SOLUTIONS FOR ORGANIC SYNTHESIS

SILICYCLE

SiliCycle

Founded in 1995, SiliCycle is specialized in the development, manufacturing and commercialization of high value silica gels and specialty products for chromatography, purification and synthesis.

Solutions for Organic Synthesis

Reagents & Oxidants for Organic Synthesis

- Easy product / API isolation and purification
- Eliminates or strongly reduces the need for laborious purifications
- No leaching of silica or catalyst and no cross contamination
- Suitable for either batch or continuous flow applications
- Compares very favourably to polymer-based reagents: no swelling, thermally & mechanically stable, and compatible with all solvents.

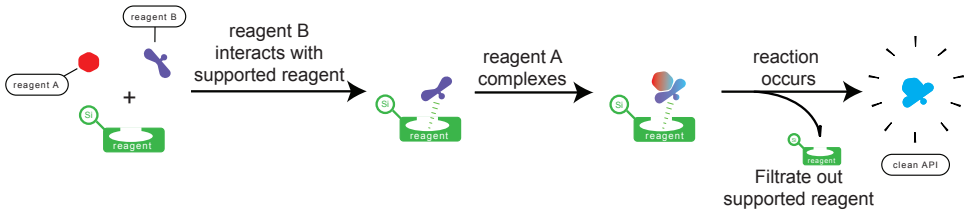
Silia*Bond*® Silica-Based Reagents & Oxidants

Increasingly, the use of heterogeneous reagents in organic synthesis and chemical production is growing in importance.

Although the strength of this technology has been acknowledged for a long time for applications in a large number of diverse and interesting chemical manipulations – thanks to its efficiency and eco-friendly character – the number of available reagents has lagged behind. At this time, SiliCycle has developed the most complete offer of heterogeneous reagents.

This technology is completely in line with the industries seeking improved sustainability and reduced ecological footprint.
This strong trend is directly derived from the inherent benefits offered by silica-based heterogeneous reagents & oxidants stated herein.

Here is the reaction mechanism:



Compatibility with Different Technologies

Functionalized Silicas in Flow Chemistry

Flow chemistry is being used more and more for large scale manufacturing because it only requires a small investment but enables the production of large quantities in a short time. The use of supported reagents and catalysts in flow chemistry offers many advantages over the traditional homogeneous ones, including ease of handling and purification. Silica presents no swelling, much higher mechanical and thermal stability and ease of scalability than polymer.

Reaction can be achieved using Silia*MetS* or Silia*Bond* in flow chemistry applications. Simply place the silica-based reagent or catalyst inside the solid-phase reactors provided with your flow system and let the solution to be purified flow through these reactors.

Solid-Supported Reagents & Catalysts in Microwave-Assisted Chemistry

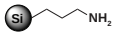
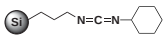
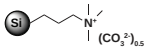
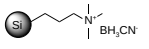
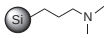
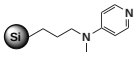
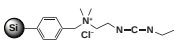
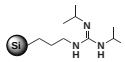
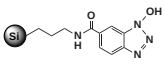
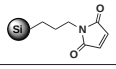
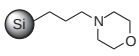
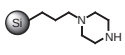
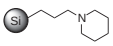
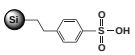
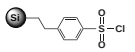
Fast kinetics, higher yields, excellent purity, wide compatibility of solvents and their applicability to a variety of reactions and applications are just some of the advantages that make scavengers very important tools in the laboratory.

After their introduction, chemists started to use supported reagents for solution-phase synthesis, and later on heterogeneous scavengers for post-reaction purification.

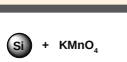
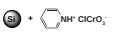
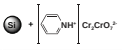
Reactions using Silia*MetS* and Silia*Bond* can also be done under microwave irradiation to provide excellent yield and purity in just minutes.

Functionalized Silia*Bond* Reagents Portfolio

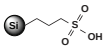
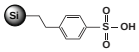
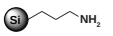
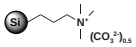
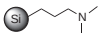
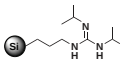
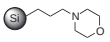
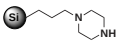
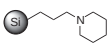
Catalysts & Reagents Portfolio

Name / Product Number	Structure	Loading / Density
Silia*Bond* $AlCl_3$ PN: R74530B		≥ 1.60 mmol/g 0.781 g/mL
Silia*Bond* Amine PN: R52030B		≥ 1.20 mmol/g 0.700 g/mL
Silia*Bond* Carbodiimide PN: R70530B		≥ 0.91 mmol/g 0.751 g/mL
Silia*Bond* Carbonate PN: R66030B		≥ 0.46 mmol/g 0.608 g/mL
Silia*Bond* Cyanoborohydride PN: R66730B		≥ 0.87 mmol/g 0.705 g/mL
Silia*Bond* Dimethylamine PN: R45030B		≥ 1.14 mmol/g 0.705 g/mL
Silia*Bond* Diphenylphosphine PN: R39030B		≥ 0.75 mmol/g 0.588 g/mL
Silia*Bond* DMAP PN: R75630B		≥ 0.53 mmol/g 0.674 g/mL
Silia*Bond* EDC PN: R70630B		≥ 0.32 mmol/g 0.770 g/mL
Silia*Bond* Guanidine PN: R68230B		≥ 0.80 mmol/g 0.732 g/mL
Silia*Bond* HOBt PN: R70730B		≥ 0.56 mmol/g 0.766 g/mL
Silia*Bond* Maleimide PN: R71030B		≥ 0.64 mmol/g 0.644 g/mL
Silia*Bond* Morpholine PN: R68030B		≥ 0.99 mmol/g 0.666 g/mL
Silia*Bond* Piperazine PN: R60030B		≥ 0.83 mmol/g 0.671 g/mL
Silia*Bond* Piperidine PN: R71530B		≥ 1.03 mmol/g 0.660 g/mL
Silia*Bond* Tosic Acid PN: R60530B		≥ 0.54 meq/g 0.698 g/mL
Silia*Bond* Tosyl Chloride PN: R44030B		≥ 0.63 mmol/g 0.761 g/mL

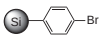
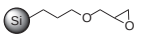
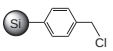
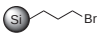
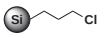
Oxidants Portfolio

Name / Product Number	Structure	Loading / Density
Silia*Bond* $KMnO_4$ PN: R23030B		10 % w/w 0.593 g/mL
Silia*Bond* PCC PN: R24030B		20 % w/w 0.693 g/mL
Silia*Bond* PDC PN: R24530B		20 % w/w 0.651 g/mL

Acids & Bases Portfolio

Name / Product Number	Structure	Loading / Density
Silia*Bond* Carboxylic Acid PN: R70030B		≥ 0.92 mmol/g 0.687 g/mL
Silia*Bond* Propylsulfonic Acid PN: R51230B		≥ 0.63 mmol/g 0.728 g/mL
Silia*Bond* Tosic Acid PN: R60530B		≥ 0.54 mmol/g 0.698 g/mL
Silia*Bond* Amine PN: R52030B		≥ 1.20 mmol/g 0.700 g/mL
Silia*Bond* Carbonate PN: R66030B		≥ 0.46 mmol/g 0.608 g/mL
Silia*Bond* Dimethylamine PN: R45030B		≥ 1.14 mmol/g 0.705 g/mL
Silia*Bond* Guanidine PN: R68230B		≥ 0.80 mmol/g 0.732 g/mL
Silia*Bond* Morpholine PN: R68030B		≥ 0.99 mmol/g 0.666 g/mL
Silia*Bond* Piperazine PN: R60030B		≥ 0.83 mmol/g 0.671 g/mL
Silia*Bond* Piperidine PN: R71530B		≥ 1.03 mmol/g 0.660 g/mL

Linkers Portfolio

Name / Product Number	Structure	Loading / Density
Silia*Bond* Allyl PN: R53530B		≥ 1.08 mmol/g 0.613 g/mL
Silia*Bond* Bromophenyl PN: R55030B		≥ 0.99 mmol/g 0.742 g/mL
Silia*Bond* Glycidoxy PN: R36030B		≥ 0.82 mmol/g 0.662 g/mL
Silia*Bond* Phenylmethylchloride PN: R56530B		≥ 1.14 mmol/g 0.637 g/mL
Silia*Bond* Propyl Bromide PN: R55530B		≥ 1.39 mmol/g 0.748 g/mL
Silia*Bond* Propyl Chloride PN: R59030B		≥ 1.39 mmol/g 0.751 g/mL

Available Formats

from 5 g to 25 kg, 50 kg, 100 kg, etc.

Not finding what you are looking for?

Contact us: info@silicycle.com

Typical Reactions Selection Tables

Reagents & Oxidants

A quick overview of how leach-free supported silicas can improve & ease your synthesis, either acting as:

1) supported Silia*Bond* reagents or oxidant

2) metal/organic chelator to purify final mixtures contaminated by excess homogeneous reagent or metallic residue

Silia*Bond* Reagents & Oxidants Typical Reactions Selection Table			
Reaction		**Best Silia*Bond* Reagent or Oxidant for Synthesis**	**Best Silia*Bond* Organic Scavenger to Remove Excess Reagent — OR — Best Silia*MetS* Metal Scavenger to Remove Excess Metal from Catalyst**
Acylation / Esterification		• **Silia*Bond* DMAP** • **Silia*Bond* $AlCl_3$** • **Silia*Bond* Tosic Acid**	• Various Silia*Mets* Metal Scavenger to remove metallic residues from homogeneous catalyst
Alkylation / Etherification		• **Silia*Bond* Guanidine** • **Silia*Bond* $AlCl_3$**	• Various Silia*Mets* Metal Scavenger to remove metallic residues from homogeneous catalyst • Silia*Bond* Carbonate to remove excess homogeneous HOBt
Amide Coupling	With acids, acid chlorides and amines	• **Silia*Bond* Carbodiimide** • **Silia*Bond* EDC**	• Silia*Bond* Amine to remove excess acid chloride • Silia*Bond* Carbamate or Tosic Acid to remove excess amine
	Using HOBt	-	• Silia*Bond* Carbonate to remove excess homogeneous HOBt
Catalytic Hydrogenation		-	• Silia*MetS* Thiol, Thiourea or DMT to remove Pd • Silia*MetS* DMT, Diamine or Triamine to remove Pt • Silia*MetS* DMT, DOTA, Imidazole or TAAcONa to remove Ni
Coupling Reactions Buchwald Amination, Heck, Kumada, Negishi, Sonogashira, Stille & Suzuki Couplings and more...		-	• Silia*Bond* Isocyanante or Tosic Acid to remove excess amine • Silia*MetS* Thiol, Thiourea or DMT to remove Pd • Silia*MetS* DMT, DOTA, Imidazole or TAAcONa to remove Ni • Silia*MetS* DOTA, Imidazole or TAAcONa to remove Cu
Deprotection of Aromatic Ether		• **Silia*Bond* Tosic Acid**	-
Ether Formation		• **Silia*Bond* $AlCl_3$** • **Silia*Bond* Tosic Acid**	-
Fmoc, Bsmoc Deprotection of Amino Acid		• **Silia*Bond* Piperazine**	• Silia*Bond* Amine, DMAP, Piperazine, Silia*MetS* Diamine or Triamine to remove excess FMOC-Cl or Bsmoc-Cl
Friedel-Crafts Alkylation		• **Silia*Bond* $AlCl_3$**	-
Fries-Speier Esterification		• **Silia*Bond* Tosic Acid**	-
Grubbs Metathesis		-	• Silia*MetS* DMT or Cysteine to remove Ru
Knoevenagel Condensation		• **Silia*Bond* Amine** • **Silia*Bond* Dimethylamine** • **Silia*Bond* Piperidine** • **Silia*Bond* Piperazine**	-
Michael Addition		• **Silia*Bond* Dimethylamine** • **Silia*Bond* Guanidine**	• Silia*MetS* TAAcONa to remove Li • Silia*MetS* Thiol, Thiourea or DMT to remove Pd
Nitro-Aldol (*or Henry*) Reaction		• **Silia*Bond* Carbonate**	• Silia*MetS* DOTA, Imidazole or TAAcONa to remove Cu
Oxidation	Alcohols to acids	• **Silia*Bond* $KMnO_4$**	-
	Alcohols to ketones / aldehydes	• **Silia*Bond* PCC & PDC**	-
Reduction (*Reductive Amination, Alkylation, etc.*)		• **Silia*Bond* Cyanoborohydride**	• Silia*Bond* Tosic Acid to remove excess borohydride or excess amine
Sharpless Dihydroxylation		-	• Silia*MetS* Thiol, DMT, Cysteine, Imidazole, TAAcOH or TAAcONa to remove Os
Sulfonamide Synthesis		• **Silia*Bond* EDC**	• Silia*Bond* Amine to remove excess sulfonyl chloride
Tosylate Formation		• **Silia*Bond* Tosyl Chloride**	-
Urea Synthesis		• **Silia*Bond* DMAP**	• Silia*Bond* Amine to remove excess isocyanate
Williamson Ether Synthesis		• **Silia*Bond* Guanidine**	-

Acids & Bases

SiliaBond Acids & Bases Typical Reactions Selection Table		
Classification	**Best SiliaBond Acids & Bases**	**Typical Reactions & Applications Examples**
Acids	**SiliaBond Carboxylic Acid**	• Nucleophilic acyl substitutions: esters hydrolysis, Fisher esterifications, amides hydrolysis, etc. • A chromatographic phase week cation exchanger at pH ≥ 6.8 that can be eluted at a pH ≤ 2.8
	SiliaBond Propylsulfonic Acid	• Nucleophilic acyl substitutions such as transesterifications, etc. • Carbon-carbon coupling reactions
	SiliaBond Tosic Acid	• A chromatographic phase strong cation exchanger that is permenently negatively charged (*pKa < 1*) • Ionic scavenging • Deprotections of aromatic ethers • Fries rearrangements
Bases	**SiliaBond Amine**	• Organic scavenging of electrophiles • Ionic scavenging • Nucleophilic-catalyzed reactions • Acid-catalyzed reactions: Aldol reactions, Retro-Claisen reaction, Mannich reactions...
	SiliaBond Carbonate	• Ionic scavenging • Nitro-Aldol (*Henry*) reactions & Michael additions • Amine free-basing • Compatible with solvent-free conditions
	SiliaBond Dimethylamine	• Knoevenagel condensations • Catch andd release purification of compounds bearing a permanent negative charge such as salts of sulfonic acids
	SiliaBond Guanidine	• Alkylations • Strecker-type reactions • Etherifications such as Williamson synthesis • Michael additions and more generally speaking 1,4 addition reactions • Ionic scavenging • Deprotonates moderately acidic hydrogens
	SiliaBond Morpholine	• Acid sponge • Enamine formations • Mannich condensations • Less nucleophilic and less basic than piperidine hence forming stable chloramines
	SiliaBond Piperazine	• Deprotecting and scavenging agent for Fmoc and Bsmoc amino protecting groups • Knoevenagel condensations • Ionic & nucleophile scavenger
	SiliaBond Piperidine	• Deprotecting and scavenging agent for Fmoc and Bsmoc amino protecting groups • Knoevenagel condensations • Ketones to enamines conversions • Production of dipiperidinyl dithiuram tetrasulfide (*rubber vulcanization accelerator*)

Linkers

SiliaBond Linkers Typical Reactions Selection Table		
Classification	**Best SiliaBond Acids & Bases**	**Typical Reactions & Applications Examples**
Synthesis of homemade functionalized silicas according to your very own application	**SiliaBond Allyl**	• Allylic oxidations • Ene reactions • Tsuji-Trost reactions • Rancidification
	SiliaBond Bromophenyl	• Introduction of phenyl groups via Pd-catalyzed couplings • Synthesis of Grignard reagents
	SiliaBond Glycidoxy	• Immobilization of molecules bearing amino, hydroxy, mercapto and thiocarboxylic acid groups • Ring-opening reactions & hydrolysis • Reduction with tungsten hexachloride • Reduction with lithium aluminum hydride
	SiliaBond Phenylmethylchloride	• Nucleophilic substitutions for introduction of phenyl linker
	SiliaBond Propyl Bromide	• Nucleophilic substitutions for introduction of n-propyl linker

Reagents for Organic Synthesis

Acylation/Esterification & Alkylation/Etherification Reagents

Acylations are the addition of an acyl group (*RCO*) via electrophilic substitution, whereas esterifications are the formation of esters (*RCOOR*) from a derived carboxylic acid. The typical acylation reaction is the Friedel-Crafts, and other acyl transfers include the Boekelheide, Kostanecki, Passerini reactions, the Pummerer rearrangement, etc. The typical esterification reaction is the Fischer reaction, and other ester synthesis include the Fisher-Speier modification, Mitsunobu reaction, the Steglich esterification, etc.

For both reactions DMAP (*4-dimethylaminopyridine*) is well-known as an acyl-transfer catalyst, to increase speed and yield of alcohol and phenol acylations over acetic and benzoic anhydrides. Tosic Acid, on the other hand, is a very popular acid catalyst for esterification & transesterification of esters and $AlCl_3$ is probably one of the most commonly used Lewis acid as a catalyst for Friedel-Crafts reactions.

Alkylation reactions are the transfer of an alkyl group from one molecule to the other via alkylating agents, that may have an electrophilic or nucleophilic character. Etherifications are a type of C-O bond formation reaction, usually from the S_N2 reaction between an organohalide and an alcohol. Just like for acylation reactions, the most common type of alkylation is the Friedel-Crafts reaction and the typical etherification reaction is the Williamson synthesis.

Silia*Bond* Aluminum Chloride (*Si-AlCl$_3$*) - R74530B

Typical Application: Acylations, esterifications

Loading: ≥ 1.60 mmol/g **Density**: 0.781 g/mL **Endcapping**: No

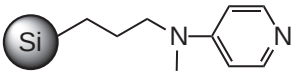
Solvent Compatibility: Anhydrous, desgassed and organic solvents

Storage: Keep cool (< 8°C), dry and under argon

Silia*Bond* Aluminum Chloride is a milder Lewis acid compared to homogeneous aluminum chloride which is so reactive that it often lacks selectivity and causes the formation of unwanted by-products.[1] It is an effective catalyst for Friedel-Crafts alkylations[2-4], acylations, and ethers formation. This reagent is removed by a simple filtration, avoiding the destructive water quench which produces large amounts of hazardous waste.[5,6] Silia*Bond* 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 and is no longer reactive.

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

Silia*Bond* DMAP (*Si-DMAP*) - R75630B



Typical Application: Acylations, esterification

Loading: ≥ 0.32 mmol/g **Density**: 0.674 g/mL **Endcapping**: Yes

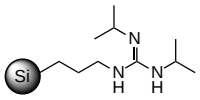
Solvent Compatibility: All solvents, aqueous and organic

Storage: Keep cool (< 8°C), dry and under argon

Silia*Bond* DMAP is the heterogeneous catalyst equivalent of 4-dimethylaminopyridine, which is used as a nucleophilic catalyst in a wide variety of reactions such as acylations and Baylis-Hillman reactions. These reactions are well known in organic synthesis and are very useful in various applications. Silia*Bond* DMAP has an advantage over its free counterpart as it can be removed by a simple filtration.

[1] *The Peptides: Analysis, Synthesis, Biology; Academic: New York*, **1979**, *1*, 241

Silia*Bond* Guanidine (*Si-GUA*) - R68230B



Typical Application: Williamson ether synthesis, Strecker-type reactions, 1,4 addition reactions

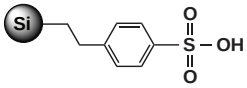
Loading: ≥ 0.80 mmol/g **Density**: 0.732 g/mL **Endcapping**: No

Solvent Compatibility: All solvents, aqueous and organic

Storage: Keep dry

Silia*Bond* Guanidine is a silica-bound guanidine moiety that is sufficiently basic to deprotonate moderately acidic hydrogens. It is most commonly used in Williamson synthesis, 1,4 addition reactions, Strecker-type reactions, etc.

Silia*Bond* Tosic Acid (*Si-SCX*) - R60530B



Typical Application: Esterification, deprotection of aromatic thers

Loading: ≥ 0.54 mmol/g **Density**: 0.698 g/mL **Endcapping**: Yes

Solvent Compatibility: All solvents, aqueous and organic

Storage: Keep dry

Silia*Bond* Tosic Acid is in a class of strong acids used in different fields of synthetic organic chemistry. The aromatic ring makes it slightly more acidic than other supported sulfonic acids. Silia*Bond* Tosic Acid used as an acid catalyst for Fischer-Speier esterification provides excellent conversion. Silia*Bond* Tosic Acid can also be used as a metal scavenger.

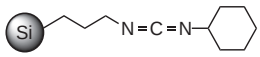
Amide Coupling Reagents

The amide bond is the defining molecular structure of proteins and peptides. Usually, the amide bond formation relies on the use of an excess of toxic coupling reagents such as carbodiimides or supernucleophiles.

These chemicals produce a large amount of by-products, which tends to complicate the isolation and purification of the desired amide product. Solid-phase reagents are valuable for amide coupling with a carboxylic acid because they generate less unwanted side products.

Other advantages of using solid-supported reagents include improved stability, toxic chemical immobilization, the ability to run multiple transformations in a single pot and the flexibility to use batch reactions, microwave irradiation and flow chemistry.

Silia*Bond* Carbodiimide (*Si-DCC*) - R70530B



Typical Application: Amide coupling with acids, acyl chlorides and amines

Loading: ≥ 0.91 mmol/g **Density**: 0.751 g/mL **Endcapping**: Yes

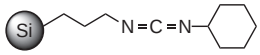
Solvent Compatibility: Aprotic sovents

Storage: Keep cool (< *8°C*), dry and under argon

1,3-Dicyclohexylcarbodiimide (*DCC*) has arguably become the most commonly used reagent in peptide synthesis and other amide bond-forming reactions of primary and secondary amines with carboxylic acids.[1] The major drawback associated with using DCC is the formation of the urea by-product (*DCU*) which remains in solution and requires additional purification steps to remove. However, by using covalently bonded DCC on silica, it is possible to avoid problematic purifications. Only a simple filtration step is needed to remove the unwanted DCU.

[1] *Chem. Rev*., **1981**, *81*, 589

Silia*Bond* Ethyl-Dimethylamino Carbodiimide (*Si-EDC*) - R70630B



Typical Application: Amide coupling with acids, acyl chlorides and amines

Loading: ≥ 0.32 mmol/g **Density**: 0.770 g/mL **Endcapping**: Yes

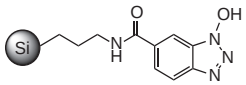
Solvent Compatibility: Aprotic sovents

Storage: Keep cool (< *8°C*), dry and under argon

A recent literature review shows that 1-ethyl-3 (3-dimethylaminopropyl) carbodiimide (*EDC*) has become recognized as one of the best reagents for amide coupling reactions. Unfortunately, using the EDC basic tertiary amine results in the formation of urea, which has to be separated from the product by acidic aqueous extractions.[1] By attaching EDC to silica, it is possible to avoid this problematic work-up without sacrificing the useful carbodiimide reactivity. In fact, Silia*Bond* EDC behaves in a similar fashion as EDC in solution, but the by-product remains on the solid support.

[1] *The Peptides: Analysis, Synthesis, Biology; Academic: New York*, **1979**, *1*, 241

Silia*Bond* HOBt (*Si-HOBt*) - R70730B



Typical Application: Avoiding or reducing racemization during chiral amide synthesis

Loading: ≥ 0.56 mmol/g **Density**: 0.766 g/mL **Endcapping**: Yes

Solvent Compatibility: Aprotic solvents

Storage: Keep dry

Hydroxybenzotriazole (*HOBt*) has been used for increasing yield and decreasing racemization during chiral amide synthesis. However, dry HOBt can undergo exothermic decomposition. Bonding HOBt to silica eliminates this risk of explosion. Silia*Bond* HOBt can be easily activated and should ideally be used with a base such as N,N-diisopropylethylamine in the same conditions as in homogeneous solution. Moreover, this supported reagent can be reused a few times without adversely affecting its performance.

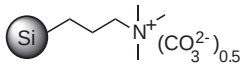
Nitro-Aldol (*or Henry*) Reaction Reagents

The Henry reaction is commonly used to form carbon-carbon bonds by addition of nitroalkanes over aldehydes.

This reaction is a useful technique in organic chemistry due to the synthetic utility of its corresponding products, as they can be easily converted to other useful synthetic intermediates such as nitroalkenes by dehydrogenation, α-nitro ketones by oxidation and β-amino alcohols by reduction.

Usually, the Henry reaction is carried out in presence of bases in homogeneous solution, giving low yield due to side reactions such as retroaldol and Cannizarro reactions.

Silia*Bond* Carbonate (*Si-CO$_3$*) - R66030B



Typical Application: Nitro-Aldol reactions (Henry reaction) ree basing of amines

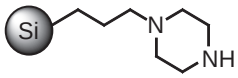
Loading: ≥ 0.46 mmol/g **Density**: 0.608 g/mL **Endcapping**: Yes

Solvent Compatibility: Aprotic sovents

Storage: Keep dry

Used as a heterogenous catalyst in the Henry reaction, Silia*Bond* Carbonate is replacing the use of expensive and toxic heterogeneous catalysts. Silia*Bond* Carbonate in catalytic amounts drive the reaction forward to high yield with or without solvent. Silia*Bond* Carbonate is also an excellent product for amine free-basing.

Silia*Bond* Piperazine (*Si-PPZ*) - R60030B



Typical Application: Knoevenagel synthesis, Fmoc and Bsoc deprotection, organic scavenger

Loading: ≥ 0.83 mmol/g **Density**: 0.671 g/mL **Endcapping**: Yes

Solvent Compatibility: All solvents, aqueous & organic

Storage: Keep cool (< 8°C) and dry

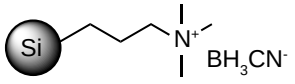
Silia*Bond* Piperazine (*Si-PPZ*) is a very useful solid-phase Knoevenagel catalyst. According to the results of a study, Si-PPZ is superior to its polystyrene-based equivalent.[1-3] Silia*Bond* Piperazine is a useful deprotecting and scavenging agent for Fmoc and Bsmoc amino protecting groups, as well as a great electrophile scavenger.

[1] *J. Org. Chem.,* **1983**, *48*, 666 [2] *J. Org. Chem.,* **1999**, *64*, 4324 [3] *J. Org. Chem.,* **2010**, *51*, 6670

Reductive Amination Reagents

Reductive amination involves the conversion of a carbonyl group, most of the time a ketone or an aldehyde, to an amine via an intermediate imine or iminium. The intermediate imine is reduced by sodium cyanoborohydride. This is known as direct reductive amination and is carried out with reducing agents that are more reactive toward protonated imines (*or iminiums*) than ketones and are stable under moderately acidic conditions.

Silia*Bond* Cyanoborohydride (*Si-CBH*) - R66730B



Typical Application: Reductive amination

Loading: ≥ 0.87 mmol/g **Density**: 0.705 g/mL **Endcapping**: Yes

Solvent Compatibility: All solvents, aqueous and organic

Storage: Keep cool (< *8°C*), dry and under argon

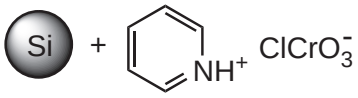
Silia*Bond* Cyanoborohydride is the silica-bound equivalent of sodium cyanoborohydride. Bound cyanoborohydride is very useful in reductive amination and in the reduction of imines and aldehydes. However, when using the solution phase equivalent, cyanide contamination of the product is a concern. This problem is minimized with the use of silica-bound materials since the toxic cyanide residue remains on the silica. To see if any cyanide ion was leaching from the silica, 1 g of Silia*Bond* Cyanoborohydride was slurried in 10 mL of methanol for 24 h. Cyanide strips indicated less than 3 ppm in each test performed. In addition to providing superior conversions, acetic acid was not needed (*eliminating issues with acid labile groups*), the work-up required only a filtration and HCN nor NaCN were liberated during work-up.

Oxidation Reaction Reagents

Oxidations are such a key and fundamental organic reaction in synthesis. Alcohols having at least one hydrogen atom bonded to the hydroxyl group bearing carbon, namely primary and secondary alcohols, can be oxidized to carbonyl compounds.

Primary alcohols (*R-CH_2-OH*) can be oxidized to aldehydes (*R-CHO*), which can further be oxidized into carboxylic acids (*R-CO_2H*), while the oxidation of secondary alcohols (*R_1R_2CH-OH*) will terminate at the ketone (*R_1R_{22}=O*) stage. Tertiary alcohols (*$R_1R_2R_3$C-OH*) will not undergo similar oxidation.

Silia*Bond* Pyridinium Chlorochromate (*Si-PCC*) - R24030B



Typical Application: Oxidation of alcohols to aldehydes / ketones

Loading: 20 % w/w **Density**: 0.693 g/mL **Endcapping**: No

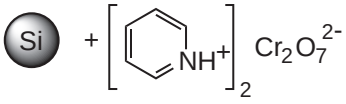
Solvent Compatibility: Anhydrous CH_2Cl_2

Storage: Keep cool (< 8°C) and dry

Commonly used for the oxidation of alcohols to carbonyl compounds, selective oxidation of allylic and benzylic alcohols, organometallic oxidations, oxidative transpositions, oxidative cleavages, allylic and benzylic oxidation and oxidative cyclizations.[1-4] Using PCC immobilized onto silica gel provides anhydrous conditions that minimize the risk of side reactions and reduced 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]

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

Silia*Bond* Pyridinium Dichromate (*Si-PDC*) - R24530B



Typical Application: Oxidation of alcohols to aldehydes / ketones

Loading: 20 % w/w **Density**: 0.651 g/mL **Endcapping**: No

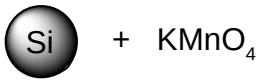
Solvent Compatibility: Anhydrous CH_2Cl_2

Storage: Keep cool (< 8°C) and dry

Great alternative to *Si*-PCC in nucleoside and carbohydrate oxidation, particularly for fragile molecules.[1] Silia*Bond* PDC can also be used in conjunction with *tert*butylhydroperoxide for a variety of oxidative transformations.[2] *Si*-PDC is a very convenient and effective reagent for oxidizing allylic and benzylic alcohols, saturated with acid-sensitive groups, such as cyclopropane rings or ketal functions.[3]

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

Silia*Bond* Potassium Permanganate (*Si-$KMnO_4$*) - R23030B



Typical Application: Oxidation of alcohols to acids

Loading: 10 % w/w **Density**: 0.593 g/mL **Endcapping**: No

Solvent Compatibility: Anhydrous CH_2Cl_2

Storage: Keep dry

This product is a strong oxidant that will oxidize alcohols and aldehydes to carboxylic acids. Silia*Bond* Potassium Permanganate increases recovery, facilitates work-up and expands the scope of the chemistry because it can be used in all organic solvents eliminating solubility issues.[1] With Silia*Bond* Potassium Permanganate, the manganese salt by-products stay adsorbed onto the silica.

[1] *Synlett*, **2001**, *10,* 1555

Application Notes and Case Studies

Below are a few examples of applications notes and case studies using Silia*Bond* Reagents & Oxidants.

Please contact us if you're interested in some of these: info@silicycle.com.

Acylation & Esterification Reactions

- Acylation & Esterification Reactions with Si-DMAP
- Baylis-Hillman Reaction (*Si-DMAP*): Comparative Study with PS-DMAP
- Fischer-Speier Esterifications (*Si-SCX*)
- Case Study: Synthesis of pyran-based macrocycles (*Si-DCC*)
- SN1 Acylation of Triphenylcarbinol (*Si-$AlCl_3$*): Comparative Study with PS-$AlCl_x$

Deprotection Reactions

- Deprotection of Methoxymethyl Groups (*Si-SCX*)

Alkylation & Etherification Reactions

- General Williamson Ether Synthesis (*Si-GUA*)
- Friedel-Crafts Alkylation of Benzene (*Si-$AlCl_3$*): Comparative Study with Homogeneous $AlCl_3$
- Comparative Study of Alkylation in Flow Chemistry

Amide Coupling Reactions

- Synthesis of Capsaicin Analogues (*Si-DCC or Si-EDC*)
- Synthesis of Amide Derivatives of Indomethacin (*Si-DCC*)
- Synthesis of Formylated Amino Acids (*Si-EDC*)
- Amine Protection Using Benzylcarbamate Group (*Si-HOBt*)
- Amine Protection Reaction (*Si-HOBt*)
- Weinreb and Acylsulfonamide Synthesis (*Si-DCC*)

Oxidation Reactions

- Oxidation of Alcohols to Ketones and Aldehydes

Aldol Reactions

- 3-Nitrooctan-4-ol Synthesis via a Nitro-Aldol (or Henry) Reaction (*Si-CO_3*)
- Knoevenagel Condensations (*Si-PPZ*)
- Jasminaldehyde Synthesis via Aldol Reaction (*Si-PPZ*)

Reductive Amination Reactions

- Reductive Aminations (*Si-CBH*)
- Case Study: Synthesis of Histone Deacethylase Inhibitors
- ...

Ordering Information for Batch Reactor Mode (*Bulk*)

Our reagents are available in the following sizes: 5 g, 10 g, 25 g, 50 g, 100 g, 250 g, 500 g, 1 kg, 5 kg, 10 kg, 25 kg, etc.
All Particle Size and Pore Size are respectively 40 - 63 µm, 60 Å. Other matrices are available upon request. Contact us: info@silicycle.com

SiliaBond Reagents	
Reagent Name	**Reagent PN**
SiliaBond $AlCl_3$	R74530B
SiliaBond Amine	R52030B
SiliaBond Carbodiimide	R70530B
SiliaBond Carbonate	R66030B
SiliaBond Cyanoborohydride	R66730B
SiliaBond Dimethylamine	R45030B
SiliaBond Diphenylphosphine	R39030B
SiliaBond DMAP	R75630B
SiliaBond EDC	R70630B
SiliaBond Guanidine	R68230B
SiliaBond HOBt	R70730B
SiliaBond Maleimide	R71030B
SiliaBond Morpholine	R68030B
SiliaBond Piperazine	R60030B
SiliaBond Piperidine	R71530B
SiliaBond Tosic Acid	R60530B
SiliaBond Tosyl Chloride	R44030B

SiliaBond Oxidants	
Oxidant Name	**Oxidant PN**
SiliaBond $KMnO_4$	R23030B
SiliaBond PCC	R24030B
SiliaBond PDC	R24530B

SiliaBond Acids & Bases	
Acid / Base Name	**Acid / Base PN**
SiliaBond Carboxylic Acid	R70030B
SiliaBond Propylsulfonic Acid	R71230B
SiliaBond Tosic Acid	R60530B
SiliaBond Amine	R52030B
SiliaBond Carbonate	R66030B
SiliaBond Dimethylamine	R45030B
SiliaBond Guanidine	R68230B
SiliaBond Morpholine	R68030B
SiliaBond Piperazine	R60030B
SiliaBond Piperidine	R71530B

SiliaBond Linkers	
Linker Name	**Linker PN**
SiliaBond Allyl	R53530B
SiliaBond Bromophenyl	R55030B
SiliaBond Glycidoxy	R36030B
SiliaBond Phenylmethylchloride	R56530B
SiliaBond Propyl Bromide	R55530B
SiliaBond Propyl Chloride	R59030B

Available Kits

For screening purposes, especially if you are new to this technology, we have convenient kits for testing various funtionalities and various experimental conditions, to select the ones that best fit your synthetic application.

How to order

All these kits are procurable in 5 g, 10 g, 25 g, 50 g and 100 g formats (*custom formats are also available, contact us for more details*).

To build your kit's product number, just add the Format to the Kit PN: **[Kit PN]-[Format]**
Example: 10 g of each one of the '*Oxidant Kit*' reagents: K30330B-10G.

SiliaBond Kits		
Kit Name	**Kit PN**	**Composition**
SiliaBond Base Kit	K31630B	Amine, Carbonate, Dimethylamine, Diethylamine, Morpholine, Pyridine & Guanidine
SiliaBond Oxidant Kit	K30330B	Potassium Permanganate, TEMPO, Pyridinium Chlorochromate & Pyridinium Dichromate
SiliaBond Reversed-Phase Kit	K32530B	C8 mono, C18 (*17 %*), C18 (*17 %*) Mono, C18 (*23 %*), Cyano & Phenyl
SiliaBond Acid Kit	K31330B	Carboxylic Acid, Propylsulfonic Acid, TAAcOH & Tosic Acid
SiliaBond Reagent Kit	K32230B	Carbodiimide, Cyanoborohydride, DMAP, EDC & HOBt
SiliaBond Ion Exchanger Kit	K31430B	WAX, WCX, SCX-2, SCX, SAX & TMA Acetate

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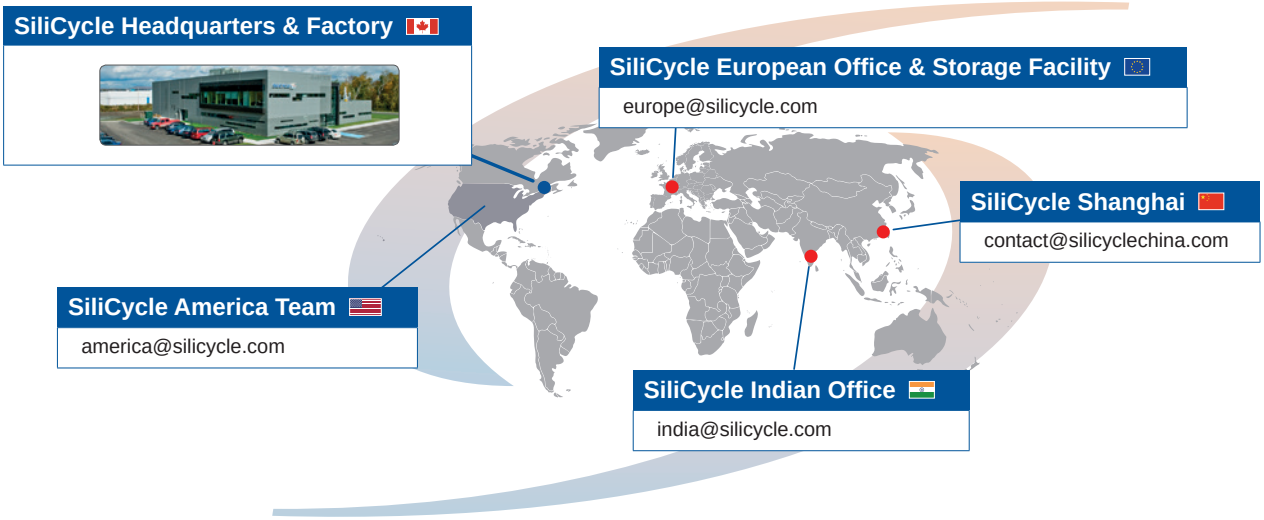
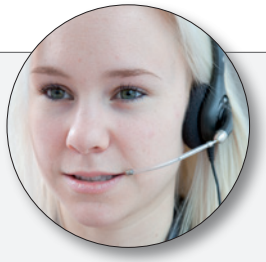


Technical Support



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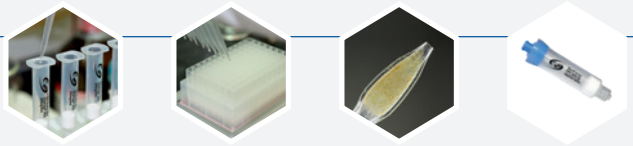
Silia*Prep*™ – Micro-SPE Tips
Silia*Fast*™ – FaPEx® Cartridges for Pesticide Residue Analysis



ANALYTICAL & PREPARATIVE CHROMATOGRAPHY

Silia*Sphere*™ – Spherical Silica Gels
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ORGANIC SYNTHESIS

Silia*Bond*® – Reagents & Oxidants

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