residual reagents, presence of by-products, and temperature also have their importance.

Metal Scavenger selection table

Scavenger	Pd (II)	Pd (0)	Sn	Zn	Pb	Ni (II)	Ni(0)	Pt	Cu	Co	Rh (III)	Ru	Cd	Ag	Hg	Fe	Cr (III)	V
Si-DIA				■	■	■	■		■	■			■		■	■	■	
Si-NH_2				■	■				■	■	■	■	■		■		■	
Si-PYR	■															■	■	
Si-TAAcOH			■				■									■		
Si-TAAcONa	■			■		■			■	■	■	■				■	■	
Si-TBD	■		■				■			■	■	■				■	■	
Si-Thiol	■	■	■		■			■	■		■	■		■	■			
Si-THU	■	■						■				■		■	■			■
Si-TRI	■				■		■		■	■		■				■	■	■

■ *Preferred scavenger* ■ *Scavenges* ■ *Reported in the literature or by customers*

Visit our website to see metal scavenger video.

[1] US 6,894,145 B2

The following tables show scavenging results for Pd complexes and other metals.

Scavenging Palladium complexes

Scavenger	Product page	$Pd(AcO)_2$	$Pd_2(C_3H_5)_2Cl_2$	$Pd(PPh_3)_4$	$Pd_2(dba)_3$	$Ni(AcO)_2$
Si-Thiol	53	0.07	0.04	150	20	–
Si-THU	54	0.8	1.3	95	50	–
Si-TAAcOH	51	0.06	0.25	1.4	50	60
Si-TAAcONa	51	42	–	144	–	10
Si-TRI	58	0.3	1.3	220	280	–

Conditions: 4 eq. of scavenger in THF at RT for 1 hour or 18 hours. Starting concentration of 1000 ppm.

Scavenging metal complexes

Scavenger	$FeCl_3$	$CrCl_3$	$Cu(OAc)_2$	$Pd(OAc)_2$	$Pd_2(Allyl)_2Cl_2$	$Pd(PPh_3)_4$	$RhCl_3$	$RuCl_3$	$Ni(acac)_2$	$ZnCl_2$[b]	$Co(NO_3)_2$
Si-DIA	99.9	99.4	98.7	99.0	99.7[a]	–	–	–	97.3[a]	94.1[a]	99.9
Si-NH_2	99.9[a]	99.9	99.7	99.7	99.7[a]	–	100.0[a]	100.0[a]	93.4[a]	96.9[a]	99.9
Si-PYR	99.5[a]	96.7[a]	–	99.7[a]	96.1[a]	–	–	–	–	–	–
Si-TAAcOH	99.9	99.3	98.2	99.9[a]	–	–	–	–	99.8	–	97.7[a]
Si-TAAcONa	100.0	99.9	99.8	100.0[a]	95.5	95.7[a]	100.0[a]	100.0[a]	99.4	98.1	100.0
Si-TBD	100.0	99.9	96.3	99.9[a]	97.0	–	100.0	98.0[a]	–	–	100.0
Si-Thiol	–	–	99.6	99.9	99.7	98.5	–	–	89.9[a]	–	–
Si-THU	–	–	98.6[a]	100.0	99.7[a]	97.8[a]	–	–	–	–	–
Si-TRI	97.0	100.0[a]	99.8	100.0	99.1	–	–	–	99.1	–	99.8

Conditions: 4 eq. THF, rt, 30 min. [a]*After 4 hours.* [b]*Done in water.*

Visit our website to see metal scavenger video.

MICROWAVE ASSISTED SCAVENGING

You can obtain pure compounds in five minutes by using our scavengers in microwave synthesizers. Below are the results using **Silia*Bond*®** Amine to scavenge a variety of nucleophiles. The scavenging was carried out using 4 eq. under microwave conditions: 4 min., 150°C, 300 W. No leaching of the reactive groups was observed by GC-MS. The integrity of the **Silia*Bond*®** under microwave conditions (700 W, 30 min.) is not affected.

Scavenging under microwave irradiation

Nucleophile	% Scavenged
Benzaldehyde	97
Benzoyl Chloride	100
Acetic Anhydride	99
1,1,3,3-Tetramethylbutylisocyanate	100
Tert-butylphenylisocyanate	100

Reactions were run on a Discover™ Synthesizer (CEM Corporation).

INCREASING SCAVENGER EFFICIENCY

1. **Temperature:** Increasing the reaction temperature will increase the scavenger efficiency. Scavengers have a high thermal stability and can be used in microwave synthesizers.

2. **Number of Equivalents:** The more equivalent of scavenger are added, the more effective is the scavenging.

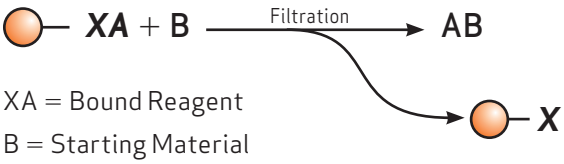
3. **Reaction Time:** In some cases, it maybe more cost effective to leave the reaction overnight and add only a slight excess of scavenger.

4. **Solvent:** Since silica does not swell, the reactions are solvent-independent. However, the kinetics will be influenced by the solvent to some extent in the same way as it would in homogeneous conditions. It is to say that the support does not restrict the choice of solvent, but the type of chemistry does. In the case of metals, coordinating solvents such as DMF can reduce scavenger efficiency. This effect can be overcome by adding more scavenger, or increasing reaction time and temperature.

SUPPORTED REAGENTS

A supported reagent, like a scavenger, is a reactive functional group grafted onto insoluble silica. Unlike a scavenger, which is added once a reaction is complete a supported reagent is added at the beginning of the reaction. It replaces the solution phase reagent and allows the supported reagent to be used in greater excess, driving the reaction to completion. It also facilitates one-pot multiple-step reactions. The spent reagent is easily removed by filtration. Bound reagents are an excellent alternative in cases where the reagent is used in excess and can be difficult to remove. Purification is now a simple filtration and evaporation process. The supported reagents are bound to the silica by an ionic or covalent bond, or simply adsorbed on the surface. This is the case for **Silia***Bond*® PCC, **Silia***Bond*® PDC, and **Silia***Bond*® Potassium Permanganate. The following scheme depicts a typical reaction involving a supported reagent.

Silica bound reagents



XA = Bound Reagent
B = Starting Material
AB = Product
X = Spent Reagent

NEW

SiliCycle® can test your solutions and find the optimal conditions to remove any leftover metal catalyst. Contact us for details.

SCAVENGER SELECTION CHART

For Electrophiles

Function to be scavenged	Recommended SiliaBond® scavengers
Acid Chlorides/Sulfonyl chlorides	SiliaBond® *Amine, Diamine, Triamine* SiliaBond® *DMAP* SiliaBond® *Piperazine*
Carbonyls	SiliaBond® *Amine* SiliaBond® *Tosyl Hydrazine*
Isocyanates	SiliaBond® *Amine, Diamine, Triamine*
Anhydrides	SiliaBond® *Amine*

For Nucleophiles

Function to be scavenged	Recommended SiliaBond® scavengers
Acids/Acidic Phenols	SiliaBond® *Amine, Diamine, Triamine* SiliaBond® *Carbonate* SiliaBond® *TBD*
Alcohols	SiliaBond® *Tosyl Chloride*
Alkoxides	SiliaBond® *Isocyanate* SiliaBond® *Tosyl Chloride*
Amines	SiliaBond® *Isocyanate* SiliaBond® *Propylsulfonic Acid (SCX-2)* SiliaBond® *Succinic Anhydride* *(selective for RNH_2, R^1R^2NH over alcohol)* SiliaBond® *Tosic Acid (SCX)* SiliaBond® *Tosyl Chloride*
Hydrazines	SiliaBond® *Tosyl Chloride*
Organometallics	SiliaBond® *Tosyl Chloride*
Thiol/Thiolates	SiliaBond® *Isocyanate* SiliaBond® *Maleimide (for Thiol)*

Visit our website for new SiliaBond® scavengers.

COMMON ORGANIC REACTIONS

SiliaBond® reagents and scavengers for typical organic reactions.

Reaction	Scavenger / Reagent
Amide coupling	
with acid chlorides and amines	***Si-Carbodiimide*** (reagent)
with acids and amines	***Si-Amine*** (scavenger) - removes excess acid chloride ***Si-DCT*** (reagent) ***Si-Isocyanate / Si-Succinic Anhydride / Si-TsOH*** (scavengers) - remove excess amine
using HOBt or Pentafluorophenol	***Si-Carbonate*** (scavenger) - removes excess HOBt and Pentafluorophenol
Friedel-Crafts alkylation	***Si-Aluminum Chloride*** (reagent)
Heck coupling	***Si-Thiol / Si-Thiourea / Si-TAAcOH*** (scavengers) - remove palladium
Knoevenagel condensation	***Si-Amine/ Si-Dimethylamine/Si-Piperidine*** (catalysts)
Oxidation	
alcohols to acids	***Si-KMnO4*** (reagent)
alcohols to ketones or aldehydes	***Si-TEMPO / Si-PDC / Si-PCC*** (reagents)
Reduction	
with borohydride reducing agents	***Si-TsOH*** (scavenger) - removes excess and spent borohydride
of alkyl halides	***Si-SnH*** (reagent)
Reductive amination	***Si-Cyanoborohydride*** (reagent) ***Si-TsOH*** (scavenger) - removes excess amine
Sulfonamide synthesis	***Si-Amine*** (scavenger) - removes excess sulfonyl chloride
Suzuki coupling	***Si-Carbonate*** (scavenger) - removes excess HOBt ***Si-DPP*** (reagent) - must be complexed with Pd ***Si-Thiol / Si-Thiourea / Si-TAAcOH*** (scavengers) - remove palladium
Tosylate formation	***Si-TsCl*** (reagent)
Urea synthesis	***Si-Amine*** (scavenger) - removes excess isocyanate
Williamson ether synthesis	***Si-TBD*** (reagent)

Visit our website for new SiliaBond® and SiliaCat®.

SiliaBond® Kits

For your convenience, we have combined our most popular scavengers and reagents for the most common applications into kits so you can be prepared with material in hand for any application.

SiliaBond® Kits are available in 5 g, 10 g, 25 g or 100 g format.

SiliaBond® Acid Kit (K31330B)

Products included in the kit	
R70030B	SiliaBond® Carboxylic Acid
R51230B	SiliaBond® Propylsulfonic Acid
R69030B	SiliaBond® TAAcOH
R60530B	SiliaBond® Tosic Acid

SiliaBond® Base Kit (K31630B)

Products included in the kit	
R52030B	SiliaBond® Amine
R66030B	SiliaBond® Carbonate
R45030B	SiliaBond® Dimethylamine
R76530B	SiliaBond® Diethylamine
R68030B	SiliaBond® Morpholine
R43030B	SiliaBond® Pyridine
R68530B	SiliaBond® TBD

SiliaBond® Electrophile Scavenger Kit (K34230 B)

Products included in the kit	
R52030B	SiliaBond® Amine
R49030B	SiliaBond® Diamine
R75530B	SiliaBond® DMAP
R60030B	SiliaBond® Piperazine
R61030B	SiliaBond® Tosyl Hydrazine
R48030B	SiliaBond® Triamine

SiliaBond® Ion Exchanger Kit (K31430B)

Products included in the kit	
R52030B	SiliaBond® Amine (WAX)
R70030B	SiliaBond® Carboxylic Acid (WCX)
R51230B	SiliaBond® Propylsulfonic Acid (SCX-2)
R66530B	SiliaBond® TMA Chloride (SAX)
R60530B	SiliaBond® Tosic Acid (SCX)

SiliaBond® Metal Scavenger Kit (K30730B)

Products included in the kit	
R49030B	SiliaBond® Diamine
R69030B	SiliaBond® TAAcOH
R69230B	SiliaBond® TAAcONa
R51030B	SiliaBond® Thiol
R69530B	SiliaBond® Thiourea
R48030B	SiliaBond® Triamine

SiliaBond® Nucleophile Scavenger Kit (K30630B)

Products included in the kit	
R50030B	SiliaBond® Isocyanate
R64530B	SiliaBond® Succinic Anhydride
R60530B	SiliaBond® Tosic Acid
R44030B	SiliaBond® Tosyl Chloride

SiliaBond® Oxidant Kit (K30330B)

Products included in the kit	
R23030B	SiliaBond® Potassium Permanganate
R24030B	SiliaBond® Pyridinium Chlorochromate
R24530B	SiliaBond® Pyridinium Dichromate
R723-100	SiliaCat® TEMPO

SiliaBond® Reagent Kit (K32230B)

Products included in the kit	
R70530B	SiliaBond® Carbodiimide
R66730B	SiliaBond® Cyanoborohydride
R52230B	SiliaBond® Dichlorotriazine
R75530B	SiliaBond® DMAP

SiliaBond® Reversed-Phase Kit (K32530B)

Products included in the kit	
R32030B	SiliaBond® C4
R31030B	SiliaBond® C8
R53030B	SiliaBond® C12
R30230B	SiliaBond® C18 (17%)
R30030B	SiliaBond® C18 nec (23%)
R38030B	SiliaBond® Cyano
R34030B	SiliaBond® Phenyl

SiliaBond® µ-wave Reagent Kit (K31230B)

Products included in the kit	
R70530B	SiliaBond® Carbodiimide
R66730B	SiliaBond® Cyanoborohydride
R39030B	SiliaBond® Diphenylphosphine

SiliaBond® µ-wave Scavenger Kit (K31530B)

Products included in the kit	
R52030B	SiliaBond® Amine
R51230B	SiliaBond® Propylsulfonic Acid
R69030B	SiliaBond® TAAcOH
R68530B	SiliaBond® TBD
R51030B	SiliaBond® Thiol
R69530B	SiliaBond® Thiourea

SiliaBond® µ-wave Ultimate Kit (KK030B)

Products included in the kit	
K31230B	SiliaBond® µ-wave Reagent Kit
K31530B	SiliaBond® µ-wave Scavenger Kit

SiliaBond® Kits are also available in SiliaPrep™ SPE format of 2 g / 6 mL

SiliaPrep™ Kits

Catalog #	Product Name
SPE-K31330B-06U	SiliaPrep™ Acid Kit
SPE-K31630B-06U	SiliaPrep™ Base Kit
SPE-K34230B-06U	SiliaPrep™ Electrophile Scavenger Kit
SPE-K31430B-06U	SiliaPrep™ Ion Exchanger Kit
SPE-K30730B-06U	SiliaPrep™ Metal Scavenger Kit
SPE-K30630B-06U	SiliaPrep™ Nucleophile Scavenger Kit
SPE-K30330B-06U	SiliaPrep™ Oxidant Kit
SPE-K32230B-06U	SiliaPrep™ Reagent Kit
SPE-K32530B-06U	SiliaPrep™ Reversed-Phase Kit

SiliaBond® C1 (R33030B)

Loading: 5% carbon
Category: Chromatographic Stationary Phase

DESCRIPTION

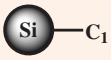
SiliaBond® C1 (or **C1**) has the lowest degree of hydrophobicity of all reversed stationary phases, useful in the separation of large biomolecules that have extensive hydrophobic regions.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® C4 (R32030B), NON-ENDCAPPED (R32130B)

Loading: 8% carbon
Category: Chromatographic Stationary Phase

DESCRIPTION

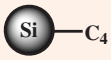
SiliaBond® C4 (or **C4**) provides less retention towards non-polar compounds than **C18** and **C8,** and is useful in ion-pairing chromatography. Used to separate large biomolecules. It works best for molecules with a large hydrophilic region or in case where the hydrophobic region is buried within the 3D structure.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® C8 (R31030B), NON-ENDCAPPED (R31130B)

Loading: 12% carbon
Category: Chromatographic Stationary Phase

DESCRIPTION

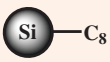
SiliaBond® C8 (or **C8**) is a reversed-phase matrix with a moderate degree of hydrophobicity that works well for separating a wide range of compounds. May be used instead of **C18** when shorter retention times are desired.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® C12 (R53030B)

Loading: 16% carbon
Category: Chromatographic Stationary Phase

DESCRIPTION

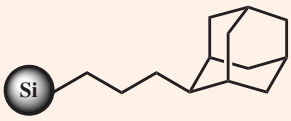
SiliaBond® C12 (or **C12**, ***Si*-Adamantyl**) has a polarity similar to **C18** with additional steric bulk to provide additional separation characteristics.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry.



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® C18

Loading: 23% carbon (R30030B), non-endcapped (R30130B) – 17% carbon (R30230B) – 11% carbon (R30430B)
Category: Chromatographic Stationary Phase

DESCRIPTION

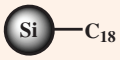
SiliaBond® C18 (or **C18**) is the traditional matrix for reversed-phase chromatography. The high loading provides the highest degree of hydrophobicity commercially available. Considered as the least selective phase since it interacts with a wide range of organic molecules. Also used in ion-pairing chromatography (non-endcapped) for the separation of biomolecules.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Allyl (R53530B)

Loading: 1.2 mmol/g
Category: Other

DESCRIPTION

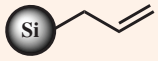
SiliaBond® Allyl (or ***Si*-Allyl**) can be used as solid support for further derivatization.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Aluminum Chloride NON-ENDCAPPED (R74530B)

Loading: 1.6 mmol/g
Category: Reagent, Catalyst

DESCRIPTION

SiliaBond® Aluminum Chloride (or ***Si*-$AlCl_x$**) is the silica supported version of the most widely used Lewis acid, aluminum chloride.[1] It is an effective catalyst for Friedel-Crafts alkylations[2-4] and acylations. It also catalyzes the formation of ethers. The silica supported product has several advantages over the free catalyst[5,6]:

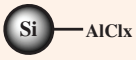
- Milder Lewis acid. $AlCl_3$ is so reactive that it often lacks of selectivity generating unwanted by-products.
- The steric bulk of the silica reduces over alkylation and increases shelf life.
- Easy work up. The reagent is removed by a simple filtration, avoiding the destructive water quench, which produces large amount of hazardous waste.

SiliaBond® Aluminum Chloride's activity can be determined by its color. The material should only be used when it's yellow or violet. The product turns white in presence of moisture.

APPLICATION NOTES

Friedel-Crafts Alkylation

Formation of Linear Alkyl Benzene (LAB) (p.70)
Ether formation (p.71)



10g
25g,100g
500g
1kg
5kg
10kg
25kg*

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (<8°C) and dry; store under argon .

RELATED PUBLICATIONS

1) Acc. Chem. Res., 35 (2002) 791
2) Org. Process Res. Dev., 2 (1998) 221
3) J. Catal., 195 (2000) 237
4) J. Catal., 195 (2000) 412
5) Chem. Rev., 103 (2003) 4307
6) Tetrahedron, 59 (2003) 1781

SiliaBond® Amine (R52030B), NON-ENDCAPPED (R52130B)

Loading: 1.6 mmol/g
Category: Base, Metal Scavenger, Chromatographic Stationary Phase, and Ion Exchanger

DESCRIPTION

SiliaBond® Amine (or ***Si*-NH$_2$**) is an effective scavenger of acid chlorides, sulfonyl chlorides, isocyanates and other electrophiles. Researchers at Hoffman La–Roche compared PS-trisamine to **SiliaBond® Amine** for the removal of acid chlorides in the synthesis of 2,9-disubstituted guanines. Using **SiliaBond® Amine** to remove excess acid chloride gave higher yields and better purity in 15 of the 16 reactions compared to PS-trisamine. For complete details please view the scavenger case study[1].

SiliaBond® Amine eliminates tedious post reaction purification. It scavenges faster, is easier to add and filter, and can be used in any solvent. ***Si*-NH$_2$** has been shown to be effective metal scavenger[2] and catalyst for Knoevenagel reactions.[3,4] It has also been used as support in solid-phase chemistry for chemical peptide synthesis followed by enzymatic hydrolysis[5] and for Claisen rearrangement.[6]

SiliaBond® Amine is also used in chromatography as a normal phase sorbent. It is used in the separation of peptides, drugs and metabolites from physiological fluids, and for the separation of mono- and polysaccharides. ***Si*-NH$_2$** is more retentive towards basic compounds. When used at pH ≤ 7.8, it is protonated and may be used as a weak anion exchanger (WAX). **SiliaBond® Amine** has been used for the solid-phase extraction of sugars[7], for the separation of steroids[8], cholesterol, and triglycerides,[9] and in size exclusion chromatography of cationic polymers.[10] ***Si*-NH$_2$** is also available non-endcapped (R52130B) for additional retention of polar and cationic compounds.

APPLICATION NOTES

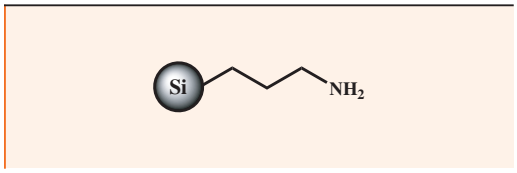
Scavenging Acid Chloride (p.74)

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (< 8°C) and dry.



25g
100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) Tetrahedron Lett., 41 (2000) 3573
2) Chem. Commun., (2000) 1145
3) Tetrahedron Lett., 29 (1988) 2261
4) J. Chem. Soc. Perkin Trans. I, (1989) 105
5) Tetrahedron Lett., 44 (2003) 6083
6) Molecular Diversity, 3 (1998) 161
7) J. Liq. Chromatogr., 14 (1991) 1185
8) J. Chromatogr., 392 (1987) 464
9) J. Chromatogr., 434 (1988) 395
10) J. Chromatogr., 389 (1987) 103

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Bromophenyl (R55030B)

Loading: 1.1 mmol/g
Category: Other

DESCRIPTION

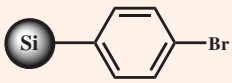
SiliaBond® Bromophenyl (or ***Si*-BRP**) can be used as solid support for further derivatization.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Carbodiimide (R70530B)

Loading: 1.0 mmol/g
Category: Reagent

DESCRIPTION

SiliaBond® Carbodiimide (or ***Si*-DCC**) is a bound neutral carbodiimide that may be used for the synthesis of amides, esters, and activated esters. Loading is determined by titration of the excess oxalic acid after formation of oxalic anhydride. The use of silica-supported DCC eliminates purification issues caused by the formation of DCU as it remains bound to the silica.

Applications and uses of **SiliaBond® Carbodiimide** are not restricted to standard amides. As a matter of fact, it has been successfully used for the synthesis of Weinreb amides and acylsulfonamides.

APPLICATION NOTES

Amide Synthesis

- In bulk – Method 1 (p.64)
- In bulk – Method 2 (p.64)
- In cartridges, using **SiliaPrep™** DCC (p.65)

Weinreb Amide Synthesis (p.84)

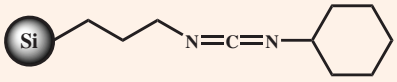
Acylsulfonamide Synthesis (p.81)

SOLVENT COMPATIBILITY

Aprotic solvents

PROLONGED STORAGE

Keep cool (< 8°C) and dry; store under argon.



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Carbonate (R66030B)

Loading: 0.7 mmol/g
Category: Base

DESCRIPTION

SiliaBond® Carbonate (or ***Si*-CO_3**) is the silica bound equivalent of tetramethyl ammonium carbonate. It can be used as a general base to quench a reaction, to free base amines in their ammonium salt form and to scavenge acids and acidic phenols, including HOBt, which is widely used in amide coupling reactions.

SiliaBond® Carbonate is also very efficient at scavenging boronic acids: 99% removal of phenylboronic acid with one equivalent in DCM at room temperature after 1h. ***Si*-CO_3** can free base ephedrine hydrochloride quantitatively (100% yield determined by ^{1}H NMR) using 4 equivalents at room temperature for two hours.

APPLICATION NOTES

Amine Free Basing[1]

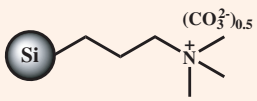
In bulk (p.67)

In cartridges (p.67)

Scavenging HOBt[2]

In bulk (p.77)

In cartridges (p.77)



25g
100g
500g
1kg
5kg
10kg
25kg*

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry

RELATED PUBLICATIONS

1) Org. Lett., 4(7) 2002, 1167
2) Org. Lett., 5(24) 2003, 4721

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Carboxylic Acid (R70030B)

Loading: 1.4 mmol/g
Category: Acid and Ion Exchanger

DESCRIPTION

SiliaBond® Carboxylic Acid (or ***Si*-CAA**) can be used as a scavenger for amines or carbonates, and for the quench of alkoxides and organometallic reagents.

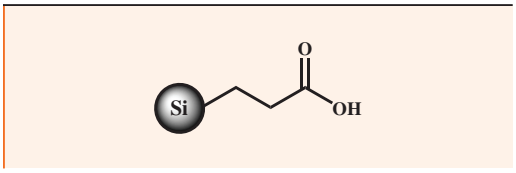
SiliaBond® Carboxylic Acid is primarily used as a weak cation exchanger (WCX) in solid phase extraction (SPE) and in HPLC. It is preferred over **SiliaBond® Tosic Acid** (SCX) when performing *"Catch and Release"* purification of compounds bearing a permanent positive charge such as tetraalkylammoniums. Using the SCX in this case could make the release of the compound of interest difficult, not to say irreversible, due to the strong interaction between the two strong ions. **SiliaBond® Carboxylic Acid** has a pka of ~ 4.5. It can also be used in ion exchange chromatography (IEC) and has a different selectivity than **SiliaBond® Tosic Acid** (SCX).[1,2]

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) J. Chromatogr., 117 (1976) 269
2) J. Chromatogr., 123 (1976) 109

SiliaBond® Cyano (R38030B), NON-ENDCAPPED (R38130B)

Loading: 7% Carbon
Category: Chromatographic Stationary Phase

DESCRIPTION

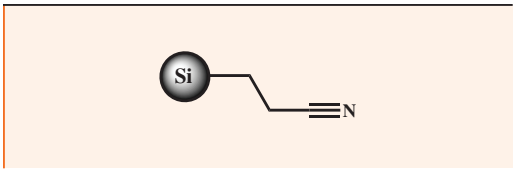
SiliaBond® Cyano (or ***Si*-CN**) can be used both in normal and reversed-phase chromatography as its polarity marks the separation between the polar and non-polar phases. **SiliaBond® Cyano** is the least retentive of the polar sorbents in normal phase chromatography and the least retentive of the non-polar sorbents in reversed-phase chromatography. This sorbent is particularly useful when dealing with extremes in the polarity scale since it reduces the retention time and help improving the peak shapes.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Cyanoborohydride (R66730B)

Loading: 1.0 mmol/g
Category: Reagent

DESCRIPTION

SiliaBond® Cyanoborohydride (or ***Si*-CBH**) is the silica bound equivalent of sodium cyanoborohydride. Bound cyanoborohydride is very useful in reductive amination and in the reduction of imines and aldehydes. Cyanide contamination of the product is a concern when using the solution phase equivalent. This problem is minimized with the use of silica bound materials since the toxic cyanide residue remains on the silica. Additionally, it may be used to reduce α,β-unsaturated carbonyl to the corresponding unsaturated alcohol.

To see if any cyanide ion was leaching from the silica, 1g of **SiliaBond® Cyanoborohydride** has been washed in 10 mL of methanol for 24 hours. Cyanide strips indicated less than 3 ppm in each test performed.

APPLICATION NOTES

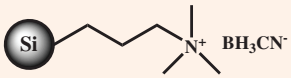
Reductive Amination (p.73)

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (< 8°C) and dry; store under argon.



25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Cyclohexyl (R61530B)

Loading: 10% Carbon
Category: Chromatographic Stationary Phase

DESCRIPTION

SiliaBond® Cyclohexyl (or ***Si*-CYH**) is a sorbent of medium polarity used in reversed-phase chromatography. It has a different selectivity than the alkyl sorbents (C18, C8, C4...) and phenyl, and may be used when these fail to provide the desired selectivity.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry

10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

All these products are available in different sizes from lab scale to multi-ton scale.

SiliaBond® Diamine (R49030B)

Loading: 1.4 mmol/g
Category: Base and Metal Scavenger

DESCRIPTION

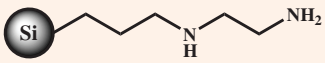
SiliaBond® Diamine (or ***Si*-DIA**) is a proven scavenger for metals and electrophiles. It scavenges acids, acid chlorides, anhydrides, aldehydes, isocyanates, and chloroformates as well as Pb, Ni, and Cd.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (<8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Dichlorotriazine (R52230B)

Loading: 0.7 mmol/g
Category: Reagent

DESCRIPTION

Amide bond formation is certainly among the most common chemical transformation in organic synthesis. This explains the diversity of possible reaction pathways and reagents available on the market. SiliCycle® is part of this trend and this is why we have developed the bound equivalent of 2,4,6-trichloro-1,3,5-triazine (cyanuric chloride), **SiliaBond® Dichlorotriazine** (or ***Si*-DCT**).

SiliaBond® Dichlorotriazine must be activated with N-methylmorpholine (NMM) to give a morpholinium salt that can react with the carboxylic acid to form the activated ester. It may then be reacted with an amine to form the amide or safely stored for use at a later date. ***Si*-DCT** may also be used for synthesis of Weinreb amides and acylsulfonamides. ***Si*-DCT** provides a fast clean route for amides formation with complete conversion in about an hour compared to most bound coupling reagents, which require overnight incubation.

Using **SiliaPrep™ Dichlorotriazine** (in cartridges) offers certain advantages over the bulk ***Si*-DCT**. They can be summarized as follow:

- Shorter reaction time (few minutes compared to 1 h);
- No need for use of supported scavengers (saves an additional step and costs);
- Amenable to automation.

APPLICATION NOTES

Acylsulfonamide Synthesis (p.81)

Weinreb Amide Synthesis

SiliaBond® Dichlorotriazine (p.85)

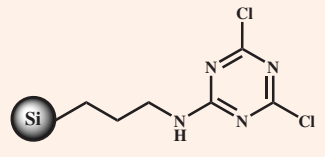
SiliaPrep™ Dichlorotriazine (p.86)

SOLVENT COMPATIBILITY

Anhydrous aprotic solvents

PROLONGED STORAGE

Keep cool (<8°C) and dry; store under argon.



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Diethylamine (R76530B)

Loading: 1.2 mmol/g
Category: Base and Ion Exchanger

DESCRIPTION

SiliaBond® Diethylamine (or **SiliaBond® WAX-2**) is a silica bound tertiary amine base and can be used in most applications requiring a tertiary amine, particularly as a HCl sponge. Silica bound ammonium salt by-products are easily separated by filtration. This diethylamine immobilized on silica gel can be used for the same applications as **SiliaBond® Dimethylamine**. It is a very common supported tertiary amine.

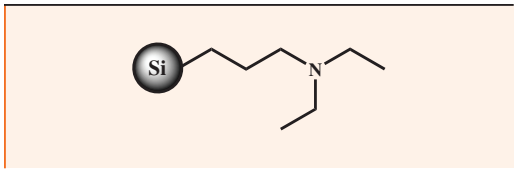
SiliaBond® Diethylamine is also used as weak anion exchanger (WAX) in SPE. It has a pka of ~ 10.5 and is preferred over **SiliaBond® TMA Chloride** (SAX) when performing *"Catch and Release"* purification of compounds bearing a permanent negative charge such as salts of sulfonic acids. Using SAX in this case could make the release of the compound of interest difficult, not to say irreversible, due to the strong interaction between the two strong ions.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (< 8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Dimethylamine (R45030B)

Loading: 1.4 mmol/g
Category: Base

DESCRIPTION

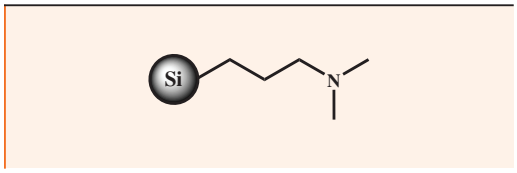
SiliaBond® Dimethylamine is a silica bound tertiary amine base that can be used in most applications requiring a tertiary amine, particularly as a HCl sponge. Silica bound ammonium salt by-products are easily separated by filtration. This dimethylamine immobilized on silica gel can be used as a base catalyst in Michael[1] or Knoevenagel[2] reactions. It can also be used as a catalyst for alkane oxidation.[3] Unlike polystyrene based resins, the amine is not a benzyl amine and therefore is not susceptible to cleavage by electrophiles.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (< 8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) Synlett, (1998) 625
2) J. Chem. Soc. Perkin Trans. I, (1989) 105
3) J. Org. Chem., 56 (1991) 1981

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Diol NON-ENDCAPPED (R35030B)

Loading: 1.0 mmol/g
Category: Scavenger and Chromatographic Stationary Phase

DESCRIPTION

SiliaBond® Diol may be used as boronic acid scavenger in the same way as PS-DEAM.

SiliaBond® Diol may also be used as polar sorbent in normal phase and aqueous size exclusion chromatography. Like bare silica, **SiliaBond® Diol** has the ability to form hydrogen bonds and the capacity to separate structural isomers. Since most of its surface is covered with organic functions, the **SiliaBond® Diol** absorbs less water, which leads to a more reproducible activity. It is also the sorbent of choice when working in normal phase in the presence of water. It has a different selectivity than bare silica gel and slight modifications in the composition of the solvent mixture may be necessary to obtain a similar retention. In SPE, it is used for the isolation of drugs and metabolites from physiological fluids.

APPLICATION NOTES

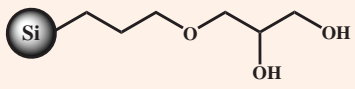
Scavenging Boronic Acid (p.75)

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Diphenylphosphine (R39030B)

Loading: 0.9 mmol/g
Category: Reagent, Other

DESCRIPTION

SiliaBond® Diphenylphosphine (or ***Si*-DPP**) can be used as linker for catalyst immobilization such as tetrakis-(triphenylphosphine)palladium(0) for Suzuki coupling.

APPLICATION NOTES

Suzuki Coupling

Palladium immobilization on **SiliaBond® Diphenylphosphine** (p.83)

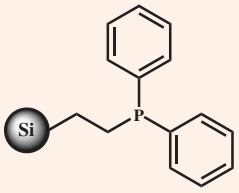
Suzuki Coupling using Palladium immobilized on **SiliaBond® Diphenylphosphine** (p.83)

SOLVENT COMPATIBILITY

Degassed solvents

PROLONGED STORAGE

Store under argon.



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® DMAP (R75530B)

Loading: 0.8 mmol/g
Category: Reagent

DESCRIPTION

SiliaBond® DMAP (or ***Si*-DMAP**) is the supported equivalent of 4-dimethylaminopyridine commonly used as a nucleophilic catalyst in a wide variety of reactions such as acylation, amidation or acetylation. These reactions are well known in organic synthesis and are very useful in various domains. **SiliaBond® DMAP** presents the advantage over its free counterpart as it can be removed by a simple filtration. One of the most important applications for this product is the *"Catch and Release"* for amide synthesis.

SiliaBond® DMAP can also be used in other types of reactions like esterification of alcohols and nucleophilic rearrangements.

APPLICATION NOTES

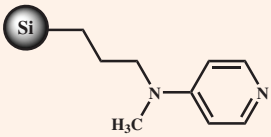
Sulfonamide Synthesis by Catch and Release (p.82)

SOLVENT COMPATIBILITY

All organic solvents

PROLONGED STORAGE

Keep cool (<8°C) and dry; store under argon.



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Fluorochrom NON-ENDCAPPED(R63730B)

Loading: 7% carbon
Category: Fluorous Phase

DESCRIPTION

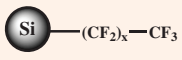
SiliaBond® Fluorochrom (or ***Si*-FCM**) is a fluorinated sorbent particularly useful for the separation of fluorine containing molecules from non-fluorous ones. This new technique is slowly gaining acceptance in the scientific community mostly for applications in SPE and HPLC. The applications of these types of sorbents are not limited to fluorous compounds but may also be used in chromatographic separations of non-fluorinated molecules, just like a regular reversed-phase would do, but with a different selectivity. **SiliaBond® Fluorochrom** presents the highest level of fluorine available on the market and has a proprietary structure.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

All these products are available in different sizes from lab scale to multi-ton scale.

SiliaBond® Glycidoxy NON-ENDCAPPED (R36030B)

Loading: 1.1 mmol/g
Category: Other

DESCRIPTION

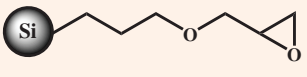
SiliaBond® Glycidoxy (or ***Si*-GLY**) may be used as a linker for further modification of the surface and for the immobilization of molecules bearing amino, hydroxy, mercapto and thiocarboxylic acid groups.

SOLVENT COMPATIBILITY

Anhydrous aprotic organic solvents

PROLONGED STORAGE

Keep cool (<8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Isocyanate (R50030B)

Loading: 1.2 mmol/g
Category: Nucleophile Scavenger

DESCRIPTION

SiliaBond® Isocyanate (or ***Si*-ISO**) is a versatile scavenger for nucleophiles. It readily scavenges amines, thiols, thiolates, alkoxides, and acidic phenols. Loading is determined by the removal of benzylamine in hexane at room temperature. ***Si*-ISO** has been used to scavenge amines in a *"cap-tag"* methodology for oligosaccharide preparation by automated solid phase synthesis[1] and in the preparation of 2,5-diketopiperazines and 1,4-benzodiazepine-2,5-diones.[2]

Silica supported isocyanate has superior reactivity in a broader range of solvents when compared to polymer-based materials. **SiliaBond® Isocyanate** is also available in SPE (**SiliaPrep™ Isocyanate**) format for flow through scavenging.

APPLICATION NOTES

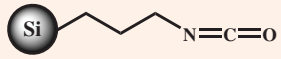
Scavenging Benzylamine (p.75)

SOLVENT COMPATIBILITY

Anhydrous aprotic organic solvents

PROLONGED STORAGE

Keep cool (<8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) Angew. Chem. Int. Ed., 40 (2001) 4433
2) Org. Lett., 4 (2002) 1167

SiliaBond® Maleimide (R71030B)

Loading: 0.7 mmol/g
Category: Other

DESCRIPTION

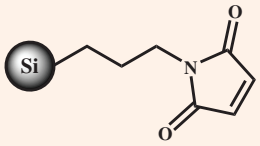
SiliaBond® Maleimide (or ***Si*-MAL**) has been designed to scavenge thiols. 90% of mercaptoethanol was removed with 4 eq. after 15 minutes in an aqueous environment. Complete scavenging was observed after 3 hours. **SiliaBond® Maleimide** can also be used to immobilize peptides and proteins through the cysteine residues. 95% of L-cysteine was immobilized after only 15 minutes in a pH 4.5 buffered solution using 4 eq. of **SiliaBond® Maleimide**.

SOLVENT COMPATIBILITY

Polar organic solvents (MeOH, DMF) and water

PROLONGED STORAGE

Keep cool (<8°C) and dry.



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Morpholine (R68030B)

Loading: 1.1 mmol/g
Category: Base

DESCRIPTION

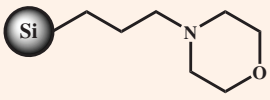
SiliaBond® Morpholine (or ***Si*-MOR**) is commonly used as an acid sponge. It is used in the same manner as morpholine functionalized polymers.[1,2]

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (<8°C) and dry.



25g
100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) J. Am. Chem. Soc., 119 (1997) 4882
2) Tetrahedron, 54 (1998) 3983

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Pentafluorophenyl (R67530B)

Loading: 9% carbon
Category: Fluorous Phase

DESCRIPTION

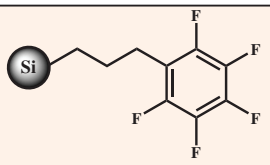
SiliaBond® Pentafluorophenyl (or ***Si*-PFP**) is a sorbent primarily used in the separation of molecules bearing fluorine atoms but may also be used in the separation of non-fluorous compounds[1] such as Taxol® and its derivatives.[2,3] Because of its phenyl ring, ***Si*-PFP** has a higher selectivity for aromatics containing molecules compared to the other fluorinated sorbents.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) J. Chromatogr., 511 (1990) 79
2) J. Nat. Prod., 61 (1998) 57
3) J. Chromatogr. B, 696 (1997) 99

SiliaBond® Phenyl (R34030B), NON-ENDCAPPED (R34130B)

Loading: 9% Carbon
Category: Chromatographic Stationary Phase

DESCRIPTION

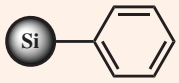
SiliaBond® Phenyl (or ***Si*-PHE**) is a sorbent of medium polarity used in reversed-phase chromatography. It has similar retention to **C8** but with a different selectivity especially for molecules containing aromatics and fatty acids.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Phenylmethylchloride (R56530B)

Loading: 1.2 mmol/g
Category: Other

DESCRIPTION

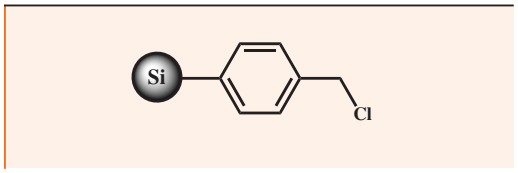
SiliaBond® Phenylmethylchloride (or ***Si*-PMC**) can be used as solid support for further derivatization.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



5 g, 10 g
25 g, 100 g
500 g
1 kg
5 kg
10 kg
25 kg*

SiliaBond® Piperazine (R60030B)

Loading: 0.8 mmol/g
Category: Base

DESCRIPTION

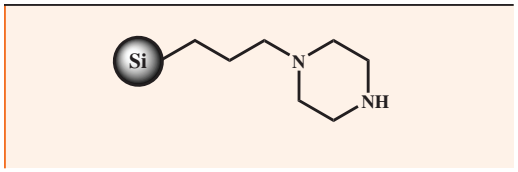
This **SiliaBond® Piperazine** (or ***Si*-PPZ**) is a useful deprotecting and scavenging agent for Fmoc[1] and Bsmoc[2] amino protecting group and as a solid phase Knoevenagel catalyst. According to the results of a study[1], ***Si*-PPZ** is superior to its polystyrene-based equivalent. **SiliaBond® Piperazine** may also be used to scavenge electrophiles.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (< 8°C) and dry.



10 g
25 g, 100 g
500 g
1 kg
5 kg
10 kg
25 kg*

RELATED PUBLICATIONS

1) J. Org. Chem., 48 (1983) 666
2) J. Org. Chem., 64 (1999) 4324

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Piperidine (R71530B)

Loading: 1.1 mmol/g
Category: Base

DESCRIPTION

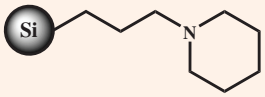
SiliaBond® Piperidine (or ***Si*-PIP**) is a general purpose tertiary amine base to scavenge acids, thereby avoiding salt elimination problems. Also used as a base catalyst for Knoevenagel condensation.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (< 8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Potassium Permanganate NON-ENDCAPPED (R23030B)

Loading: 20% wt/wt, adsorbed onto silica
Category: Oxidant

DESCRIPTION

Potassium permanganate is a strong oxidant that will oxidize methyl groups and alcohols to carboxylic acids. **SiliaBond® Potassium Permanganate** (or ***Si*-$KMnO_4$**) increases recoveries, facilitates work-up, and expands the scope of the chemistry because it can be used in all organic solvents eliminating solubility issues.[1] With **SiliaBond® Potassium Permanganate**, the manganese salt by-products stays adsorbed onto the silica.

SOLVENT COMPATIBILITY

All anhydrous organic solvents with the exception of the alcohol, aldehyde, and ketone-containing solvents.

PROLONGED STORAGE

Keep dry

RELATED PUBLICATION

1) Synlett, 10 (2001) 1555

25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Propyl Bromide (R55530B)

Loading: 1.5 mmol/g
Category: Other

DESCRIPTION

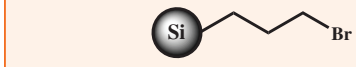
SiliaBond® Propyl Bromide (or ***Si*-PBR**) is a versatile solid support for amines and alcohols; more reactive than the chloride towards displacement.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Propylsulfonic Acid (R51230B)

Loading: 1.0 mmol/g
Category: Acid and Ion Exchanger

DESCRIPTION

Supported sulfonic acids such as **SiliaBond® Propylsulfonic Acid** (or **SiliaBond® SCX-2**) are in a class of strong acids widely used in different fields of synthetic organic chemistry. Their applications are well known and are used in a large number of settings going from drug discovery laboratories up to manufacturing processes. Among the applications we denote their use as acid catalysts, stationary phases for ion chromatography, and basic impurity scavengers. Meanwhile, the most common use is probably as strong cation exchanger (SCX) for the amine *"Catch and Release"* purification in SPE cartridge. This technique is becoming popular and some Universities have introduced the use of SCX SPE purification in their undergraduate teaching laboratories.

SiliCycle® offers **SiliaBond® Propylsulfonic Acid** and **SiliaBond® Tosic Acid (SCX)**. Both are considered strong cation exchanger, meaning that they maintain a negative charge throughout the pH scale. The aromatic ring of the **SiliaBond® Tosic Acid** makes it slightly more acidic than the other. However, internal tests have demonstrated that they both have comparable strength. The difference between the two products is mainly in the selectivity. **SiliaBond® SCX-2** presents a slightly more non-polar character than the **SiliaBond® SCX**, thus reducing secondary non-polar interactions with compounds.

APPLICATION NOTES

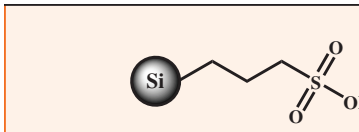
Amine purification – Catch and Release using **SiliaPrep™ Propylsulfonic Acid** (p.68)

SOLVENT COMPATIBILITY

All solvents aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
500g
1kg
5kg
10kg
25kg*

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Pyridine (R43030B)

Loading: 1.3 mmol/g
Category: Base

DESCRIPTION

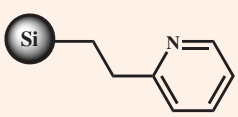
SiliaBond® Pyridine (or ***Si*-PYR**) can be used as a tertiary amine for acid removal. It can also be used to anchor Cu^{2+} ions, which can then be used as a catalyst for hydroquinone oxidation.[1]

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (<8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATION

1) J. Polym. Sci., Part A Polym. Chem., 32 (1994) 2457

SiliaBond® Pyridinium Chlorochromate NON-ENDCAPPED (R24030B)

Loading: 20% wt/wt, adsorbed onto silica
Category: Oxidant

DESCRIPTION

SiliaBond® Pyridinium Chlorochromate (or ***Si*-PCC**) is used for the general oxidation of alcohols to carbonyl compounds, selective oxidation of allylic and benzylic alcohols, organometallic oxidation, oxidative transpositions, oxidative cleavages, allylic and benzylic oxidation, and oxidative cyclizations.[1-4]

Using PCC immobilized onto silica gel provides anhydrous conditions that may otherwise promote side reactions and reduce yields. It greatly facilitates removal of polymeric reduced chromium by-products, and is compatible with acid-sensitive protecting groups.[5,6] When used in conjunction with ultrasounds, kinetics are increased and the amount of oxidant required to complete the reaction is decreased.[7-9]

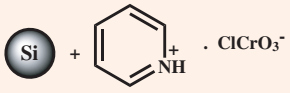
Oxidation of 4-*tert*-butylcyclohexanol to 4-*tert*-butylcyclohexanone with 2 eq. in anhydrous DCM at room temperature is complete after 6 hours.

SOLVENT COMPATIBILITY

Anhydrous DCM

PROLONGED STORAGE

Keep cool (<8°C) and dry.



25g
100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) J. Org. Chem., 54 (1989) 5387
2) Tetrahedron Lett., 42 (2001) 2141
3) Synlett, 10 (1999) 1630
4) Synth. Commun., 26 (1996) 225
5) J. Org. Chem., 58 (1993) 2509
6) J. Chem. Educ., 76 (1999) 974
7) J. Org. Chem., 48 (1983) 666
8) Liebigs Ann. Chem., (1993) 173
9) J. Org. Chem., 57 (1992) 3867

SiliaBond® Pyridinium Dichromate NON-ENDCAPPED (R24530B)

Loading: 20% wt/wt, adsorbed onto silica
Category: Oxidant

DESCRIPTION

SiliaBond® Pyridinium Dichromate (or ***Si*-PDC**) may be used as an alternative to ***Si*-PDC** in nucleoside and carbohydrate oxidation, particularly for fragile molecules.[1] **SiliaBond® PDC** can also be used in conjunction with *tert*butylhydroperoxide for a variety of oxidative transformations.[2]

PDC immobilized onto silica is a very convenient and effective reagent for oxidizing allylic and benzylic alcohols, saturated primary and secondary alcohols, and is compatible with acid-sensitive groups, such as cyclopropane ring or ketal function.[3]

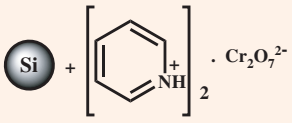
Oxidation of 4-*tert*-butylcyclohexanol to 4-*tert*-butylcyclohexanone with 2 eq. of ***Si*-PDC** in anhydrous DCM at room temperature is complete after 6 hours.

SOLVENT COMPATIBILITY

Anhydrous DCM

PROLONGED STORAGE

Keep cool (< 8°C) and dry.



25g
100g
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1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) J. Chem. Soc. Perkin Trans. I, (1982) 1967
2) J. Chem. Soc. Chem. Commun., 7 (1993) 651
3) Tetrahedron, 35 (1979) 1789

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Silver Nitrate NON-ENDCAPPED (R23530B)

Loading: 10% wt/wt, adsorbed onto silica
Category: Chromatographic Stationary Phase

DESCRIPTION

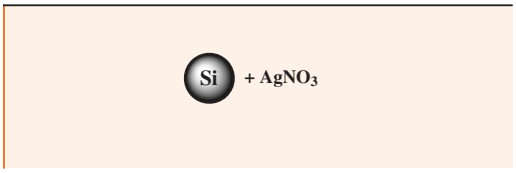
Chromatography with silver nitrate is a well known and established methodology for the separation of non polar compounds with similar polarities. Generally, with a mixture of unsaturated compounds such as alkenes, lipids, steroids, terpenes, etc, standard separation procedures lack in efficiency. However, by using **SiliaBond® Silver Nitrate** for your column chromatography along with **SiliaPlate™ Ag** for your TLC you will have the necessary tools to circumvent the problem. The theory behind this separation is based on the fact that silver ions can complex with unsaturated compounds (π bond). This kind of chromatography with silver nitrate impregnated silica gel is very easy to perform and is well documented.[1-4]

SOLVENT COMPATIBILITY

Anhydrous organic solvents. DMF and DMSO should be avoided.

PROLONGED STORAGE

Keep dry in the dark.



25g
100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) Tetrahedron, 57 (2001) 425
2) J. Chromatogr. A, 715 (1995) 372
3) J. Chromatogr., 38 (1968) 535
4) Microchem. J., 9 (1965) 227

SiliaBond® Succinic Anhydride (R64530B)

Loading: 1.0 mmol/g
Category: Nucleophile Scavenger

DESCRIPTION

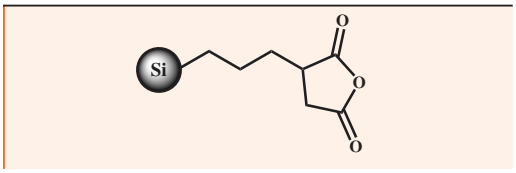
SiliaBond® Succinic Anhydride (or ***Si*-SAN**) scavenges primary and secondary amines. It is selective for amines in the presence of alcohols and is the preferred scavenger for anilines.

SOLVENT COMPATIBILITY

Anhydrous aprotic solvents

PROLONGED STORAGE

Keep cool (<8°C) and dry; store under argon.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® TAAcOH (R69030B)

Loading: 0.4 mmol/g
Category: Acid and Metal Scavenger

DESCRIPTION

SiliaBond® TAAcOH (or **SiliaBond® Triaminetetraacetic Acid**) is a silica bound metal scavenger for Pd(0), Ni(0) and Cu. It is the supported version of EDTA in its free form. It is an effective scavenger for metals in low or zero oxidation states, which includes many of the most synthetically useful catalysts such as tetrakis-(triphenylphosphine)palladium(0).

APPLICATION NOTES

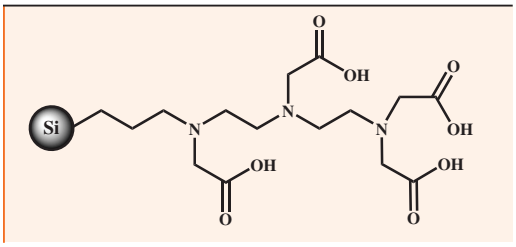
Scavenging Pd Complexes (p.78)

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® TAAcONa (R69230B)

Loading: 0.4 mmol/g
Category: Metal Scavenger

DESCRIPTION

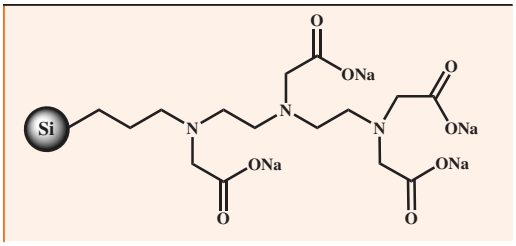
SiliaBond® TAAcONa or (or **SiliaBond® Triaminetetraacetate, sodium salt**) is a silica bound metal scavenger for Pd(II), Ni(II) and Cu. It is the supported version of EDTA in its sodium salt form. It is an effective scavenger for metals in higher oxidation state, 2+ or higher.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



10g
25g, 100g
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1kg
5kg
10kg
25kg*

All these products are available in different sizes from lab scale to multi-ton scale.

SiliaBond® TBA Chloride NON-ENDCAPPED(R65530B)

Loading: 0.5 mmol/g
Category: Ion Exchanger

DESCRIPTION

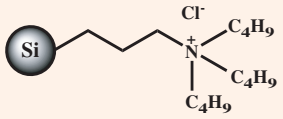
SiliaBond® TBA Chloride (or ***Si*-TBACl**) may be used in the same way as **SiliaBond® TMA Chloride** (see p.56). ***Si*-TBACl** is more sterically hindered, which confers a different selectivity than the other anion exchangers.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® TBD (R68530B)

Loading: 0.9 mmol/g
Category: Base and Metal Scavenger

DESCRIPTION

SiliaBond® TBD (1,5,7-Triazabicyclo[4.4.0]dec-5-ene) (or ***Si*-TBD**) is a silica bound bicyclic guanidine moiety that is sufficiently basic to deprotonate moderately acidic hydrogens. It is most commonly used for the alkylation of phenols (Williamson ether synthesis) and amines, and the esterification of carboxylic acids using alkyl halides. It may also be used to scavenge boronic acids such as phenylboronic acid: 98% removal with 1 equivalent in DCM at room temperature after 1 h.

The alkylation reaction can be done with a *"Catch and Release"* approach, in which the acidic phenol is bound to the **SiliaBond® TBD** and released upon addition of the electrophile. Excess phenol will remain bound to the silica facilitating work up and purification.

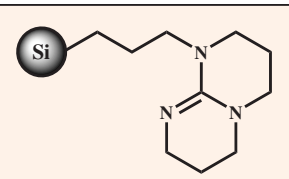
Additional published applications include the alkylation of activated methylene containing compounds, dehalogenation of organic halides, and the synthesis of aryl triflates. As a base catalyst it has been used in Michael reactions, Knoevenagel condensations, Robinson annulations and nucleophilic epoxidations.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (<8°C) and dry; store under argon.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) Angew. Chem. Int. Ed., 36 (1997) 2661
2) Micro. Meso. Mater., 35-36 (2000) 143

SiliaBond® Thiol (R51030B)

Loading: 1.2 mmol/g
Category: Metal Scavenger

DESCRIPTION

SiliaBond® Thiol (or ***Si*-Thiol**) is a robust metal scavenger for a variety of metals including Pd, Pt, Cu, Ag, and Pb under a wide range of conditions. It has been used in pharmaceutical processes up to the pilot plant scale. It is our most versatile metal scavenger and is the preferred metal scavenger for Pd(II), Cu, Ag, and Hg.

APPLICATION NOTES

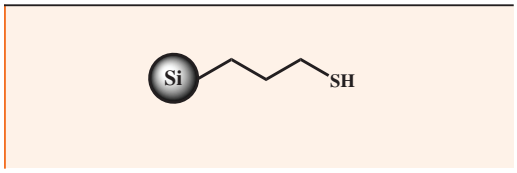
Scavenging Pd Complexes (p.78)

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

Visit our website for **SiliaBond® Thiol** metal scavenger video.

RELATED PUBLICATIONS

1) Chem. Commun., 1 (1999) 69
2) Chem. Commun., 13 (2000) 1145
3) Langmuir, 17 (2001) 528

SiliaBond® Thiourea (R69530B)

Loading: 1.2 mmol/g
Category: Metal Scavenger

DESCRIPTION

SiliaBond® Thiourea (or ***Si*-THU**) is a versatile metal scavenger for all forms of palladium and is widely used in the pharmaceutical industry. It works particularly well in organic solvents. It can also be used to scavenge Ag,[1] Pt,[2] Ru, Rh, and Hg. Once complexed with a transition metal, it has been reported as being an effective catalyst.[3]

APPLICATION NOTES

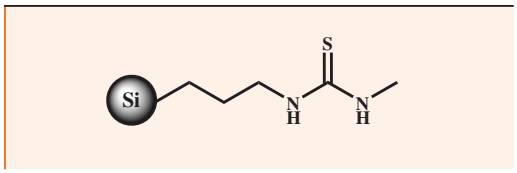
Scavenging Pd Complexes (p.79)

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATION

1) Analyst, 125 (2000) 147
2) Analyst, 125 (2000) 1205
3) Chem. Mater., 4 (1992) 243

**All these products are available in different sizes from lab scale to multi-ton scale.*

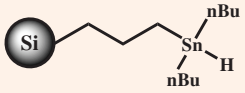
SiliaBond® Tin Hydride (R72530B)

Loading: 0.6 mmol/g
Category: Reagent

DESCRIPTION

SiliaBond® Tin Hydride (or ***Si*-SnH**) is a highly selective reagent for the reduction or dehalogenation of alkyl halides. This is a very efficient reagent that gives good yields after only few hours. Solution phase organotin compounds are highly toxic and the silica support allows them to be safely used and removes the risk of tin contamination in the final product. As its homologous solution phase product, **SiliaBond® Tin Hydride** can be used in radical reaction in catalytic cycle or not. The transformation of 1-bromoadamantane to adamantane gave 92% yields in the conditions described in the sample procedure (p.72).

This product can also be used for the Barton-McCombie deoxygenation, for Giese reaction and for ring enlargement cyclohexadienones.



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

APPLICATION NOTES

Reduction of Halides (p.72)

SOLVENT COMPATIBILITY

Anhydrous and degassed solvents

PROLONGED STORAGE

Keep cool (<8°C) and dry; store under argon in the dark.

RELATED PUBLICATIONS

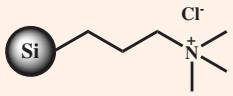
1) Green Chem., 3 (2001) 71
2) J. Am. Chem. Soc., 119 (1997) 6949

SiliaBond® TMA Chloride NON-ENDCAPPED(R66530B)

Loading: 1.1 mmol/g
Category: Ion Exchanger

DESCRIPTION

SiliaBond® TMA Chloride (or **SiliaBond® SAX**, ***Si*-TMACl**) is mainly used as a strong anion exchanger (SAX) in ion chromatography[1,2] and ion exchange SPE. The function bears a positive charge across the whole pH range as well as in organic solvents. It is especially used for the *"Catch and Release"* purification of weak acids. For the purification of strong acids, a weak anion exchanger (WAX) such as **SiliaBond® Triamine**, **Amine** or **Diethylamine** is preferred. Since the chloride ion is bound relatively strongly to the ammonium, it may be suited to activate the ion exchanger by changing the chloride for an acetate counter ion. This can be done following the procedure described on page 69.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

APPLICATION NOTES

Counter Anion Exchange (p.69)

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry

RELATED PUBLICATIONS

1) J. Chromatogr. 119 (1976) 25
2) J. Chromatogr. 123 (1976) 109

SiliaBond® Tosic Acid (R60530B)

Loading: 0.8 mmol/g
Category: Acid, Nucleophile Scavenger, and Ion Exchanger

DESCRIPTION

SiliaBond® Tosic Acid (or **SiliaBond® SCX**, ***Si*-TsOH**) is a versatile bound strong acid with a pka <<1. It is widely used for the scavenging of amines and other basic functionalities, including weakly basic anilines, borohydrides, and metals such as Ni and Ag. It can also be used as an acid catalyst for organic reactions.[1] ***Si*-TsOH** can serve as an alternative method to quench reactions replacing aqueous or organic soluble acids.

SiliaBond® Tosic Acid has been optimized for use in organic applications. It will not dissolve in methanol or any other solvents. It delivers much higher recovery and has better flow characteristics than corresponding polymer.

The main application of **SiliaBond® Tosic Acid** is as strong cation exchanger (SCX) in *"Catch and Release"* purification of amines. It is also widely used as stationary phase in ion exchange chromatography (IEC).[2,3] Please see **SiliaBond® SCX-2**'s product (p.49) for more information.

APPLICATION NOTES

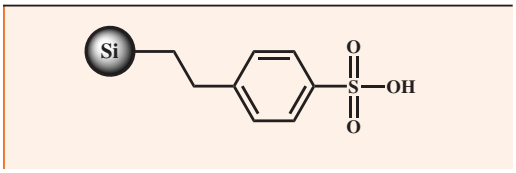
Catch and Release – Amines purification using **SiliaPrep™ Tosic Acid** (p.69)

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



25g
100g
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1kg
5kg
10kg
25kg*

RELATED PUBLICATIONS

1) J. Org. Chem., 54 (1989) 5437
2) J. Chromatogr., 117 (1976) 269
3) J. Chromatogr., 123 (1976) 109

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Tosyl Chloride (R44030B)

Loading: 1.0 mmol/g
Category: Nucleophile Scavenger

DESCRIPTION

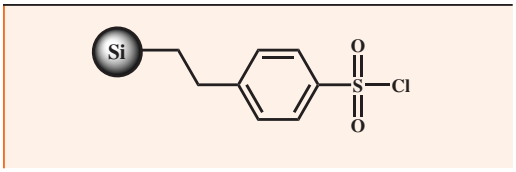
SiliaBond® Tosyl Chloride (or ***Si*-TsCl**) readily reacts with nucleophiles such as amines and alcohols. Reaction with alcohols yields the bound tosylate, which can then be used to synthesize amines and oxazolines.

APPLICATION NOTES

Scavenging nucleophile with **SiliaBond® Tosyl Chloride** (p.77)

Solid Support: Linking alcohol to **SiliaBond® Tosyl Chloride** (p.80)

Solid Support: Cleavage of the tosylate (p.80)



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SOLVENT COMPATIBILITY

Anhydrous aprotic solvents. Unstable in DMF.

PROLONGED STORAGE

Keep cool (<8°C) and dry; store under argon.

SiliaBond® Tosyl Hydrazine (R61030B)

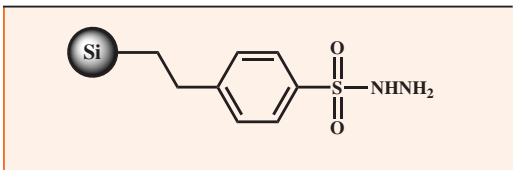
Loading: 1.5 mmol/g
Category: Scavenger

DESCRIPTION

SiliaBond® Tosyl Hydrazine (or ***Si*-TsNHNH$_2$**) is an effective scavenger of carbonyl compounds. It has been used to selectively remove aldehydes in the presence of ketones since the scavenging of ketones requires an acid catalyst. Once bound, sulfonyl hydrazones can be utilized in other synthetic transformations.

APPLICATION NOTES

Scavenging Aldehyde/Ketone with **SiliaBond® Tosyl Hydrazine** (p. 74)



5g, 10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SOLVENT COMPATIBILITY

Aprotic, non carbonyl containing solvents

PROLONGED STORAGE

Keep cool (<8°C) and dry; store under argon.

SiliaBond® Triamine (R48030B)

Loading: 1.2 mmol/g
Category: Base and Metal Scavenger

DESCRIPTION

SiliaBond® Triamine (or **SiliaBond® WAX-3**, ***Si*-TRI**) is effective for scavenging metals such as Pb, Co, Ru, and Pd. Our internal tests have shown it to be the preferred scavenger for Pb. It can also be used as a scavenger for acid chlorides, isocyanates, and other electrophiles.

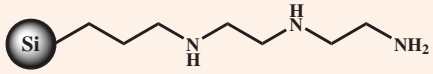
SiliaBond® Triamine may also be used as a weak anion exchanger (WAX) in the same way as **SiliaBond® Amine** and **SiliaBond® Diethylamine,** and is the closest silica equivalent of polyethyleneimine commonly used on the market.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep cool (< 8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

SiliaBond® Tridecafluoro (R63530B)

Loading: 10% carbon
Category: Fluorous Phase

DESCRIPTION

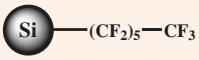
SiliaBond® Tridecafluoro (or ***Si*-TDF**) is a sorbent primarily used to separate fluorous molecules. It can also be used in fluorous solid-phase extraction (FSPE) of fluorous-tag compounds as reported for the synthesis of oligosaccharide.[1] It has a fluorine content that is between the **SiliaBond® Fluorochrom** and the **SiliaBond® Pentafluorophenyl**.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATION

1) Org. Lett. 9 (2007) 2285

**All these products are available in different sizes from lab scale to multi-ton scale.*

SiliaBond® Urea (R67030B)

Loading: 1.3 mmol/g
Category: Other

DESCRIPTION

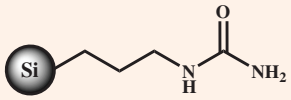
Used for adsorbing cationic surfactants[1] and as polar stationary phase for chromatography.

SOLVENT COMPATIBILITY

All solvents, aqueous and organic

PROLONGED STORAGE

Keep dry



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

RELATED PUBLICATION

1) Tenside Surf. Det., 37 (2000) 381

Visit our website for new metal scavengers and application notes.

SiliaCat® product line introduction

Being attentive to customers' needs, SiliCycle® is introducing a new product line: **SiliaCat®**. **SiliaCat®** is a new heterogeneous catalyst family made using innovative technology that confers very desirable properties in catalysis. The **SiliaCat®** is an organically modified silica made by a proprietary encapsulation process resulting in a new chemical matrix structure[1]. **SiliaCat®** is a leach-proof organoceramic matrix with high catalyst loading and surface area that in turns, provide high Turn Over Number (TON).

The **SiliaCat® TEMPO** is a highly efficient and selective catalyst compared to homogeneous phase TEMPO and has a superior performance compared to silica supported **TEMPO** in terms of both selective activity and stability[2].**SiliaCat® TEMPO** can be used for solid-state synthesis in which no contamination of the product and high selectivity are required. It is made of a proprietary class of organosilica-entrapped radicals suitable for the selective oxidation of delicate substrates into valued carbonyl and carboxylic acid derivatives.

SiliaCat® TEMPO is an effective and useful oxidizing catalyst that presents unique advantages:

- Excellent yield
- Selective activity
- High stability
- Recyclable

Such sol-gel hybrid organic-inorganic materials meet all the industry requirements for oxidation catalyst including versatility, as they do not require the use of stoichiometric amounts of inorganic (Cr, Mn, Co, etc) or organic oxidants.

SiliaCat® TEMPO (R723-100)

Loading: 0.9 mmol/g
Category: Oxidant

DESCRIPTION

SiliaCat® TEMPO is an effective and useful oxidizing catalyst for delicate primary and secondary alcohol substrates into valued carbonyl derivatives.

APPLICATION NOTES

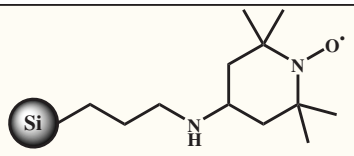
Oxidation of Alcohols (p.72)

SOLVENT COMPATIBILITY

All organic solvents (grade HPLC) except alcohols.

PROLONGED STORAGE

Keep cool (<8°C) and dry.



10g
25g, 100g
500g
1kg
5kg
10kg
25kg*

Visit our website for new **SiliaCat®** products to come.

RELATED PUBLICATIONS

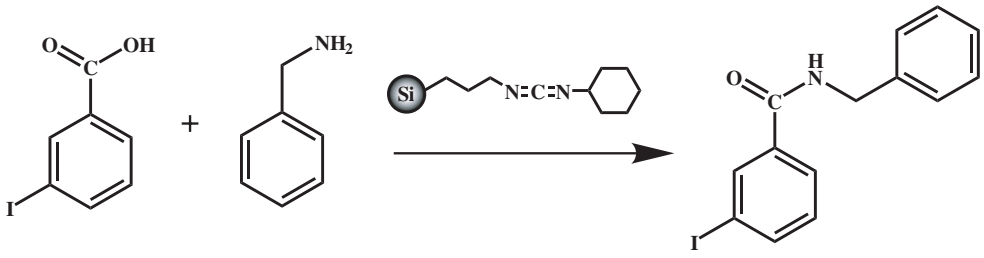
1) Org. Process Res. Dev., (2007) published on Web 05/16/2007.
2) Adv. Synth. Catal. 344 (2002) 159.

**All these products are available in different sizes from lab scale to multi-ton scale.*

Tel.: 1 418.874.0054 Fax: 1 418.874.0355 Toll-free: 1 877.SILICYCLE (North America only) info@silicycle.com
www.SiliCycle.com

Application notes

Amide synthesis



Results of amide bond formation using SILIABOND® Carbodiimide

#	Acid	Amine	% Yield (% Purity)		
			Bulk – 1	Bulk – 2	Cartridge
1	Benzoic Acid	Aniline	73.5 (99.1)	70.1 (96.4)	81.2 (97.2)
2	Benzoic Acid	Benzylamine	100.0 (95.4)	80.1 (98.1)	100.0 (98.7)[A]
3	Benzoic Acid	Phenylethylamine	98.7 (97.1)	78.7 (98.3)	100.0 (98.8)
4	Phenoxyacetic Acid	*Tert*-Butylamine	100.0 (97.4)	100.0 (94.0)	98.2 (94.5)[A,C]
5	Phenoxyacetic Acid	1,2,3,4-Tetrahydro-isoquinoline	99.8 (95.0)	100.0 (92.5)	97.2 (92.4)[A]
6	Boc-Phe-OH (L)	Phenylethylamine	100.0 (97.6)	100.0 (97.6)	99.2 (90.1)
7	Fmoc-Phe-OH (D)	Phenylethylamine	N.A.	100.0 (>95)[B]	N.A.
8	Z-Val-OH	Phenylethylamine	100.0 (>95)[B]	93.5 (>95)[B]	100.0 (>95)[B]
9	3-Iodobenzoic Acid	Benzylamine	100.0 (98.5)	100.0 (97.1)	100.0 (94.5)
10	Heptanoic Acid	Ethanolamine	72.2 (95.5)	84.3 (98.0)	81.3 (93.0)[D]

A: 4 hours reaction time; B: Determined by ^{1}H NMR; C: in DMF; D: No HOBt, Conditions: As described in the sample procedure unless stated otherwise. Bulk – 1 and – 2 refer to the method 1 and 2 for bulk synthesis as described in the following pages.

AMIDE SYNTHESIS USING **SILIA*BOND*® Carbodiimide**

SAMPLE PROCEDURE

In Bulk – Method 1

- **Silia*Bond*® Carbodiimide** (2.0g, 2.0 eq.), acid (1.5 eq.), and HOBt (1.7 eq.) were briefly mixed in DCM (10mL).
- The amine (1.0 eq.) was added.
- The reaction was stirred overnight at room temperature.
- The reaction was monitored by TLC (EtOAc/Hexanes, 1:1).
- When the reaction was completed, excess HOBt, acid, and amine (if necessary) were scavenged using **Silia*Bond*® Carbonate** (6.8 eq.) and **Silia*Bond*® Tosic Acid** (2.0 eq.). Total volume of the mixture was adjusted to keep a silica/solvent ratio of 1g/5 mL.
- Scavengers were allowed to react for 1 hour at room temperature.
- **Silia*Bond*®** were filtered off and rinsed with DCM (3 × 10 mL).
- Solvent evaporation gave the desired crude mixture.
- The corresponding amides were analyzed by ^{1}H and ^{13}C NMR, or GC-MS.
  (Yield corresponds to the mass of isolated product. Purity was determined by GC-FID.)

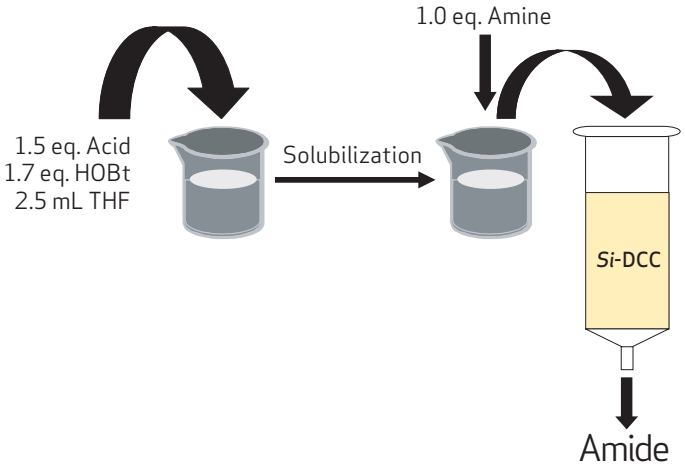
In Bulk – Method 2

- Amine (1.0 eq.), acid (2.0 eq.), and **Silia*Bond*® Carbodiimide** (2g, 3.0 eq.) in DCM (10 mL) or DCM/DMF mixture were mixed together.
- The reaction was stirred overnight at room temperature.
- The reaction was monitored by TLC (EtOAc/Hexane, 1:1).
- The excess of acid was scavenged with **Silia*Bond*® Amine** (4.0 eq.) and excess amine (if necessary) was scavenged with **Silia*Bond*® Tosic Acid** (2.0 eq.).
- Total volume of the mixture was adjusted to keep a silica/solvent ratio of 1 g/5 mL.
- Scavengers were allowed to react for 1 hour at room temperature.
- **Silia*Bond*®** were filtered off and rinsed with DCM (3 × 10 mL).
- Solvent evaporation gave the desired crude mixture.
- The corresponding amides were analyzed by ^{1}H and ^{13}C NMR, or GC-MS.
  (Yield corresponds to the mass of isolated product. Purity was determined by GC-FID.)

In Cartridges

- **Silia*Prep*™ Carbodiimide** (2.0 g, 12 mL, 3.0 eq.) was placed on a vacuum manifold equipped with individual valves (closed).
- The acid (1.5 eq.) and HOBt (1.7 eq.) were mixed together in 2.5mL of THF (or DCM) until complete solubilization, after which the amine (1.0 eq.) was added.
- The resulting solution was loaded onto the **Silia*Prep*™ Carbodiimide** and left to react overnight.
- The reaction can be monitored by TLC (EtOAc/Hexane, 1:1).
- The valve was open to drain the solution rinsing with DCM (3 × 10mL).
- The excess of HOBt, acid, and amine (if necessary), were scavenged using **Silia*Bond*® Carbonate** (6.8 eq.) and **Silia*Bond*® Tosic Acid** (2.0 eq.).
- Scavengers were allowed to react for 1 hour at room temperature.
- **Silia*Bond*®** were filtered off and rinsed with DCM (3 × 5 mL).
- Solvent evaporation gave the desired crude mixture.
- The corresponding amides were analyzed by ^{1}H and ^{13}C NMR, or GC-MS.
  (Yield corresponds to the mass of isolated product. Purity was determined by GC-FID.)

How to use **Silia*Prep*™** Carbodiimide for amide synthesis.



AMIDE SYNTHESIS USING SILIA*BOND*® Dichlorotriazine

Silia*Bond*® Dichlorotriazine (***Si*-DCT**) must be activated with *N*-methylmorpholine (NMM) to give a morpholinium salt that can react with the carboxylic acid to form the activated ester. It may then react with an amine to form the amide or safely be stored for use at a later date. ***Si*-DCT** may also be used for synthesis of Weinreb amides and acylsulfonamides.

Silia*Bond*® Dichlorotriazine provides a fast clean route to amide bond formation with complete conversion in about an hour compared to most bound coupling reagents that requires overnight incubation.

Amide bond formation using **Silia*Bond*® Dichlorotriazine**

#	Acid	Amine	% Yield (% Purity)	
			Bulk	Cartridge
1	Benzoic acid	Aniline	100.0 (98.9)[C]	100.0 (98.5)[D]
2	Benzoic acid	Benzylamine	100.0 (97.8)	99.6 (96.7)
3	Benzoic acid	Phenylethylamine	100.0 (98.4)	99.9 (96.5)
4	Phenoxyacetic acid	*Tert*-Butylamine	99.5 (99.0)	92.7 (95.3)[E]
5	Phenoxyacetic acid	1,2,3,4-Tetrahydro-isoquinoline	91.1 (92.0)	70.0 (94.0)[E]
6	Boc-Phe-OH (L)	Phenylethylamine	99.8 (97.9)	91.1 (95.4)
7	Fmoc-Phe-OH (D)[H]	Phenylethylamine	100.0 (>95)[B]	100.0 (>95)[B]
8	Z-Val-OH	Phenylethylamine	100.0 (>95)[B]	98.3 (>95)[B]
9	3-Iodobenzoic acid	Benzylamine	100.0 (99.0)	99.9 (98.9)
10	Heptanoic acid	Ethanolamine	78.2 (68.0)[F]	53.2 (95.0)[G]

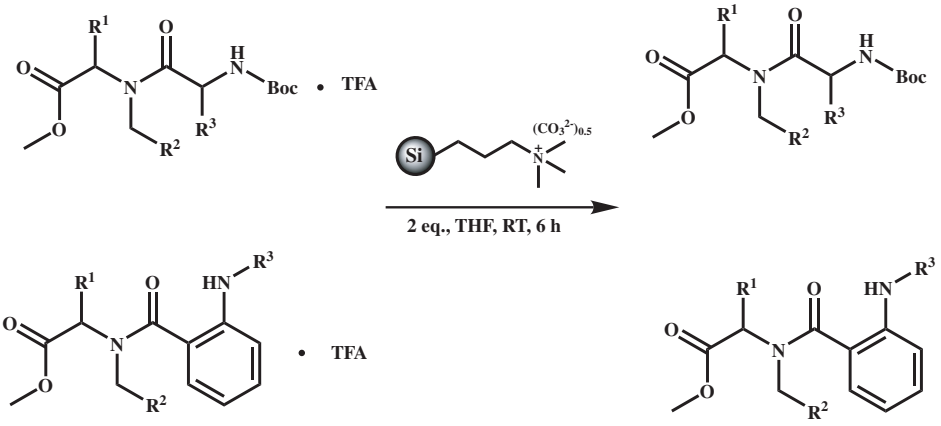
A: Determined by GC-FID; B: Determined by ^{1}H NMR; C: 3 hours reaction time; D: Amine overnight incubation; E: Acid overnight incubation; F: Use 4 eq. of acid and allow to react for 1 hour before adding amine; G: Use 4 eq. of acid; H: Do not use scavengers which would deprotect the amine.
Conditions: As described in the sample procedure unless stated otherwise.

SAMPLE PROCEDURE

Same as described for Weinreb amides synthesis on page p. 85.

Amine free basing

Silia*Bond*® Carbonate (***Si*-CO_3**) has been used to free base diversified libraries of 2,5-diketopiperazines and 1,4-benzo-diazepine-2,5-diones[1]



SAMPLE PROCEDURE

Amine free basing – using Silia*Bond*® Carbonate

- Add 2-4 eq. of *Si*-CO_3 in respect to the amine.
- Stir for 2 hours at room temperature.
- Remove the **Silia*Bond*® Carbonate** by filtration and rinse with THF.
- Solvent evaporation lead to the desired salt free amine.

Amine free basing – using Silia*Prep*™ Carbonate

- **Silia*Prep*™** Carbonate (2-4 eq. of *Si*-CO_3 in respect to the amine) is conditioned with THF.
- Load the amine solution (in THF) onto the **Silia*Prep*™ Carbonate**
- Free salt amine is eluted with THF.

RELATED PUBLICATION

1) Org. Lett., 4(7) 2002, 1167

Catch and Release

AMINES PURIFICATION USING SILIA*PREP*™ PROPYLSULFONIC ACID

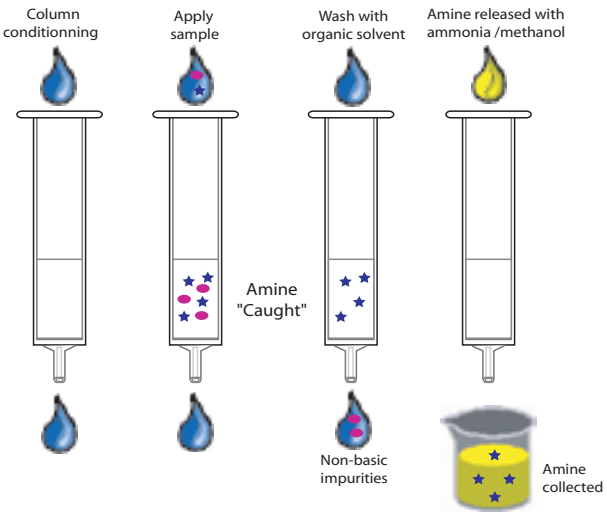
SAMPLE PROCEDURE

Amines purification – Catch and Release using **Silia*Prep*™ Propylsulfonic Acid**

The amine (1 eq.) was dissolved in methanol (2500 ppm)

Silia*Prep*™ Propylsulfonic Acid: 0.5g (2 eq.) or 1g (4 eq.)

1 – Column conditioning: 10mL of methanol
2 – Sample application
3 – Washing with 10mL of methanol (1mL/min)
4 – Release of the amine: 10mL of 2 M ammonia/methanol



Amine "Catch and Release" purification with **Silia*Bond*® SCX** *or* **SCX-2**

Catch and Release of amines using Silia*Prep*™ Propylsulfonic Acid (SCX-2)

Amine	# eq.	Catch	Release
Tributylamine	2	98%	90%
Tributylamine	4	100%	100%
Aniline	2	100%	100%
2-Aminothiazole	4	100%	100%
4-Nitroaniline	4	100%	100%

Starting with amine concentration of 2500 ppm in MeOH.

AMINES PURIFICATION USING SILIAPREP™ TOSIC ACID

SAMPLE PROCEDURE

Amines purification – Catch and Release using **SiliaPrep™ Tosic Acid**

The amine (1 eq.) was dissolved in methanol (2500 ppm)

SiliaPrep™ Tosic Acid: 0.5g (2 eq.) or 1g (4 eq.)

1 – Column conditioning: 10mL of methanol
2 – Sample application
3 – Wash with 10mL of methanol (1mL/min)
4 – Release of the Amine: 10mL of 2 M ammonia/methanol

Catch and Release of amines using **SiliaPrep™ Tosic Acid (SCX)**

Amine	# eq.	Catch	Release
Tributylamine	2	98%	97%
Tributylamine	4	100%	100%
Aniline	2	100%	100%
Aniline	4	100%	100%
2-Aminothiazole	4	100%	100%
4-Nitroaniline	4	100%	100%

Starting with amine concentration of 2500 ppm in MeOH.

Counter anion exchange using SiliaBond® TMA Chloride

SAMPLE PROCEDURE

Counter chloride anion exchange for acetate using SiliaPrep™ TMA Chloride

1 – Rinse the **SiliaPrep™ TMA Chloride** with deionized water.
2 – Rinse the cartridge with 0.1 M solution of acetic acid/acetate buffer (2 times its volume).
3 – Equilibrate the column with 0.01 M of the same buffer (1 column volume).
4 – It is now as **SiliaPrep™ TMA Acetate**

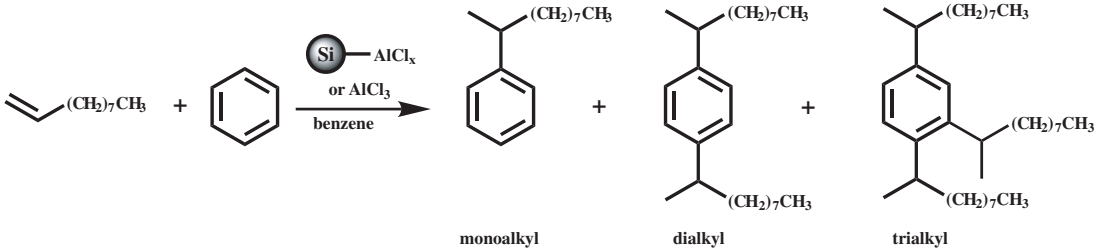
Weak acids purification - Catch and Release **using SiliaPrep™ TMA Acetate**

Use the SiliaBond® TMA Acetate prepared as described above.

1 – Sample application.
2 – Interference elution: use the equilibration buffer (0.01 M acetic acid/acetate buffer) to which 10-30% of an organic solvent (MeOH or MeCN) may be added to remove organic non-ionized species. In order to make sure the analyte is not eluted in this part, test its presence in the filtrate before continuing.
3 – Analyte elution: the analyte may be eluted by increasing the ionic strength of the eluent to 0.1-0.3 M. It is also possible to neutralize the charge of the analyte by lowering the pH to two units below its pKa. If the analyte is in organic solvent, use 5% of acetic acid in methanol.

Friedel-Crafts alkylation

Formation of Linear Alkyl Benzene (LAB) by Friedel-Crafts alkylation
in homogeneous and heterogeneous conditions



Formation of Linear Alkyl Benzene (LAB) by Friedel-Crafts alkylation
in homogeneous and heterogeneous conditions

Alkene	Catalyst	Alkene Conversion (%)	Selectivity Towards Alkylbenzene		
			Mono	Di	Tri
1-hexene[1]	$AlCl_3$	100	58.6	31.1	10.3
1-hexene	*Si*-$AlCl_x$	100	71.0	28.0	1.0
1-decene[1]	$AlCl_3$	100	68.5	22.5	9.0
1-decene	*Si*-$AlCl_x$	100	80.0	20.0	–

Conversion determined by GC-MS

SAMPLE PROCEDURE

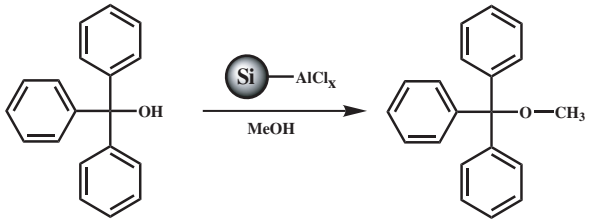
Friedel-Crafts alkylation

- Stir **Silia*Bond*® Aluminum Chloride** (0.03 eq.) in anhydrous benzene.
- Typical reaction solvent volume: 5mL/g of **Silia*Bond*® Aluminum Chloride**
- Add the alkene (1.0 eq.) slowly (over 30 min)(small exotherm observed).
- After the addition is completed, remove the catalyst by filtration. (The resulting products were analyzed by GC-MS.).

RELATED PUBLICATION

1) J. Catal., 195 (2000) 412

Formation of methyl triphenylmethyl ether



SAMPLE PROCEDURE

Ether formation

- Triphenylcarbinol (1.0 eq.) was added to a solution of **Silia*Bond*® Aluminum Chloride** (1.15 eq.) in anhydrous methanol.
- The mixture was heated to 60°C until completed by TLC (90 min).
- The catalyst was removed by filtration and the product analyzed by ^{1}H NMR.

Ether formation with supported Aluminum Chloride

Alcohol	Catalyst	Conversion (%)
Triphenylmethanol	Si- AlCl$_x$	95.0
	P-AlCl$_3$	81.5
tert-Butyl alcohol	Si-AlCl$_x$	60.0
	P-AlCl$_3$	0.0
Benzyl alcohol	Si-AlCl$_x$	40.0
	P-AlCl$_3$	0.0

Conversion determined by ^{1}H NMR

Oxidation of alcohols using SiliaCat® TEMPO

SAMPLE PROCEDURE

Method 1 - Stoechiometric

Activation

- Add 4 M HCl (6 eq.) in dioxane to a stirred 0.5 M solution of *N*-chlorosuccinimide (5 eq.) in DCM.
- Stir for 5 min before adding ***Si*-TEMPO** (1 eq.).
- Stir for 15 min.
- Filter and wash with anhydrous dioxane and DCM.
- Dry under vacuum.

Oxidation

- Mix the alcohol (1 eq.) with freshly activated ***Si*-TEMPO** (4 eq.) in anhydrous DCM.
- For the oxidation of primary alcohols, stir for 1 hour and for secondary alcohols, for 2 hours.
- **SiliaCat® TEMPO** was filtered off and rinsed with DCM to give the desired crude mixture.

Oxidation of alcohols using **SiliaCat® TEMPO**

Alcohol	PS-TEMPO (5 eq.)	Si-TEMPO (4 eq.)
Benzyl alcohol	>95%	>95%
3-phenyl-1-propanol	90%	>95%
1-phenyl-1-propanol	>95%	>95%

Conversion determined by GC-MS

Method 2 - Catalytic[1]

- Add 0.4 M solution of the alcohol (1 eq.) in DCM and 0.5 M solution of KBr (0.1 eq.) to ***Si*-TEMPO** (0,01 eq.)
- Cool the mixture to 0° C.
- Add 0.74 M solution of aqueous NaOCl (2.5 eq) buffered with $NaHCO_3$ to pH 9.1
- Stir vigorously for 1 hour.
- Filter and dry the organic phase with $MgSO_4$ to give the desired crude product.

RELATED PUBLICATION

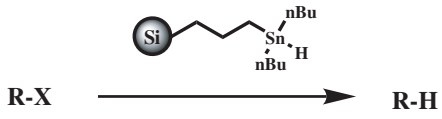
1) Org. Process Res. Dev., (2007) published on Web 05/16/2007.

Reduction of halides using SiliaBond® Tin Hydride

SAMPLE PROCEDURE

- To a dry flask under argon atmosphere were added **SiliaBond® Tin Hydride** (2.0 eq.), the halide (1.0 eq.), and AIBN (0.02 eq., optional catalyst) in anhydrous and degassed benzene.
- The solution was stirred at 60°C for 4 hours.
- The mixture was filtered, washed with benzene, and the solvent removed under vacuum.
- The yield was determined by the mass of isolated product and the purity by GC-MS and ^{1}H NMR.

Reduction of halides with **SiliaBond®** Tin Hydride



Reductive amination using SiliaBond® Cyanoborohydride

Reductive amination using **SiliaBond® Cyanoborohydride**

Reaction	Starting Materials		Solvent	Yield[a]
	Amine	Carbonyl		
1	Piperidine	Benzaldehyde	THF/AcOH (95/5)	100%
2	1-Benzyl-3-propylamine	Cyclohexanone	THF/AcOH (75/25)	100%
3	*N*-Benzylmethylamine	Cyclohexanone	THF/AcOH (95/5)	100%
4	*N*-Benzylmethylamine	Benzaldehyde	DCM/AcOH (90/10)	96%

[a] *Yield determined by GC-MS*
Reactions conditions: RT/24 h, 2.5 eq. of ***Si*-CBH**. *For reactions 1-3: 2.0 eq. of the amine and 1 eq. of the carbonyl. For reaction 4: 1.0 eq. of the amine and 3.0 eq. of the carbonyl.*

SAMPLE PROCEDURE

- To **SiliaBond® Cyanoborohydride** (1.0g, 2.5 eq.) was added 10mL of THF/AcOH (95/5).
- The amine (2 eq.) and the acid (1 eq.) were added.
- Mixture was shaken at room temperature for 24h.
- **SiliaBond®** was filtered off and rinsed with THF/AcOH (95/5).
- Solvent evaporation lead to the desired crude amine.

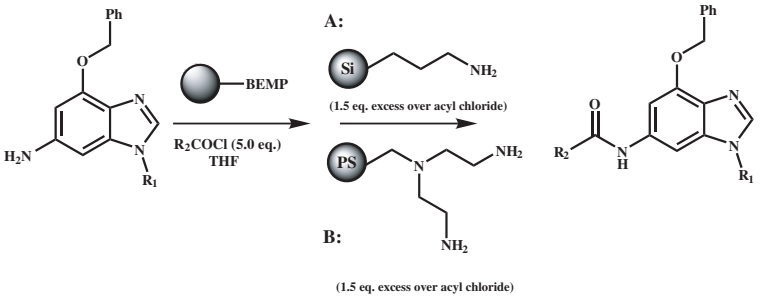
In the crude mixture, the amine is present as an acetate salt. Different procedures may be used to obtain the free amine after filtration of the silica:

1) *Acid-base extraction.*
   - Evaporation of the solvent under reduced pressure, addition of 20% $NaOH_{(aq)}$, followed by extraction with ether (3×) and evaporation.

2) *Catch and Release with* **SiliaPrep™** *TsOH*
   - Conditioning the cartridge with the reaction solvent.
   - Application of the sample.
   - Washing off the cartridge with the reaction solvent to eliminate impurities.
   - Product (amine) elution with 2 M NH_3/MeOH.

3) *Neutralization with* ***Si*-CO_3**.
   - Add 3 equivalents of ***Si*-CO_3**,
   - Stir for 30 minutes.
   - Filter off the **SiliaBond®** and rinse with solvent.
   - Pure product is obtained after purification by column chromatography with silica gel.

Scavenging acid chloride with SiliaBond® Amine



SAMPLE PROCEDURE[1]

- Add 1.5 eq. of **SiliaBond® Amine** to the reaction mixture.
- Stir for 1 hour at room temperature.
- Filter off the **SiliaBond®** rinsing with solvent to give acyl chloride free solution.

RELATED PUBLICATIONS

1) J. Catal., 195 (2000) 412

Scavenging aldehyde/ketone with SiliaBond® Tosyl Hydrazine

SAMPLE PROCEDURE

- Add 2-4 eq. of **SiliaBond® Tosyl Hydrazine** to the aldehyde containing reaction mixture.
- Stir for 1 hour at room temperature.
- Filter off the **SiliaBond®** rinsing with solvent to give aldehyde free solution.

When scavenging ketones and hindered aldehydes, a catalytic amount (0.05 eq.) of acetic acid should be added.

Scavenging benzylamine with SiliaBond® Isocyanate

Silica supported isocyanate has superior reactivity in a broader range of solvents when compared to polymer based materials. **SiliaBond® Isocyanate** is also available in SPE format (**SiliaPrep™ Isocyanate**) for flow through scavenging.

SAMPLE PROCEDURE

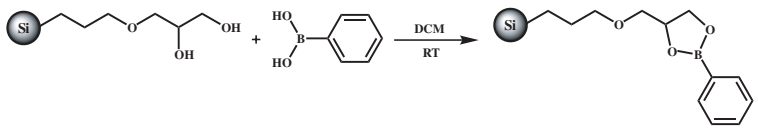
- Add 2-4 eq. of **SiliaBond® Isocyanate** to the crude reaction mixture
- Stir for 1 hour at room temperature
- **SiliaBond®** was filtered off and rinsed with solvent to give benzylamine free solution.

Scavenging benzylamine in different solvents

Scavenger	THF	DCM	MeCN
SiliaBond® Isocyanate	>98%	>98%	95%
PS-Isocyanate (1st source)	>98%	>98%	79%
PS-Isocyanate (2nd source)	>98%	>98%	88%

Conditions: 3 eq. relative to the amine, 1 hour at room temperature. Scavenging (%) determined by GC-MS.

Scavenging boronic acid with SiliaBond® Diol



SAMPLE PROCEDURE

Boronic acid scavenging

- Add 2-4 eq. of **SiliaBond® Diol** to the crude reaction mixture.
- Stir for 1h at room temperature.
- Filter off the **SiliaBond®** rinsing with DCM.
- Solvent evaporation gives the desired phenylboronic acid free solution.

Scavenging phenylboronic acid with **SiliaBond® Diol**

Equivalents	Time	Efficiency
2	1 h	75%
4	1 h	100%

Conditions: 3 eq. relative to the amine, 1 hour at room temperature. Scavenging (%) determined by GC-MS.

Scavenging HOBt with SiliaBond® Carbonate

SiliaBond® Carbonate was tested side by side with MP–Carbonate for the scavenging of HOBt after an amide coupling reaction.[1] As shown in the following table, the silica based scavenger has much faster kinetics which allows it to scavenge HOBt in only 5 minutes.

HOBt scavenging was also tested with **SiliaBond® Carbonate** (bulk) and **SiliaPrep™ Carbonate** on a 5 000 ppm HOBt solution.

Solid phase extraction using polystyrene and silica based supports

SPE media	Time (min.)	HOBt removed[a] (%)
MP–Carbonate	120	77
MP–Carbonate	240	100
Si-Carbonate	5	100

[a]HOBt scavenging determined by LC/MS and verified by ^{1}H NMR

Scavenging HOBt with **SiliaBond® Carbonate** (bulk) and **SiliaPrep™ Carbonate**

Initial HOBt Concentration	Si-CO_3 # eq.	Reaction time	Final HOBt concentration[a]	Reactivity
5000 ppm	3	5 min.	32 ppm	99.4%
5000 ppm	3	1 hour	32 ppm	99.4%
5000 ppm	3	SiliaPrep™	<5 ppm	100.0%
5000 ppm	4	5 min.	22 ppm	99.6%
5000 ppm	4	10 min.	22 ppm	99.6%
5000 ppm	4	1 hour	21 ppm	99.6%
5000 ppm	4	SiliaPrep™	<5 ppm	100.0%

[a]HOBt concentration determined by GC-MS. Solvent: DMF

Among the advantages of using **SiliaPrep™ Carbonate** *is the possibility of automation, the rapidity of execution, and the higher efficiency for the same number of equivalents.*

SAMPLE PROCEDURE

Scavenging HOBt[1] – In bulk

- Add 2-4 eq. of **Silia*Bond*® Carbonate** to the HOBt solution (in DMF).
- Stir for 1 hour at room temperature.
- Remove the **Silia*Bond*® Carbonate** by filtration rinsing with DMF.
- Solvent evaporation gives the HOBt free solution.

Scavenging HOBt[1] – In SPE Cartridges

- **Silia*Prep*™ Carbonate** (2-4 eq. of *Si*-CO_3 in respect to HOBt) was conditioned with DMF.
- Onto the **Silia*Prep*™ Carbonate** was loaded the HOBt containing solution.
- Amide was eluted with DMF.
- Solvent evaporation gave the HOBt free amide.

RELATED PUBLICATION

1) Org. Lett., 5(24) 2003, 4721

Scavenging nucleophile with Silia*Bond*® Tosyl Chloride

SAMPLE PROCEDURE

Nucleophile scavenger

- Add 20% pyridine or 6 eq. TEA to the reaction mixture.
- Add 2-4 eq. of **Silia*Bond*® Tosyl Chloride** to the reaction mixture.
- Stir at room temperature for 1 hour.
- **Silia*Bond*®** was filtered off rinsing with solvent to give a nucleophile free solution.

Scavenging Pd complexes

WITH SiliaBond® TAAcOH

Scavenging Pd complexes with SiliaBond® TAAcOH

Reaction Time	$Pd(AcO)_2$	$Pd_2(C_3H_5)_2Cl_2$	$Pd(PPh_3)_4$	$Pd_2(dba)_3$
5 min.	40	–	390	480
60 min.	9.8	0.25	150	50
18 h	0.06	–	1.4	–

Starting concentration: 1000 ppm. Scavenging with 4 eq. of SiliaBond® TAAcOH *in THF at RT.*

SAMPLE PROCEDURE

- Add 2-4 eq. of SiliaBond® TAAcOH to the crude reaction mixture
- Stir for 1h at room temperature
- SiliaBond® was filtered off rinsing with THF to give a Pd complexe-free solution.

WITH SiliaBond® THIOL

Scavenging Pd complexes with SiliaBond® Thiol

Reaction Time	$Pd(AcO)_2$	$Pd_2(C_3H_5)_2Cl_2$	$Pd(PPh_3)_4$	$Pd_2(dba)_3$
5 min.	0.9	–	360	545
60 min.	0.07	0.04	320	20
18 h	0.05	–	150	–

Starting concentration: 1000 ppm. Scavenging with 4 eq. of SiliaBond® Thiol *in THF at RT.*

SAMPLE PROCEDURE

- Add 2-4 eq. of SiliaBond® Thiol to the crude reaction mixture.
- Stir for 1h at room temperature
- SiliaBond® was filtered off rinsing with THF to give a Pd complexe-free solution.

WITH SILIABOND® THIOUREA

Scavenging Pd complexes with SiliaBond® Thiourea

Reaction Time	$Pd(AcO)_2$	$Pd_2(C_3H_5)_2Cl_2$	$Pd(PPh_3)_4$	$Pd_2(dba)_3$
5 min.	1.4	–	320	475
60 min.	0.8	1.3	95	50
18 h	0.6	–	10	–

Starting concentration: 1000 ppm. Scavenging with 4 eq. of **SiliaBond® Thiourea** *in THF at RT.*

SAMPLE PROCEDURE

- Add 2-4 eq. of **SiliaBond® Thiourea** to the crude reaction mixture.
- Stir for 1h at room temperature
- **SiliaBond®** was filtered off rinsing with THF to give a Pd complexe-free solution.

WITH SILIABOND® TRIAMINE

Scavenging Pd complexes with SiliaBond® Triamine

Reaction Time	$Pd(AcO)_2$	$Pd_2(C_3H_5)_2Cl_2$	$Pd(PPh_3)_4$	$Pd_2(dba)_3$
5 min.	20	–	540	525
60 min.	1.4	1.3	370	83
18 h	0.3	–	220	–

Starting concentration: 1000 ppm. Scavenging with 4 eq. of **SiliaBond® Triamine** *in THF at RT.*

SAMPLE PROCEDURE

- Add 2-4 eq. of **SiliaBond® Triamine** to the crude reaction mixture.
- Stir for 1h at room temperature
- **SiliaBond®** was filtered off rinsing with THF to give a Pd complexe-free solution.

Solid support: linking alcohol to SiliaBond® Tosyl Chloride

SAMPLE PROCEDURE

Solid support

- For best results, use an equivalent or excess of alcohol (1.0-3.0 eq.). Residual alcohol can be recovered by filtration at the end of the reaction.
- Stir silica and alcohol for 5-10 hours at room temperature in any standard aprotic organic solvent containing 50% pyridine or other weak tertiary amine base.
- Filter, wash with neutral solvent, and dry under vacuum.

Cleavage of the tosylate

- Mix the silica bound tosylate (obtained as described above) with 2.0 eq. of amine and 6.0 eq. of DIPEA or TEA in MeCN.
- Stir for 12 hours at 70°C. (For volatile secondary amines, use 4.0 eq. for 12 hours at room temperature.)

Acylsulfonamide synthesis

USING SILIABOND® CARBODIIMIDE

Acylsulfonamide synthesis with SiliaBond® Carbodiimide

Acid	Sulfonamide	% Yield (% Purity)
Benzoic acid	Benzenesulfonamide	95.5 (71.3)
	Methanesulfonamide	78.8 (53.1)

Purity determined by GC-FID.

SAMPLE PROCEDURE

Acylsulfonamide synthesis - using **SiliaBond® Carbodiimide**

- The acid (3.0 eq.), DMAP (3.0 eq.), sulfonamide (1.0 eq.) and **SiliaBond® Carbodiimide** (2g, 4.5 eq.) in 10mL of (4:1) DCM/DMF mixture were added to a dry reaction vessel.
- Stir 24 hours at room temperature.
- Unreacted sulfonamide and DMAP were scavenged with **SiliaBond® Tosic Acid** (12.0 eq.).
- Total volume of the mixture was adjusted to keep a silica/solvent ratio of 1g/5mL.
- Scavengers were allowed to react for 2 hours at room temperature.
- **SiliaBond®** were filtered off and rinsed with DCM (3 × 20mL).
- Solvent evaporation gave the desired crude mixture.
- The corresponding acylsulfonamides were analyzed by GC-MS. (Yield corresponds to the mass of isolated product. Purity was determined by GC-FID.)

USING SILIAPREP™ DICHLOROTRIAZINE

Acylsulfonamide synthesis using SiliaPrep™ Dichlorotriazine

Acid	Sulfonamide	% Yield (% Purity)
Benzoic acid	Benzenesulfonamide	98.0 (90.0)
	Methanesulfonamide	71.4 (82.0)

Purity determined by GC-FID.

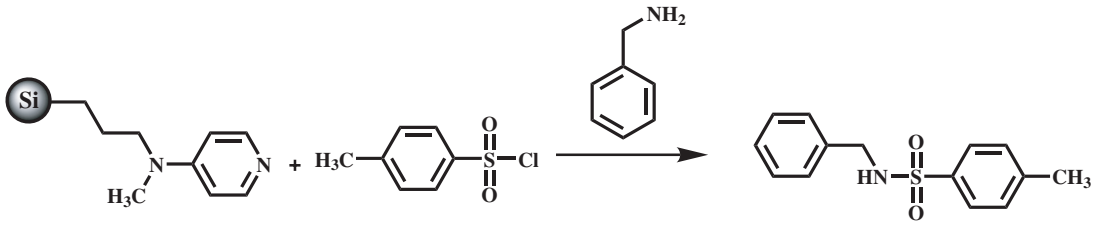
SAMPLE PROCEDURE

Acylsulfonamide synthesis – Using SiliaPrep™ Dichlorotriazine

Same application note as describe for Weinreb amide synthesis (see p.86) with the exception that the sulfonamide mixture is incubated overnight.

Sulfonamide synthesis

Sulfonamide synthesis using *"Catch and Release"* with **Silia*Bond*® DMAP**



SAMPLE PROCEDURE

Sulfonamide synthesis using *"Catch and Release"* with Silia*Bond*® DMAP

Catch of Tosyl Chloride

- To a suspension of **Silia*Bond*® DMAP** (1.0 eq) in anhydrous DCM is added tosyl chloride (2 eq.).
- The reaction mixture is stirred for one hour at room temperature, filtered, washed with anhydrous DCM and immediately carried through the second step without drying.

Release of Benzylsulfonamide

- To the mixture obtained in the first step was added benzylamine (0.65 eq. considering a 60-70% yield for the *"catch"* step) in anhydrous DCM.
- The reaction is stirred for 16 hours at room temperature.
- After completion, the reaction is filtered rinsing with DCM.
- The benzylsulfonamide was obtained in 75-80% yield by concentration of the filtrate under vacuum.
  The sulfonamide presented high purity (98% evaluated by GC-FID in THF inhibitor free).

Suzuki coupling

Suzuki coupling results with **SiliaBond® Diphenylphosphine-Pd**

Halide	Boronic Acid	% Yield (% Purity)
Iodoaniline	2-Thiopheneboronic acid	57 (57)
4-Bromopyridine	Phenylboronic acid	98 (100)
4-Bromopyridine	4-Formyl boronic acid	88 (99)

Purity determined by ^{1}H NMR

SAMPLE PROCEDURE

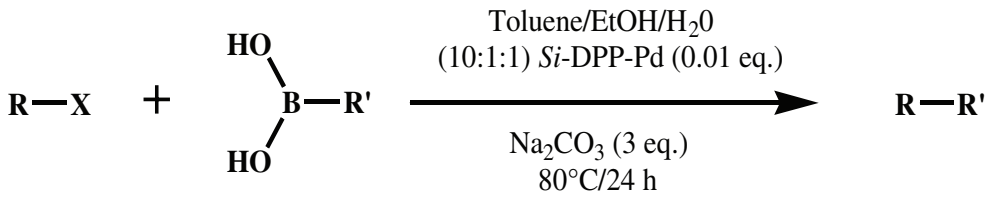
Palladium immobilization

- Mix **SiliaBond® Diphenylphosphine** (1.0 eq.) with $Pd(PPh_3)_4$ (1.0 eq.) in benzene.
- Stir for 24 hours at reflux under inert atmosphere.
- Isolate product by filtration.
- Wash with ether.
- Dry under vacuum.
  (Palladium loading was determined to be 0.76 mmol/g)

Suzuki coupling

- Mix sodium carbonate (3.0 eq.), halide (1.0 eq.), and boronic acid (1.1 eq.) in 5mL of Toluene/EtOH/H_2O (10:1:1)
- Add the palladium immobilized on silica (0.01 eq.).
- Stir at 80°C for 24 hours under inert atmosphere.
- The crude organic phase is then purified either on **SiliaPrep™ Tosic Acid** or by flash chromatography. (Products were identified by ^{13}C and ^{1}H NMR. Purity was determined by ^{1}H NMR.)

Suzuki coupling with SiliaBond® Diphenylphosphine-Pd



Weinreb amide synthesis

USING SILIABOND® CARBODIIMIDE

Weinreb amides synthesis using **SiliaBond® Carbodiimide**

Acid	Amine	% Yield (% Purity)
Benzoic acid	*N,O*–Dimethylhydroxylamine hydrochloride	98.8 (95.5)
trans-Cinnamic acid		87.3 (94.7)
2-Nitrobenzoic acid		99.5 (93.2)

Purity determined by GC-FID.

SAMPLE PROCEDURE

***Weinreb amide synthesis – using* SiliaBond® Carbodiimide**

- The acid (3.0 eq.), DMAP (0.3 eq.), pyridine (3.5 eq.), *N,O,*-dimethylhydroxylamine hydrochloride (1.0 eq.) and **SiliaBond® Carbodiimide** (2g, 4.5 eq.) in DCM (10mL) were added to a dry reaction vessel.
- The mixture was stirred overnight at room temperature. The reaction was monitored by TLC (EtOAc/Hexanes, 1:1).
- Excess acid was scavenged with **SiliaBond® Amine** (4.0 eq.) and excess amine, DMAP and, pyridine were scavenged with **SiliaBond® Tosic Acid** (14.0 eq.). Total volume of the mixture was adjusted to keep a silica/solvent ratio of 1g/5mL.
- Scavengers were allowed to react for 1 hour at room temperature.
- **SiliaBond®** were filtered off rinsing with DCM (3 × 20 mL).
- Solvent evaporation gave desired crude mixture.
- The corresponding Weinreb amides were analyzed by GC-MS. (Yield corresponds to the mass of isolated product. Purity was determined by GC-FID.)

USING SILIABOND® DICHLOROTRIAZINE

Weinreb amides synthesis using **SiliaBond® Dichlorotriazine**

Acid	Amine	% Yield (% Purity)
Benzoic acid	*N,O*–Dimethylhydroxylamine hydrochloride	96.4 (94.0)
trans-Cinnamic acid		81.9 (70.0)
2-Nitrobenzoic acid		92.4 (79.0)

Purity determined by GC-FID.

SAMPLE PROCEDURE

***Weinreb amide synthesis – using* SiliaBond® Dichlorotriazine**

- To **SiliaBond® Dichlorotriazine** (1.0g, 2.0 eq.) was added 10mL of anhydrous DCM along with *N*-methylmorpholine (NMM, 3.0 eq.), acid (1.5 eq.) and the amine (1.0 eq.).
- The solution was stirred at room temperature for 1 hour or until the amine disappears (TLC).
- **SiliaBond® TsOH** (5.0 eq.) and **SiliaBond® Amine** (2.0 eq.) were added to this mixture to scavenge the excess amines (starting amine and NMM), and excess acid. Total volume of the mixture was adjusted to have a maximum silica/solvent ratio of 1g/5mL.
- Scavengers were allowed to react for 1 hour at room temperature.
- **SiliaBond®** were filtered off rinsing with anhydrous DCM (3 × 10mL).
- Solvent evaporation gave desired crude mixture.
- The corresponding Weinreb amides were analyzed by NMR or GC-MS. (Yield corresponds to the mass of isolated product. Purity was determined by GC-FID. THF (anhydrous and inhibitor free) may be used instead of DCM.)

USING SiliaPrep™ DICHLOROTRIAZINE

SAMPLE PROCEDURE

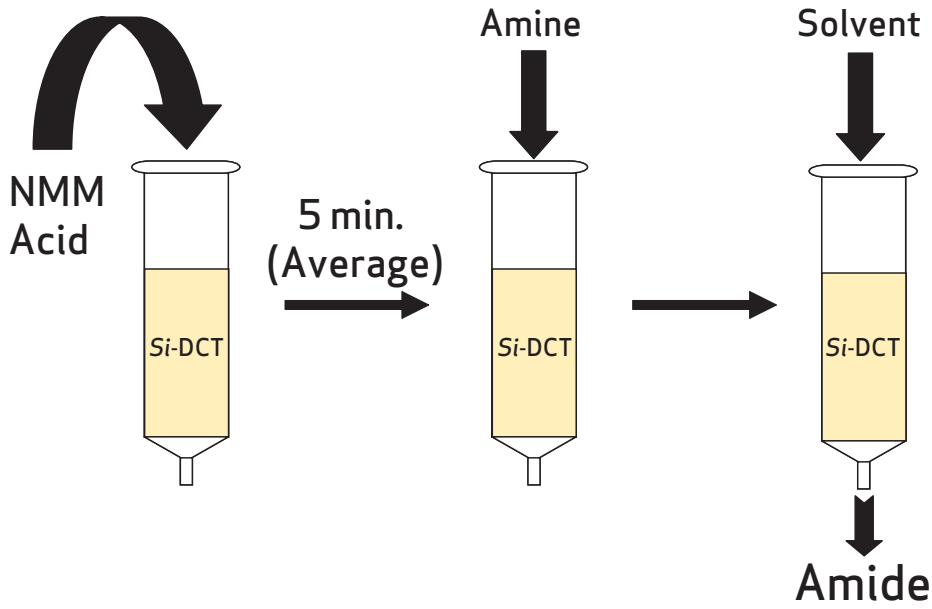
***Weinreb amide synthesis – using* SiliaPrep™ Dichlorotriazine**

- **SiliaPrep™ Dichlorotriazine** (1.0g, 12mL, 2.0 eq.) was placed on a vacuum manifold equipped with individual valves (closed).
- NMM (3.0 eq.) and the acid (1.5 eq.) were mixed together in anhydrous DCM (0.8mL). The resulting solution was loaded onto the cartridge and left to react for 1-5 minutes (or longer for less reactive acids).
- The valve was opened to drain the solution rinsing with anhydrous DCM (2x10mL).
- Vacuum was applied and the cartridge was allowed to dry.
- The valve was closed and the amine (1.0 eq., in 0.8mL of anhydrous DCM) was dripped onto the SPE cartridge.
- Incubated for a time ranging from a few minutes to overnight.
- The cartridge was drained and rinsed with anhydrous DCM (2 × 10mL) to collect the amide.
- Solvent was removed under vacuum to give the crude desired amide.
- Amides were characterized as described in previous example.
  (THF (anhydrous and inhibitor free) may be used instead of DCM.)

Notes:

1) If using Fmoc protected amino acid (or any compound bearing base sensitive groups), pass the NMM in solution first, wash, and then load the acid.
2) If using Boc protected amino acid (or any compound bearing acid sensitive groups), mix all reagents together in a minimum of DCM. Apply the solution to the cartridge and let incubate for a few minutes before washing with DCM.

The following scheme shows how **Silia*Prep*™ Dichlorotriazine** is used for amide synthesis.



Visit our website for new metal scavengers and application notes.

SiliaPrep™

2008 catalog

SiliCycle® *Ultrapure* Silia*Prep*™

Solid Phase Extraction (SPE) improves sample purity, quantification, and HPLC column lifetime. You can optimize your SPE protocols by using SiliCycle®'s Silia*Prep*™.

Silia*Prep*™ are available in different formats, SPE cartridges and 96 well plates, with different sorbents, our Silia*Flash*® and Silia*Bond*®, and bed weight up to 20 grams. Our 96 well plates are used in high throughput combinatorial chemistry, metabolite analysis, drug screening, and clinical chemistry applications.

By using our Silia*Prep*™ products you will generate higher purity samples and reduce the number of false positives in your screening, resulting in higher quality data. All our Silia*Prep*™ are packed with sorbents based on our fines-free Silia*Flash*® silica gel that is the highest purity silica gel on the market. When you use SiliCycle®'s Silia*Prep*™ we guarantee the following:

- High quality and wide variety of Silia*Bond*® sorbents available
- Very good separation (very tight particle size distribution and no fines)
- High recoveries and yields
- Less time and solvent spent conditioning the sorbent
- No silica contamination of your final product
- Reproducible flow rate from lot-to-lot cartridges and plates
- Excellent packing and storage qualities

SiliCycle® *UltraPure* Silia*Prep*™ 96 Well Plates

You can increase your recovery from HPLC purification by doing a first pass purification with our Silia*Prep*™ 96 well plates. These are the characteristics of our UltraPure Silia*Prep*™ 96 well plates:

- Small-scale versions of our syringe barrel columns
- Packed with the same high purity fines-free sorbent
- Reproducible flow rate
- Industry standard 8 × 12 array
- Excellent well-to-well packing and sorbent mass reproducibility
- Superior storage qualities
- Can be used on most instruments and will map to all major brands of collection plate.

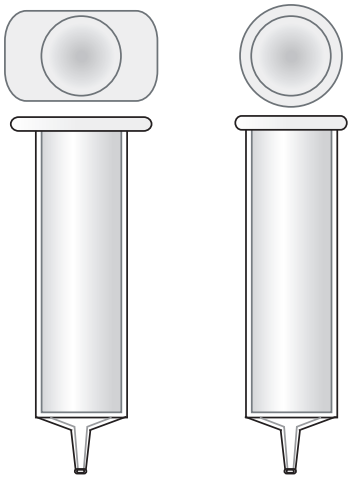
SiliCycle® *UltraPure* Silia*Prep*™ SPE cartridges

SiliCycle®'s goal is to offer to our customers products that will answer specific purification needs, and with that in mind, SiliCycle®'s UltraPure Silia*Prep*™ are available in different formats with specific characteristics. Our Silia*Prep*™ line offers the standard cartridges, but you can also customize your own SPE cartridge. In fact, with SiliCycle®, you can choose every component of the cartridge:

- Rim
- Volume
- Sorbent weight

SiliaPrep™ CARTRIDGES RIM

The rim of the cartridges will often dictate its compatibility with automated liquid handlers and other synthesis equipments. SiliCycle®'s standard SiliaPrep™ cartridge is flanged but our custom packing service can provide you with sorbent packed into any format at a competitive price.

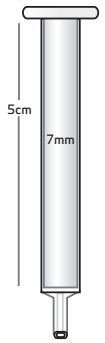
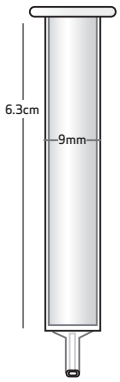
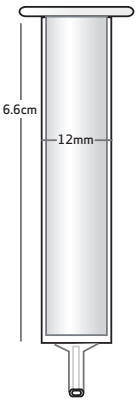
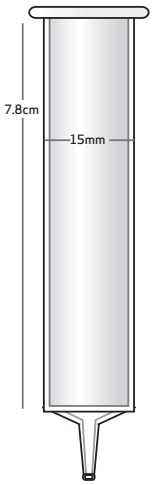
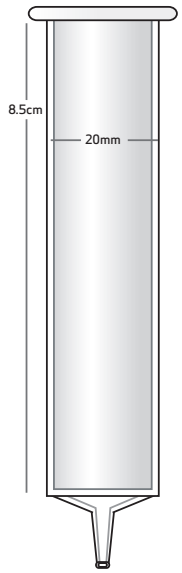


	Flanged	**Flangeless**
Product number starting with:	(SPE-...)	(SPB-...)

SiliaPrep™ CARTRIDGES SIZES AND SORBENT MASSES

The size of a SiliaPrep™ cartridge is normally listed by volume, but this fact can be confusing and misleading. The only important measurements are the **length, column diameter, and the mass of the sorbent**. Most SPE cartridges manufacturers use the same injection molds for making disposable syringes but list the volume differently. In the scheme shown on the next page, the volume (in parentheses) represents the equivalent volume used by some manufacturers. It also shows schematics of our SiliaPrep™ cartridge including the length, the diameter and the standard mass sorbent that we can provide for each cartridge size.

SiliCycle® *UltraPure* SiliaPrep™ available cartridge sizes*

1mL	3mL	6mL (8mL)	12mL (15mL)	25mL (20mL)
				
Available Standard Cartridges Sorbent Weight				
1mL	3mL	6mL (8mL)	12mL (15mL)	25mL (20mL)
• 50mg • 100mg	• 200mg • 500mg	• 500mg • 1g • 2g	• 2g	• 5g

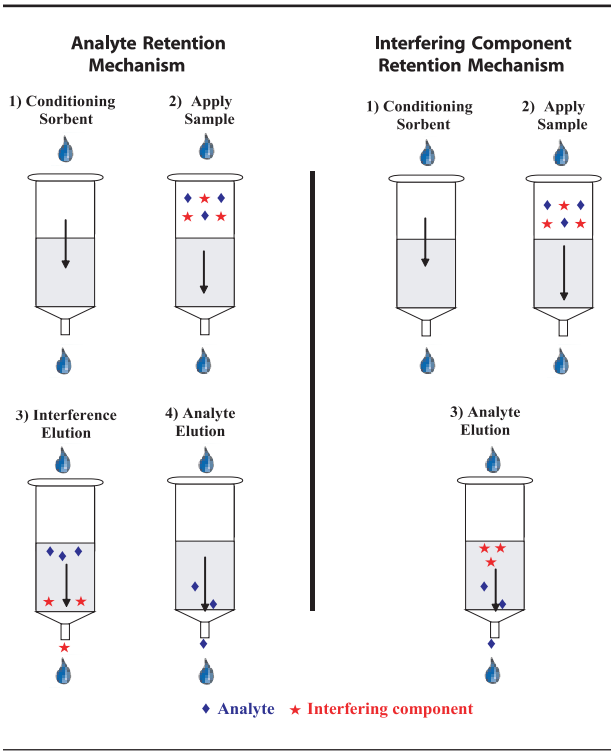
* *Please refer to* SiliaSep™ *OT on page 18 and 19 for > 25 mL SPEs.*

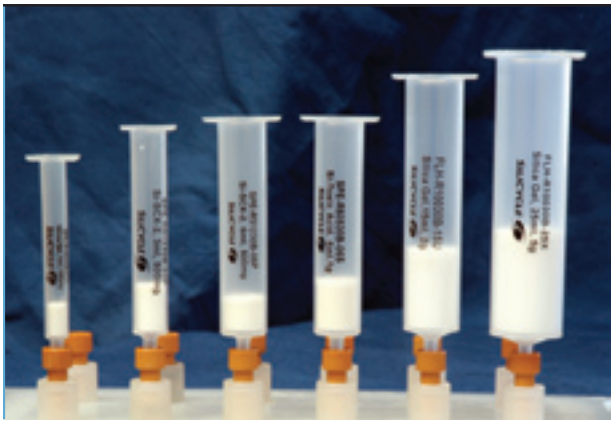
SiliaBond® and SiliaFlash® sorbents for SiliaPrep™

SPE methods were first used for the isolation of organic compounds from biological fluids such as blood and urine for toxicology and clinical work. It has also become widely used for the concentration and purification of trace organic pollutants in environmental research and analytes in agrochemical, food, forensic, and pharmaceutical analyses. Organic and medicinal chemists are now using this technique routinely to purify libraries of compounds.

Many applications use different mechanisms and sorbents. Biological and environmental applications generally process aqueous samples and therefore utilize mostly non-polar, mixed-mode non-polar/ion exchange sorbents. Chemists typically purify non-aqueous samples and utilize polar and mixed-phase polar/weak ion exchange sorbents. Silica bound scavengers are now being used in SPE formats to chemoselectively purify reactions due to their fast kinetics and solvent independence.

Typically, a solid phase extraction can be done following two different extraction pathways; using a sorbent that will retain the analyte of interest and let the interfering component pass through the cartridge or using a sorbent that can achieve the opposite (retains the interfering component and let the analyte pass through).





Visit our website for new metal scavengers video and application notes.

Sorbent selection

As previously shown, solid phase extraction can be done using different types of interactions. We can categorize the different kinds of functionalized silica gels by the molecular interactions taking place between compounds. There are three principal types of interactions:

- Polar interactions
  (normal phase silica gel)
- Non-polar interactions
  (reversed-phase silica gel)
- Ionic interactions (ion exchange silica gel)

SiliCycle® has developed many functionalized silica gels (**Silia***Bond*® products) of which many can be used in **Silia***Prep*™ cartridges or 96 well plates. The "standard sorbents" for solid phase extraction are presented in the following table. Most of our **Silia***Bond*® phases can be packed in cartridges. If the one you want is not listed, please ask us for a quote.

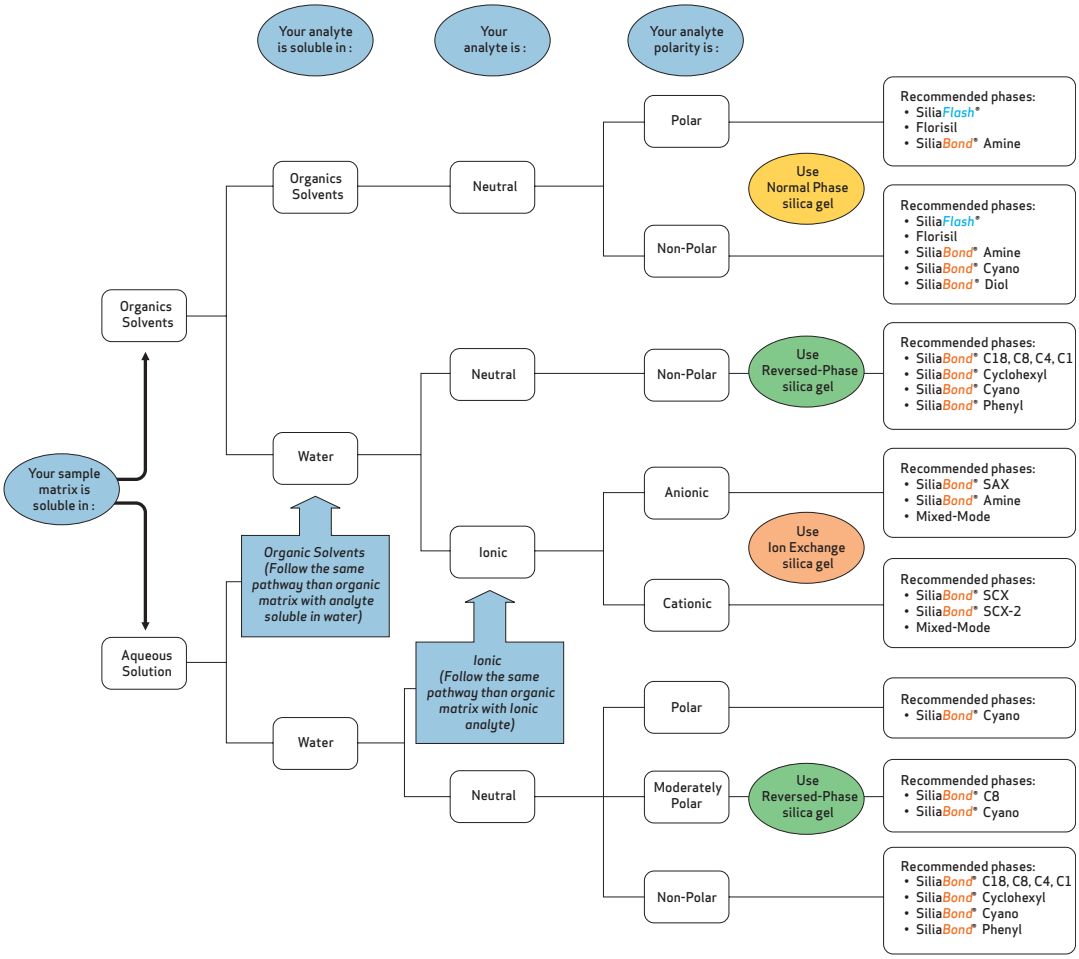
Phase	Extraction of	From
• Normal	Polar analytes	Non-polar organic solvent
• Reversed	Hydrophobic or polar analytes	Aqueous solution
• Ion Exchange	Charged analytes	Aqueous or non-polar organic matrix

Sorbent bed weight selection

In solid phase extraction, there is a general rule regarding the correlation of the sorbent bed weight and the mass of the sample to be extracted: generally, the mass of the sample or the analyte to be purified should **not represent more than 5% of the sorbent bed weight**.

The flow chart shown on the next page is designed to serve as a guide in sorbent selection by considering the nature of the solvent and the compound to be isolated. For your convenience, we offer a broad range of mixed-mode phases that allows the isolation of compounds using selective modes of interactions.

Sorbent selection guide



SPE method development

Before performing a solid phase extraction, make sure you properly select:

The sorbent:	Use the solvent of the sample and the chemical properties of your analyte to select your sorbent.
The cartridge size:	For purification, you need a column large enough to hold the reaction mixture and sufficient bed mass to bind either your product or the impurities.
The solvent:	Choose a solvent in which the analyte is very soluble.

The solid phase methodology will vary according to the sorbent used (normal, reversed, ion exchange). Here, we propose “generic methods” for each phase. The usual procedure can be slightly different from what we present but it will always be based on the properties of the sample and the sorbent.

Standard protocols for SPE

Properties	Sorbent Phase type			
	Normal Phase	Reversed-Phase	Ion Exchange Phase	
			Anion Exchange	Cation Exchange
Typical sorbents	• SiliaPrep™ • Florisil • SiliaPrep™ Amine • SiliaPrep™ Cyano • SiliaPrep™ Diol	• SiliaPrep™ C18 • SiliaPrep™ C8 • SiliaPrep™ C4 • SiliaPrep™ Cyclohexyl • SiliaPrep™ Cyano • SiliaPrep™ Phenyl	• SiliaPrep™ WAX • SiliaPrep™ SAX	• SiliaPrep™ WCX • SiliaPrep™ SCX • SiliaPrep™ SCX-2
Sorbent polarity	**High**	**Low**	**High**	
Matrix sample properties	• Organic solvents	• Organic solvents • Aqueous (buffer)	• Organic solvents • Aqueous (buffer)	
Analyte properties	• Slightly to moderately polar • Not charged	• Non-polar • Not charged	**Acid**	**Base**
Retained compounds	Polar compounds	Non-polar compounds	Ionized compounds	
Step 1: Conditioning	Sample matrix solvent or methanol	Water-miscible organic solvents *i.e.: methanol*	Water-miscible organic solvents or aqueous solution *i.e.: methanol*	
Step 2: Sample loading	Dissolve analyte in low polarity solvents *i.e.: hexanes, toluene, dichloromethane*	Dissolve analyte in high polarity solvents *i.e.: methanol/water acetonitrile/water*	Dissolve analyte in high polarity solvents *i.e.: water, buffered solutions*	
Step 3: Washing	Washing with non-polar solvents *(may contain small quantities of polar solvents ,5%)*	Washing with mixture of aqueous solution or buffer and polar solvent *i.e.: water/methanol*	Washing with mixture of aqueous solution containing salts *(may contain organic solvent)* *i.e.: water/methanol*	
Step 4: Elution	Elution with mixture of non-polar and polar solvents *(5-50% polar solvent)* *i.e.: hexanes with 10 % polar solvent*	Elution with non-polar or polar organic solvents *(may contain water or buffer)* *i.e.: methanol, water, acetonitrile*	Elution with polar solvents (may contain acid or base) *i.e.: water, buffered solutions*	

SiliCycle® standard sorbents for solid phase extraction:
SiliCycle® can provide standard sorbents for your solid phase extraction. The table below presents the sorbents commonly used in **Silia*Prep*™**. Other phases are available upon request.

Sorbent	End-capped	Sorbent code	Applications
POLAR PHASES			
Silia*Prep*™	–	100	Removal of baseline noise from organic samples
Florisil	–	AUT-0014	Clean up non-polar analytes containing polar interferences from non-polar solvent
Silia*Prep*™ Amine	Yes	520	Isolation of peptides, drugs and metabolites from physiological fluids, weak anion exchange sorbent
Silia*Prep*™ Cyano	Yes	380	Isolation of drugs and metabolites from physiological fluids, moderate polarity sorbent
Silia*Prep*™ Diol	No	350	Isolation of drugs and metabolites from physiological fluids, moderate polarity sorbent
NON-POLAR PHASES			
Silia*Prep*™ C8	Yes	310	Isolation of drugs and metabolites from physiological fluids, non-polar sorbent
Silia*Prep*™ C8 *nec*	No	311	Isolation of drugs and metabolites from physiological fluids, and pesticides from water, non-polar water wettable sorbent
Silia*Prep*™ C18 (23%) *nec*	No	301	See **Silia*Prep*™ C8**
Silia*Prep*™ C18 (17%)	Yes	302	See **Silia*Prep*™ C8 *nec***
Silia*Prep*™ Cyclohexyl	Yes	615	Isolation of drugs and metabolites from physiological fluids, non-polar sorbent
Silia*Prep*™ Phenyl	Yes	340	Isolation of drugs and metabolites from physiological fluids, non-polar and electron rich sorbent
ION EXCHANGE PHASES			
Silia*Prep*™ WCX	Yes	700	Isolation of very strong basic drugs (i.e.: quaternary amines)
Silia*Prep*™ SAX	No	665	*"Catch and Release"* purification of acidic molecules from organic solvents

(CONTINUED)

Sorbent	End-capped	Sorbent code	Applications
SiliaPrep™ SCX	Yes	605	*"Catch and Release"* purification of basic molecules from organic solvents
SiliaPrep™ SCX-2	Yes	512	*"Catch and Release"* purification of basic molecules from organic solvents
FLUORINATED PHASES			
SiliaPrep™ Fluorochrom	No	637	Separation of fluorinated molecules and non-fluorinated ones
SiliaPrep™ Tridecafluoro	Yes	635	
MIXED-MODE PHASES			
SiliaPrep™ C8/SAX	Yes	–	Isolation of acidic drugs and metabolites from physiological fluids, anionic and non-polar sorbent
SiliaPrep™ C8/SCX	Yes	–	Isolation of basic drugs and metabolites from physiological fluids, cationic and non-polar sorbent
SiliaPrep™ C8/SCX-2	Yes	–	Isolation of basic drugs and metabolites from physiological fluids, cationic and non-polar sorbent
SiliaPrep™ C18/SAX	Yes	–	See SiliaPrep™ C8/SAX
SiliaPrep™ C18/SCX	Yes	–	See SiliaPrep™ C8/SCX
SiliaPrep™ C18/SCX-2	Yes	–	See SiliaPrep™ C8/SCX-2
SiliaPrep™ SCX-2/SAX	Yes	–	Separation of acidic and basic molecules from non-ionizable molecules, anionic and cationic sorbent
SiliaPrep™ C8/SCX-2/SAX	Yes	–	Tri-mode isolation and fractionation of drugs and metabolites from physiological fluids
SPECIALITY PHASES			
SiliaPrep™ Carbonate	Yes	660	Applications available in the corresponding section of SiliaBond® silica gels
SiliaPrep™ Carbodiimide	Yes	705	
SiliaPrep™ Dichlorotriazine	Yes	522	
SiliaPrep™ Isocyanate	Yes	500	
SiliaPrep™ Thiol	Yes	510	

SiliCycle® *UltraPure* SiliaPrep™ SPE Cartridges

Volume	1mL		3mL	
Sorbent Mass	50mg	100mg	200mg	500mg
Quantity per box	100	100	50	50
POLAR PHASES				
SiliaPrep™	SPE-R10030B-01B	SPE-R10030B-01C	SPE-R10030B-03G	SPE-R10030B-03P
Florisil	SPE-AUT-0014-01B	SPE-AUT-0014-01C	SPE-AUT-0014-03G	SPE-AUT-0014-03P
SiliaPrep™ Amine	SPE-R52030B-01B	SPE-R52030B-01C	SPE-R52030B-03G	SPE-R52030B-03P
SiliaPrep™ Cyano	SPE-R38030B-01B	SPE-R38030B-01C	SPE-R38030B-03G	SPE-R38030B-03P
SiliaPrep™ Diol	SPE-R35030B-01B	SPE-R35030B-01C	SPE-R35030B-03G	SPE-R35030B-03P
NON-POLAR PHASES				
SiliaPrep™ C8	SPE-R31030B-01B	SPE-R31030B-01C	SPE-R31030B-03G	SPE-R31030B-03P
SiliaPrep™ C8 *nec*	SPE-R31130B-01B	SPE-R31130B-01C	SPE-R31130B-03G	SPE-R31130B-03P
SiliaPrep™ C18 *nec* (23%)	SPE-R30130B-01B	SPE-R30130B-01C	SPE-R30130B-03G	SPE-R30130B-03P
SiliaPrep™ C18 (17%)	SPE-R30230B-01B	SPE-R30230B-01C	SPE-R30230B-03G	SPE-R30230B-03P
SiliaPrep™ Cyclohexyl	SPE-R61530B-01B	SPE-R61530B-01C	SPE-R61530B-03G	SPE-R61530B-03P
SiliaPrep™ Phenyl	SPE-R34030B-01B	SPE-R34030B-01C	SPE-R34030B-03G	SPE-R34030B-03P
ION EXCHANGE PHASES				
SiliaPrep™ WCX	SPE-R70030B-01B	SPE-R70030B-01C	SPE-R70030B-03G	SPE-R70030B-03P
SiliaPrep™ SAX	SPE-R66530B-01B	SPE-R66530B-01C	SPE-R66530B-03G	SPE-R66530B-03P
SiliaPrep™ SCX	SPE-R60530B-01B	SPE-R60530B-01C	SPE-R60530B-03G	SPE-R60530B-03P
SiliaPrep™ SCX-2	SPE-R51230B-01B	SPE-R51230B-01C	SPE-R51230B-03G	SPE-R51230B-03P
FLUORINATED PHASES				
SiliaPrep™ Fluorochrom	SPE-R63730B-01B	SPE-R63730B-01C	SPE-R63730B-03G	SPE-R63730B-03P
SiliaPrep™ Tridecafluoro	SPE-R63530B-01B	SPE-R63530B-01C	SPE-R63530B-03G	SPE-R63530B-03P
SPECIALITY				
SiliaPrep™ Carbonate	SPE-R66030B-01B	SPE-R66030B-01C	SPE-R66030B-03G	SPE-R66030B-03P
SiliaPrep™ Isocyanate	SPE-R50030B-01B	SPE-R50030B-01C	SPE-R50030B-03G	SPE-R50030B-03P
SiliaPrep™ Thiol	SPE-R51030B-01B	SPE-R51030B-01C	SPE-R51030B-03G	SPE-R51030B-03P
COUPLING REAGENT				
SiliaPrep™ Carbodiimide	SPE-R70530B-01B	SPE-R70530B-01C	SPE-R70530B-03G	SPE-R70530B-03P
SiliaPrep™ Dichlorotriazine	SPE-R52230B-01B	SPE-R52230B-01C	SPE-R52230B-03G	SPE-R52230B-03P
MIXED-MODE				
SiliaPrep™ C8/SAX	SPM-R022830B-01B	SPM-R022830B-01C	SPM-R022830B-03G	SPM-R022830B-03P
SiliaPrep™ C8/SCX	SPM-R023830B-01B	SPM-R023830B-01C	SPM-R023830B-03G	SPM-R023830B-03P
SiliaPrep™ C8/SCX-2	SPM-R028030B-01B	SPM-R028030B-01C	SPM-R028030B-03G	SPM-R028030B-03P
SiliaPrep™ C18/SAX	SPM-R012830B-01B	SPM-R012830B-01C	SPM-R012830B-03G	SPM-R012830B-03P
SiliaPrep™ C18/SCX	SPM-R013830B-01B	SPM-R013830B-01C	SPM-R013830B-03G	SPM-R013830B-03P
SiliaPrep™ C18/SCX-2	SPM-R018030B-01B	SPM-R018030B-01C	SPM-R018030B-03G	SPM-R018030B-03P
SiliaPrep™ SCX-2/SAX	SPM-R802830B-01B	SPM-R802830B-01C	SPM-R802830B-03G	SPM-R802830B-03P
SiliaPrep™ C8/SCX-2/SAX	SPM-R02802830B-01B	SPM-R02802830B-01C	SPM-R02802830B-03G	SPM-R02802830B-03P

SiliCycle® *UltraPure* SiliaPrep™ SPE Cartridges (CONTINUED)

Volume	6mL			12mL	25mL*
Sorbent Mass	500mg	1g	2g	2g	5g
Quantity per box	50	50	50	20	20
POLAR PHASES					
SiliaPrep™	SPE-R10030B-06P	SPE-R10030B-06S	SPE-R10030B-06U	SPE-R10030B-15U	SPE-R10030B-25X
Florisil	SPE-AUT-0014-06P	SPE-AUT-0014-06S	SPE-AUT-0014-06U	SPE-AUT-0014-12U	SPE-AUT-0014-20X
SiliaPrep™ Amine	SPE-R52030B-06P	SPE-R52030B-06S	SPE-R52030B-06U	SPE-R52030B-12U	SPE-R52030B-20X
SiliaPrep™ Cyano	SPE-R38030B-06P	SPE-R38030B-06S	SPE-R38030B-06U	SPE-R38030B-12U	SPE-R38030B-20X
SiliaPrep™ Diol	SPE-R35030B-06P	SPE-R35030B-06S	SPE-R35030B-06U	SPE-R35030B-12U	SPE-R35030B-20X
NON-POLAR PHASES					
SiliaPrep™ C8	SPE-R31030B-06P	SPE-R31030B-06S	SPE-R31030B-06U	SPE-R31030B-12U	SPE-R31030B-20X
SiliaPrep™ C8 *nec*	SPE-R31130B-06P	SPE-R31130B-06S	SPE-R31130B-06U	SPE-R31130B-12U	SPE-R31130B-20X
SiliaPrep™ C18 *nec* (23%)	SPE-R30130B-06P	SPE-R30130B-06S	SPE-R30130B-06U	SPE-R30130B-12U	SPE-R30130B-20X
SiliaPrep™ C18 (17%)	SPE-R30230B-06P	SPE-R30230B-06S	SPE-R30230B-06U	SPE-R30230B-12U	SPE-R30230B-20X
SiliaPrep™ Cyclohexyl	SPE-R61530B-06P	SPE-R61530B-06S	SPE-R61530B-06U	SPE-R61530B-12U	SPE-R61530B-20X
SiliaPrep™ Phenyl	SPE-R34030B-06P	SPE-R34030B-06S	SPE-R34030B-06U	SPE-R34030B-12U	SPE-R34030B-20X
ION EXCHANGE PHASES					
SiliaPrep™ WCX	SPE-R70030B-06P	SPE-R70030B-06S	SPE-R70030B-06U	SPE-R70030B-12U	SPE-R70030B-20X
SiliaPrep™ SAX	SPE-R66530B-06P	SPE-R66530B-06S	SPE-R66530B-06U	SPE-R66530B-12U	SPE-R66530B-20X
SiliaPrep™ SCX	SPE-R60530B-06P	SPE-R60530B-06S	SPE-R60530B-06U	SPE-R60530B-12U	SPE-R60530B-20X
SiliaPrep™ SCX-2	SPE-R51230B-06P	SPE-R51230B-06S	SPE-R51230B-06U	SPE-R51230B-12U	SPE-R51230B-20X
FLUORINATED PHASES					
SiliaPrep™ Fluorochrom	SPE-R63730B-06P	SPE-R63730B-06S	SPE-R63730B-06U	SPE-R63730B-12U	SPE-R63730B-20X
SiliaPrep™ Tridecafluoro	SPE-R63530B-06P	SPE-R63530B-06S	SPE-R63530B-06U	SPE-R63530B-12U	SPE-R63530B-20X
SPECIALITY					
SiliaPrep™ Carbonate	SPE-R66030B-06P	SPE-R66030B-06S	SPE-R66030B-06U	SPE-R66030B-12U	SPE-R66030B-20X
SiliaPrep™ Isocyanate	SPE-R50030B-06P	SPE-R50030B-06S	SPE-R50030B-06U	SPE-R50030B-12U	SPE-R50030B-20X
SiliaPrep™ Thiol	SPE-R51030B-06P	SPE-R51030B-06S	SPE-R51030B-06U	SPE-R51030B-12U	SPE-R51030B-20X
COUPLING REAGENT					
SiliaPrep™ Carbodiimide	SPE-R70530B-06P	SPE-R70530B-06S	SPE-R70530B-06U	SPE-R70530B-12U	SPE-R70530B-20X
SiliaPrep™ Dichlorotriazine	SPE-R52230B-06P	SPE-R52230B-06S	SPE-R52230B-06U	SPE-R52230B-12U	SPE-R52230B-20X
MIXED-MODE					
SiliaPrep™ C8/SAX	SPM-R022830B-06P	SPM-R022830B-06S	SPM-R022830B-06U	SPM-R022830B-12U	SPM-R022830B-20X
SiliaPrep™ C8/SCX	SPM-R023830B-06P	SPM-R023830B-06S	SPM-R023830B-06U	SPM-R023830B-12U	SPM-R023830B-20X
SiliaPrep™ C8/SCX-2	SPM-R028030B-06P	SPM-R028030B-06S	SPM-R028030B-06U	SPM-R028030B-12U	SPM-R028030B-20X
SiliaPrep™ C18/SAX	SPM-R012830B-06P	SPM-R012830B-06S	SPM-R012830B-06U	SPM-R012830B-12U	SPM-R012830B-20X
SiliaPrep™ C18/SCX	SPM-R013830B-06P	SPM-R013830B-06S	SPM-R013830B-06U	SPM-R013830B-12U	SPM-R013830B-20X
SiliaPrep™ C18/SCX-2	SPM-R018030B-06P	SPM-R018030B-06S	SPM-R018030B-06U	SPM-R018030B-12U	SPM-R018030B-20X
SiliaPrep™ SCX-2/SAX	SPM-R802830B-06P	SPM-R802830B-06S	SPM-R802830B-06U	SPM-R802830B-12U	SPMR802830B-20X
SiliaPrep™ C8/SCX-2/SAX	SPM-R02802830B-06P	SPM-R02802830B-06S	SPM-R02802830B-06U	SPM-R02802830B-12U	SPM-R02802830B-20X

*Please refer to SiliaSep™ OT on page 18 and 19 for >25 mL SPEs.

SiliCycle® *UltraPure* SiliaPrep™ 96 Well Plates

Volume	2mL		
Sorbent Mass	50mg	100mg	200mg
Quantity per box	1	1	1
POLAR PHASES			
SiliaPrep™	96W-R10030B-B	96W-R10030B-C	96W-R10030B-G
SiliaPrep™ Amine	96W-R52030B-B	96W-R52030B-C	96W-R52030B-G
SiliaPrep™ Cyano	96W-R38030B-B	96W-R38030B-C	96W-R38030B-G
SiliaPrep™ Diol	96W-R35030B-B	96W-R35030B-C	96W-R35030B-G
NON-POLAR PHASES			
SiliaPrep™ C8	96W-R31030B-B	96W-R31030B-C	96W-R31030B-G
SiliaPrep™ C8 *nec*	96W-R31130B-B	96W-R31130B-C	96W-R31130B-G
SiliaPrep™ C18 *nec* (23%)	96W-R30130B-B	96W-R30130B-C	96W-R30130B-G
SiliaPrep™ C18 (17%)	96W-R30230B-B	96W-R30230B-C	96W-R30230B-G
SiliaPrep™ Phenyl	96W-R34030B-B	96W-R34030B-C	96W-R34030B-G
ION EXCHANGE PHASES			
SiliaPrep™ WCX	96W-R70030B-B	96W-R70030B-C	96W-R70030B-G
SiliaPrep™ SAX	96W-R66530B-B	96W-R66530B-C	96W-R66530B-G
SiliaPrep™ SCX	96W-R60530B-B	96W-R60530B-C	96W-R60530B-G
SiliaPrep™ SCX-2	96W-R51230B-B	96W-R51230B-C	96W-R51230B-G
MIXED-MODE			
SiliaPrep™ C8/SAX	96W-R022830B-B	96W-R022830B-C	96W-R022830B-G
SiliaPrep™ C8/SCX-2	96W-R028030B-B	96W-R028030B-C	96W-R028030B-G
SiliaPrep™ SCX-2/SAX	96W-R802830B-B	96W-R802830B-C	96W-R802830B-G
SiliaPrep™ C8/SCX-2/SAX	96W-R02802830B-B	96W-R02802830B-C	96W-R02802830B-G

SiliaPrep™ accessories

SiliaPrep™ ADAPTERS

AUT-0010 **SiliaPrep™** Adapter for 1, 3 and 6mL SPE (12/box)
AUT-0011 **SiliaPrep™** Adapter for 12, 20 and 60mL SPE (6/box)

SiliaPrep™ VACUUM MANIFOLD

AUT-0038 **SiliaPrep™** Vacuum Manifold (12 Positions)
AUT-0039 **SiliaPrep™** Vacuum Manifold (24 Positions)

EMPTY TUBES

SIM-0007-001	**Empty 1mL SPE tube with 2 frits** (100/box)
SIM-0008-003	**Empty 3mL SPE tube with 2 frits** (100/box)
SIM-0002-006	**Empty 6mL SPE tube with 2 frits** (100/box)
SIM-0003-012	**Empty 12mL SPE tube with 2 frits** (100/box)
SIM-0004-020	**Empty 25mL SPE tube with 2 frits** (100/box)
SIM-0006-060	**Empty 60mL SPE tube with 2 frits** (100/box)
SIM-0009-150	**Empty 150mL SPE tube with 2 frits** (20/box)

SiliaPrep™ VACUUM ADAPTERS

AUT-0043 24/40 **SiliaPrep™** Vacuum Adapter
AUT-0044 19/22 **SiliaPrep™** Vacuum Adapter
AUT-0045 14/20 **SiliaPrep™** Vacuum Adapter
AUT-0046 20-400 Vial – **SiliaPrep™** Vacuum Adapter without Vial Connector
AUT-0047 20-400 Vial – **SiliaPrep™** Vacuum Adapter with Vial Connector

IMPAQ®

2008 catalog

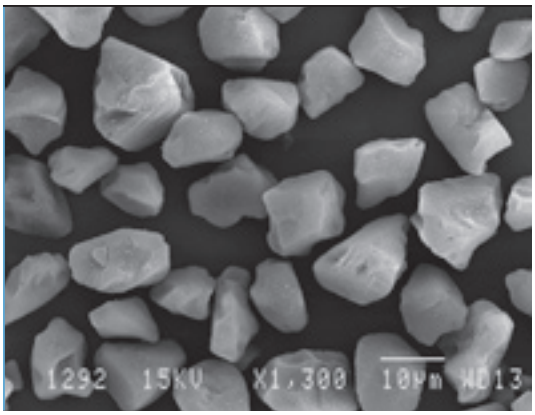
IMPAQ®

Spherical silica materials are the best performing supports for HPLC applications but they also are the most expensive. The IMPAQ® angular silica gel is the spherical alternative for preparative applications. IMPAQ® silica gels provide very efficient separations at much lower price. IMPAQ® is premium grade angular silica designed for High Performance Liquid Chromatography (HPLC) and other applications where consistent high purity and narrow distribution of pores and particle dimensions are required. It is the only non-spherical product performing successfully in large-scale HPLC applications. IMPAQ® is a fully porous gel in which the surface area, porosity and rigidity have been optimized for loading capacity and mechanical stability.

Characteristics:
This cost efficient material has an irregular particle shape with smooth edges and a very narrow particle size distribution. The high specific surface area (500 m^2/g for 60 Å pore size) allows the improvement of loading capacities for reversed-phases.

For irregular support, the average diameter is calculated by defining an equivalent average spherical diameter. From the following scanning electron microscopy (SEM) picture, it is easy to see the tight particle size distribution of this material, which is much better than standard silica and comes close to spherical silica. The graph presented on the next page shows two Microtrac analysis of a 10 µm IMPAQ® silica gel synthesized in 2002 and another one synthesized in 2004. It is easy to appreciate the batch-to-batch reproducibility of the synthetic method.

By carefully controlling the particle size of the silica precursor prior to the condensation we are able to greatly improve the robustness of the finished product. After the grinding and sieving steps are performed, there are extra steps to smooth out the edges to make the product pressure tolerant for HPLC applications. Fine particles generation is thus greatly reduced.



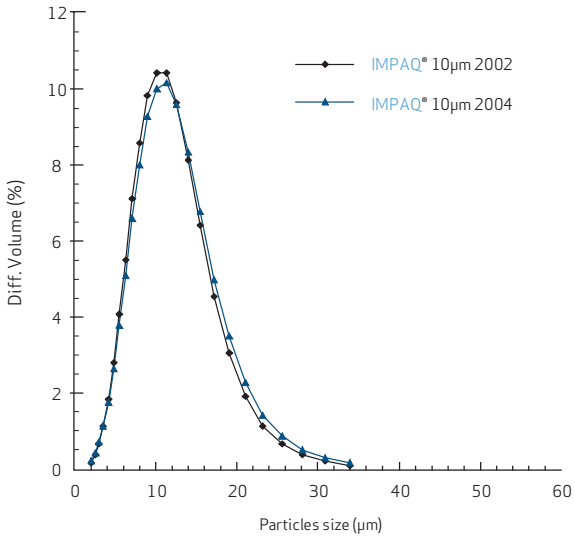
SEM picture of a 10 µm IMPAQ® silica gel

IMPAQ® angular silica supports available and their characteristics (FORMATS: 100g, 1kg*)

	Product Number	Particle size (µm)	Pore size (Å)	Specific surface area (m^2/g)
	B10005B	5	60	500
	B10007B	10	60	500
	B10009B	20	60	500
	B10025B	40	60	500
UltraPure IMPAQ®	B10005E	5	100	400
	B10007E	10	100	400
	B10009E	20	100	400
	B10025E	40	100	400
	B10005J	5	200	235
	B10007J	10	200	235
	B10009J	20	200	235
	B10025J	40	200	235

All SiliaBond® *functions presented on pages 32 to 60 are available on* IMPAQ®

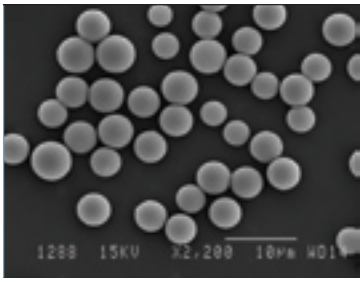
Particle size comparison of two batches

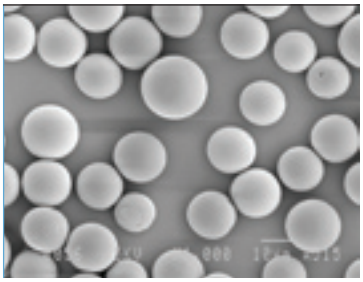


*All these products are available in different sizes from lab scale to multi-ton scale.

Tel.: 1 418.874.0054 Fax: 1 418.874.0355 Toll-free: 1 877.SILICYCLE (North America only) info@silicycle.com
www.SiliCycle.com

SILIASPHERE™





2008 catalog

SiliaSphere™ spherical silica gels

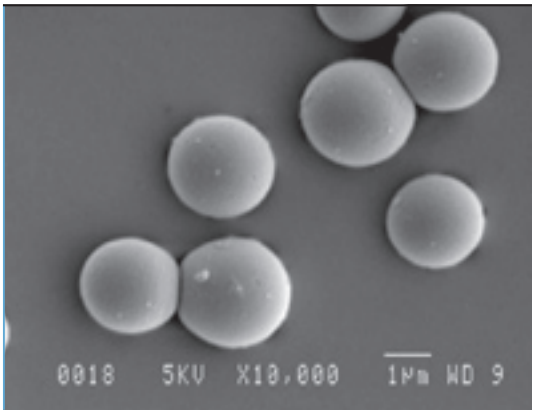
If you require the ultimate in spherical gels for High Performance Liquid Chromatography (HPLC), Supercritical Fluid Chromatography (SFC), Simulated Moving Bed (SMB), Dynamic Axial Compression (DAC), or even Ultra Performance Liquid Chromatography (UPLC), one of our **Silia***Sphere*™ spherical silica gels is your solution. There are two lines of spherical silica gel in this family: **Silia***Sphere*™, which are analytical spherical gels with monodisperse particle size and **Silia***Sphere*™ **PC** (Preparative Chromatography) which are preparative silica gels with determined particle size ranges. Both these lines are characterized by:

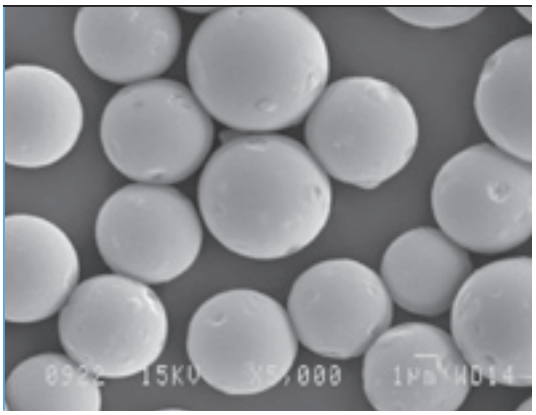
- **Low metal content:** to avoid specific interaction between acid sites and analytes
- **High mechanical stability**
- **Very high purity**
- **Reproducible loadings** (reversed-phase)

The SiliaSphere gels are manufactured from an organic form of silicon (alkoxydes). This ensures very low metal content as the starting material is purified by distillation. Deionized water is used to hydrolyze the silicon alkoxydes. Careful monitoring and control of the parameters that induce precipitation afford spherical silica gels with the desired characteristics.

The spherical family has specific applications in analytical and preparative separation. For preparative and process separation our **Silia***Sphere*™ **PC** are the most indicated. This high purity material presents an excellent mechanical stability that enables multiple column packing without significant loss. For analytical applications, the **Silia***Sphere*™ material, with its mono-disperse particle size gives the best packing for superior separations. If your chemistry requires a phase not available as standard material, please contact us. All spherical silica gels may be functionalized to suit your needs.

SiliaSphere™
MONODISPERSE SPHERICAL SILICA GELS





The **Silia***Sphere*™ family is characterized by a very low metal content and exceptionally stable media at low or high pH. The **Silia***Sphere*™ manufacturing process ensures quality and reproducibility in pore size, surface area and particles size and morphology. The high specific surface area enables a high loading capacity with a uniform and reproducible coverage. The **Silia***Sphere*™ materials are the basis for several bonded materials (C8, C18, CN and Phenyl) that are

used in HPLC column manufacturing. These columns are characterized by a long lifetime, high reliability and excellent performance. Many other custom bonded phases are available, see pages 32 to 60 for a complete listing of available functional groups.

The following table presents all the spherical silica supports available and their characteristics.

SiliaSphere™ spherical silica gels (FORMATS: 100g, 1kg*)

	Product number	Particles size (µm)	Pore size (Å)	Specific surface area (m^2/g)
SiliaSphere™	S10003B	3	60	450
	S10005B	5	60	450
	S10007B	10	60	450
	S10008B	15	60	450
	S10001G	1.8	120	340
	S10002G	2.2	120	340
	S10003G	3	120	300
	S10005G	5	120	300
	S10007G	10	120	300
	S10008G	15	120	300
	S10003M	3	300	100
	S10005M	5	300	100
	S10007M	10	300	100
	S10008M	15	300	100
	S10007T	10	1 000	50
	S10008T	15	1 000	50

SiliaSphere™ C18 Spherical silica gels (FORMATS: 100g, 1kg*)

	Product number	Particles size (µm)	Pore size (Å) (bare silica)
SiliaSphere™ C18	S33203B	3	60
	S33205B	5	60
	S33207B	10	60
	S33208B	15	60
	S33201G	1.8	120
	S33202G	2.2	120
	S33203G	3	120
	S33205G	5	120
	S33207G	10	120
	S33208G	15	120
	S33203M	3	300
	S33205M	5	300
	S33207M	10	300
	S33208M	15	300
	S33207T	10	1 000
	S33208T	15	1 000

SILIASPHERE™ PC
(FOR PREPARATIVE CHROMATOGRAPHY)

In preparative and process chromatography the cost is very important and the use of spherical particles with narrow particle size distribution is very expensive. It is possible in this case to use irregular or angular silica but the separation will not be optimal. SiliCycle® has added a very efficient material for preparative chromatography, the **SiliaSphere™ PC** spherical silica gels. These products have a very high chemical and mechanical stability. They also have the advantage to operate with very low backpressures. Furthermore, they are being offered at a very affordable price.

**All these products are available in different sizes from lab scale to multi-ton scale.*

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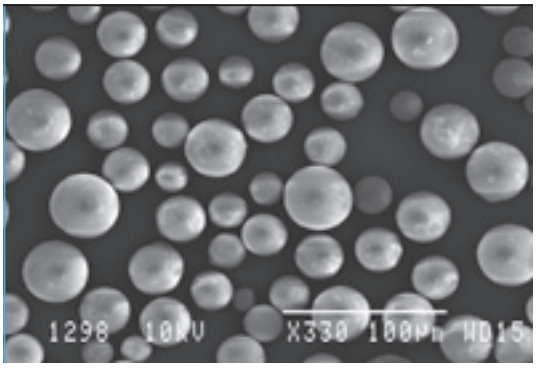
www.SiliCycle.com

Advantages of using the **Silia*Sphere*™ PC** materials over standard silica gels:

- Increased efficiency of the eluent's flow characteristics
- Improvement of the resolution between compounds of a sample
- Ease of packing
- Very high specific surface area
- High mechanical stability under higher pressures
- Affordable pricing



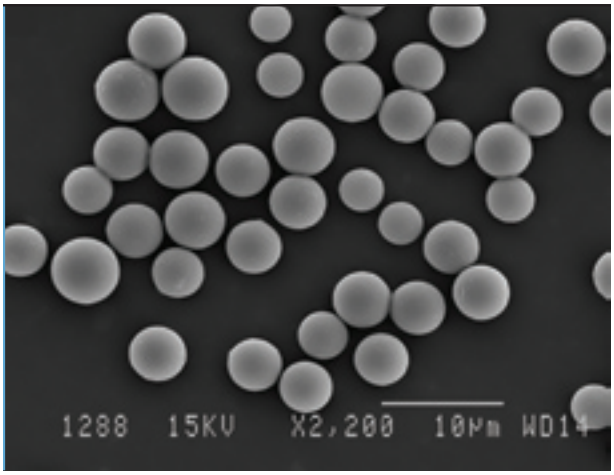
The following table presents the **Silia*Sphere*™ PC** supports available and their characteristics.

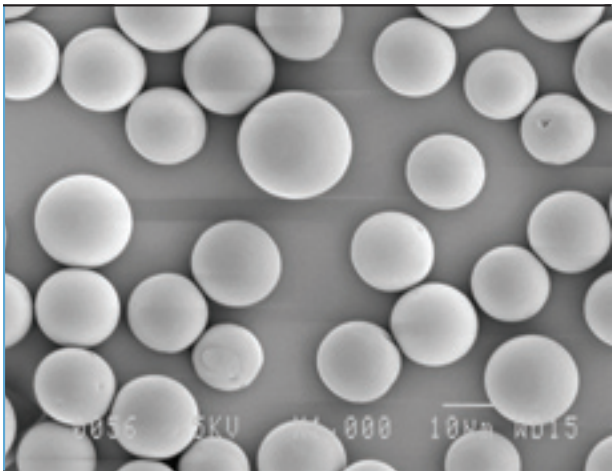
Silia*Sphere*™ PC spherical silica gels (FORMATS: 250g, 1kg*)

	Product number	Particle size (µm)	Pore size (Å)	Specific surface area (m^2/g)
Silia*Sphere*™ PC	S10020C	20-45	70	500
	S10030C	40-75	70	500
	S10040C	75-200	70	500
	S10020E	20-45	100	280
	S10030E	40-75	100	280
	S10040E	75-200	100	280
	S10020M	20-45	300	100
	S10030M	40-75	300	100
	S10040M	75-200	300	100
	S10020T	20-45	1 000	50
	S10030T	40-75	1 000	50
	S10040T	75-200	1 000	50

SiliaSphere™ PC C18 spherical silica gels (FORMATS: 250g, 1kg*)

	Product number	Particle size (µm)	Pore diameter (Å)
SiliaSphere™ PC C18	S33220C	20-45	70
	S33230C	40-75	70
	S33240C	75-200	70
	S33220E	20-45	100
	S33230E	40-75	100
	S33240E	75-200	100
	S33220M	20-45	300
	S33230M	40-75	300
	S33240M	75-200	300
	S33220T	20-45	1 000
	S33230T	40-75	1 000
	S33240T	75-200	1 000





All these products are available in different sizes from lab scale to multi-ton scale.

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Quality control

The objective of the department of Quality Control is to provide customers with default-free products to insure that they are 100% satisfied. In light of this, we have determined the critical points that need to be addressed for each product line. These points are based on clients and salespersons recommendations as well as from the scientific knowledge of our employees.

Each product family has its own quality control procedures and every procedure is rigorously followed. Finally, the results of the QC tests are validated and confirmed by the QC Specialists before signing off for shipping. The certificate of analysis is sent along with the product and the complete procedures for each product line are available upon request.

Terms and conditions

SHIPPING POLICY

SiliCycle® Inc. uses next day delivery for its **Silia*Bond*®** silica gels. For large shipment on skids, we use ground delivery and for all other products, we use 2 day delivery (or equivalent), although standard overnight delivery can also be arranged. Freight charges are prepaid and added to the invoice unless special instructions are requested. These conditions apply to North American shipments. International deliveries will vary according to the orders.

APPLICATION

All products in this catalog are sold for laboratory or manufacturing uses only and are not intended for human, drug, food additive, clinical or household use. Only professional laboratory staff should handle the chemicals.

GUARANTEE

SiliCycle® Inc. warrants, to the original purchaser, that the products listed in this catalog conform to the composition and purity described therein at the time of their shipment. Purchaser's sole remedy for failure to meet this warranty shall be replacement of the unused portion of the product, or at SiliCycle®'s option, a refund at the purchase price provided however, that the purchaser returns the alleged non-conforming product within 30 days of receipt of the product. SiliCycle® Inc makes no other warranty of suitability for a particular purpose or of the merchantability, in the use or handling of the product and does not accept any liability for consequential, special, indirect or incidental damages resulting therefrom.

RETURN

No return can be accepted without an authorization from SiliCycle® Inc. Returns received without authorization will be refused.

Product Number Index

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