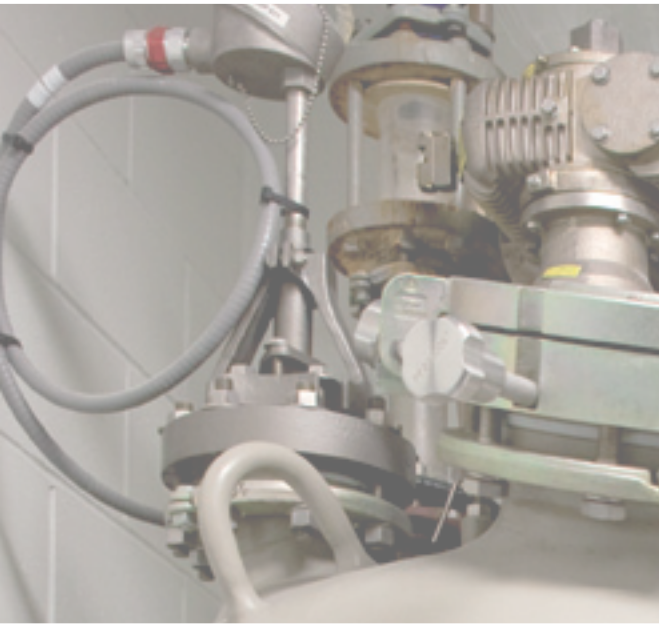

SiliCycle®

Catalog for the Pharmaceutical Industry

SILICYCLE Inc.®

UltraPure SILICA GELS

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About SiliCycle

Founded in 1995, SiliCycle® Inc. is a worldwide leader in the development, the manufacturing and the commercialization of silica gel products for chromatography, analytical and organic chemistry. With our multi-ton manufacturing capability, we are your partner of choice for all your metal removal, catalysis, synthesis, and purification requirements.

Our business extends to more than fifty countries worldwide and our customer portfolio includes companies in the pharmaceutical, biotechnology industries, contract research and manufacturing organizations as well as university laboratories and hospital research centers.

The mission of SiliCycle is to develop and market innovative silica products of high value to customers and make a technical contribution to their work.

At SiliCycle, we are at the forefront of the chromatography industry, owing to the extraordinary purity of our silica gels and our capacity to rapidly adapt these gels to meet the specific requirements of pharmaceutical professionals and university scientists.

We lead the way in offering innovative products, such as Silia*Cat*® heterogeneous catalysts, Silia*MetS*® Metal Scavengers, Silia*Bond*® functionalized silica gels , Silia*Flash*® Irregular silica gels, IMPAQ® angular silica gels, Silia*Sphere*™ spherical silica gels, Silia*Sep*™ flash cartridges, Silia*Prep*™ SPEs and Well Plates, Silia*Plate*™ TLC plates, and Silia*Chrom*® HPLC columns.

We offer a wide variety of first-rate Ultra Pure Silica Gels. Our automated manufacturing process, which includes acid washing and multiple analyses, is continuously optimized to ensure high purity and a low percentage of fine particles, thereby guaranteeing optimal performance.

We are committed to provide the highest quality products and services in the industry.

TEL.: 1 418 874.0054 FAX: 1 418 874.0355 TOLL-FREE: 1 877.SILICYCLE (NORTH AMERICA ONLY) WWW.SILICYCLE.COM INFO@SILICYCLE.COM

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SiliCycle for the Pharmaceutical Industry

Drug Discovery

SiliCycle is a recognized industry leader of innovative purification and synthesis methods for more efficiency in drug discovery.

Our products are particularly well suited for drug discovery chemists that perform amide couplings, reductive aminations, metal mediated couplings, etc., on a daily basis. Our supported reagents and catalysts greatly simplify the reaction and work-up process, enabling chemists to run more reactions and generate more compounds. Furthermore, we have a whole range of flash cartridges and TLC plates to assist chemists in the purification of these compounds.

We are your number 1 metal removal solution provider, and your partner of choice for your synthesis, heterogeneous catalysis, analysis and all of your purification requirements.

We commit ourselves to offer you best quality products accompanied by expert technical support at a competitive price!

Drug Development

SiliCycle designs, develops, and manufactures innovative products for world class pharmaceutical companies with gram to multi-ton production capabilities.

For large scale purifications, our state-of-the-art facility allows us to produce high quality chromatographic phases in large batches to supply the most demanding applications.

We also produce large amounts of Silia*MetS* Metal Scavengers for the selective removal of spent metal catalysts traces from active pharmaceutical ingredients (*API*). These ligands bound to silica gel, in bulk or in cartridge formats, are especially designed to remove metal traces down to single digit ppm levels fast and reproducibly. A simple filtration is then performed to get rid of the silica scavenger with the metals entrapped.

We produce high quality chromatographic phases for any separation project, large or small. We can supply large quantities of normal, reverse, and ion-exchange phases that will give you the best performance at a competitive price.

Our new line of Silia*Cat* Heterogeneous Catalysts are also very valuable tools for scale-up and process. These catalysts are easy to use and very efficient. At the end of the reaction, a simple filtration removes the spent catalyst and leaves the reaction mixture free of any metal traces.

Process and Manufacturing

As a world-wide supplier of premium silica-based products for pharmaceutical and biotechnology drug manufacturers, SiliCycle has become a value-added, strategic sourcing partner for our customers. At SiliCycle, we truly understand the needs and challenges you encounter when trying to satisfy both the regulatory requirements and the need for economical validated manufacturing. Listed below are some of the solutions SiliCycle provides to better serve you:

On-time Delivery

As a critical component supplier, SiliCycle understands the importance of maintaining and managing its inventory. As a manufacturer of hundreds of tons of silica-based products, you can feel confident that we will deliver your product on-time.

Batch Reservations

For our customers that do not have the storage capacity, SiliCycle can reserve specific batches of finished product and ship upon request.

Packaging sizes

The wide range of available packaging sizes and formats help eliminate waste and reduce release testing.

Batch Sizes

SiliCycle's proprietary manufacturing processes can easily be scaled-up to meet the batch size requirements of our customers.

Customized Products

Since SiliCycle controls the manufacturing process, we can customize the particle size distribution, loading, defined water content and any other specification our customers require.

Regulatory Filing

SiliCycle will work with your quality team to provide the necessary documentation and specific analytical testing needed for your regulatory filings.

Metal Scavenging Screening Service

Under a CDA, we will screen a customer's metal contaminated reaction mixture against our Silia*MetS*® Metal scavengers to determine the best scavenger and the best conditions.

Catalyst Screening Service

Looking for the right catalyst to use? SiliCycle's R&D team can find the optimal conditions for you.

Custom Phase Synthesis

We have the knowledge to graft any function (small molecules, peptides, sugars, and proteins) onto silica gel and we do that for a customer' specific application for catalysis, support, or chromatography.

CONTACT US for more details.

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Word from the President

Dear valued customers,

It is with great pleasure and pride that we present our new catalog tailored for the pharmaceutical industry. This document is specifically dedicated to meet the needs of players in the fields of drug discovery, drug development, and drug manufacturing.

For over 15 years, we have been designing, manufacturing, and commercializing high performance silica-based products for chromatography, analytical and organic chemistry. Over these years, thanks to our innovations and the quality of our products and services, we have positioned ourselves among the leaders in the fine chemical industry. Our business now extends to over fifty countries, and we are still growing. Today, we enjoy the trust of major pharmaceutical companies including Abbott, Amgen, AstraZeneca, Eli Lilly, GlaxoSmithKline, Johnson & Johnson, Novartis and Pfizer, just to name a few.[1]

From Montreal to Sanghai, from New Jersey to Paris or Mumbai, at SiliCycle, we are committed to delivering the same quality products and services, no matter where you are. Our ISO 9001:2008 certification is a testimony to the importance we place on quality. Likewise, our C-TPAT certification ensures an unfailing supply to our customers worldwide. For our North American customers, we have multiplied our sales staff and customer service agents. For our European clients, we now have a warehouse in Frankfurt, Germany. For our customers in India and Europe, we have local, Ph.D.-level staff on hand to better serve you in real time. The same will be available soon for our Chinese customers as well.

To support increasing demand, and ensure our continued growth, we recently moved into a brand new facility, equipped with cutting-edge technology and multi-ton capability. As a partner of choice for your metal removal, purification, catalysis, analysis and synthesis needs, we offer you a full range of products available in all the formats required by the industry, making us "*The one stop shop*".

Presented herein, you will find all the information you need to choose the right products for your applications. Choose from our famous Silia*MetS* – the number 1 Metal Scavengers in their category; Silia*Cat* – our new high-performance family of heterogeneous catalysts; the versatile Silia*Bond* – a complete set of functionalized silica-based products; Silia*Flash* – the best quality for price of all irregular silica gels; Silia*Sep* – Flash cartridges; Silia*Prep* – SPE cartridges; Silia*Chrom* – HPLC Columns; Silia*Sphere* – spherical silica gels; IMPAQ – angular silica gels; Silia*Plate* – TLC plates; and many others.

Much more than just products, SiliCycle's team will support you in your research and your large-scale production needs. As a human size company, I can guarantee you that our highly skilled people will give you a personalized service. Contact us and see for yourself how easy — and friendly — it is to do business with SiliCycle.

Most of all, I want to thank you for your trust and business over all these years. Enjoy our new catalog. Hopefully, it will become a reference tool for you, which was our goal when we set to design it.

Hugo St-Laurent
president & CEO

[1]Please note that the names of the companies are listed in alphabetical order, and do not reflect in any way an order of importance.

Word of the Vice-President of R&D

Dear fellow chemists,

The publication of a new catalog has been an interesting time for the R&D group. We were reminded of the hard work that has taken place since our last catalog outing: the new products, the applications, and the services. It is with great pride that our researchers see their projects succeed, the products come to market, and other chemists benefit and develop new medicines that in turn help all of us.

For all of the researchers, chemists, students, and other scientists in drug discovery, drug development and production, and university laboratories, we have silica-based products that will meet and exceed your chromatography, purification, and synthesis needs.

Over the years, we have developed extensive knowledge of silica gel and the ways it can be modified to meet the demands of diverse applications. From chromatography phases for your most demanding separations to metal scavengers used in the selective removal of spent catalysts from active pharmaceutical ingredients and our new SiliaCat catalysts, we have products that make running your applications easier.

We are also able to make custom phases for you. We have already anchored small molecules, peptides, sugars, and even enzymes for different customers. If your project would benefit from special silica-bound materials, contact us; we are up to the challenge! Finally, we also have a team of chemists that can screen your metal-contaminated products, and find the best conditions and the best scavenger for your needs. We can also find the right catalyst for you and determine the optimal conditions for you. Please contact us to get more information about this service.

I hope that you will enjoy using our products as much as we enjoyed developing them.
Happy chemistry,

François Béland, Ph.D., Chemist
vice-president, R&D

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Importance of Quality Control

The Quality Control Department's objective is to provide default-free products. In light of this goal, we have determined the critical points that need to be addressed for each product line. These points are based on customer's and Account Managers' recommendations as well as on our employees' scientific knowledge.

Each product family has its own quality control procedures, which are strictly adhered to. QC test results are checked and confirmed by the person in charge of them before being cleared for shipping. Complete procedures for each product line are available upon request.

Thus, SiliCycle is committed to high quality standards. In doing so, every product meets the quality specifications our customers demand. All products are shipped with a Certificate of Analysis (*CofA*) and a sample from every batch is kept for subsequent analysis. If you feel that the product you have received does not meet these specifications, please contact us and we will make sure you are satisfied.

Bare Silica Gel

The backbone of most of SiliCycle's products is Silia*Flash* F60 (*40-63* µm, *60 Å*) silica gel. It provides superior performance for chromatographic applications due to its narrow particle size distribution and high purity.

Before functionalization, every silica is rigorously characterized and analyzed by the procedures below to ensure lot-to-lot reproducibility.

Functionalized Silica Gel

The process for functionalizing the silica is highly dependent on the group being attached. However, it is still possible to functionalize 90% of the surface, verified by ^{29}Si MAS NMR. The remaining 10% of the surface may be endcapped to provide a completely inert support. After being functionalized, the product is submitted to further analysis and quality control as outlined below.

Quality Control

Type of Analysis	Performed by:
Bare Silica Gel	
Carbon, nitrogen & sulfur content	Elemental analyzer
Total trace metal	ICP-OES
Surface area & porosity	Nitrogen adsorption analyzer
Particle size distribution	Laser light diffraction
Tapped density analysis	Density measurement
Water content	Moisture balance
pH	pH-meter
Functionalized Silica Gel	
Residual solvent content	Moisture balance
Specific reactivity analysis	GC-FID, GC-MS, LC-MS/MS, ICP-OES
Organic function signature	Infrared spectroscopy
Purity analysis	GC-MS

Analysis Descriptions

Elemental Analysis of Organic Compounds

Silia*Flash* silica gel has a very low organic content. All lots are subjected to elemental analysis to determine the carbon, nitrogen and sulfur levels.

Total Trace Metal Analysis

To improve the quality of the separation, SiliCycle manufactures silica gels with very low traces of metal content. All silica gels are analyzed for more than 45 metals by ICP-OES down to ppm, and reach up to 99.4% silica purity. This removes any issues from metal oxides that may act as Lewis acids and prevents «Tailing» of most polar compounds (*frequently ionizable*) that can be caused by silica with a high metal content.

Surface Area and Porosity Analysis

The efficiency and reliability of silica gel depend on its surface condition. We use the Brunauer, Emmet, and, Teller analysis to determine the surface area, and the Barret-Joyner-Hatenda method to determine the pore diameter and pore volume. A larger surface area results in more contact or interaction with the analyte, thereby increasing the segregation of different products. Pore diameter and pore volume permit semi-exclusion chromatography where smaller molecules fit into pores more easily than larger ones. This justifies the use of several types of silica to achieve better discrimination in chromatographic separations.

Particle Size Distribution Analysis

Particle size distribution is determined by laser diffraction. Usually, more than 90% of the silica gel is kept within the appropriate range.

Water Content Analysis (*silica gel activity*)

The amount of water on the silica's surface affects chromatographic performance. An anhydrous silica gel will be extremely polar, while a wetted one will be considerably less polar. Every batch is carefully adjusted to a specific percentage of water content.

pH Analysis

The pH can increase the retention of some ionizable compounds. However, some products can become hydrolyzed or rearranged when in contact with acidic silica. A neutral pH, with a range between 6.5 and 7.5, is the most important factor in determining the reliability and inert behavior of the silica. This pH test involves suspending the silica gel in pure water (*5% w/w*).

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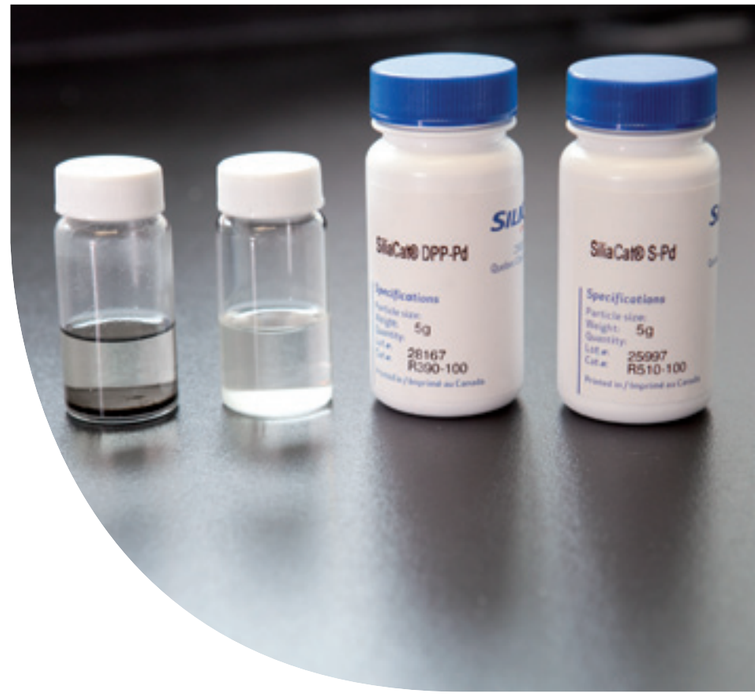
Drug Synthesis

SiliaCat®

Heterogeneous Catalysts



SILICYCLE Inc.
UltraPure SILICA GELS

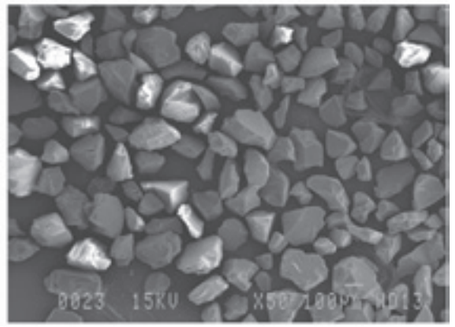
Catalytic Reactions with Silia*Cat*®

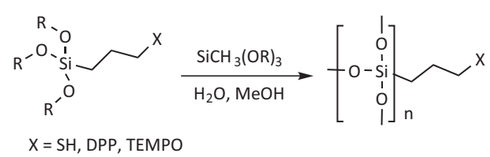
Advantages of using Silia*Cat*® heterogeneous catalysts over competitive products include:

- High stability
- Rigid & Porous Structure (*no swelling*)
- Compatibility with a wide range of solvents
- Ease of use: no swelling or static charge
- Leach-proof
- High turnover number (*TON*)
- Fast kinetics
- Accurate loading

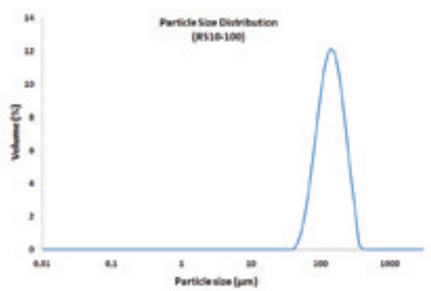
The Silia*Cat* Matrix

Inspired by the ORganically MOdified SILica (*ORMOSIL*) technology, the Silia*Cat* family is composed by new and innovative catalysts. Resulting from the co-condensation of two organosilane monomers by the sol-gel process (*confer condensation mechanism below*), the hybrid organic-inorganic materials present the highest stability and reactivity available with heterogeneous catalysts. Furthermore, the high cross linked framework presents an unmatched resistance, significantly better than the usual post-synthesis functionalized ligand.





X = SH, DPP, TEMPO

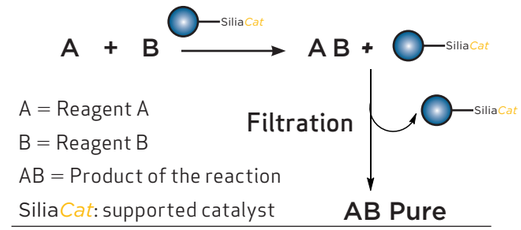


What are Silia*Cat* Heterogeneous Catalyst?

Usually, heterogeneous catalysts supported on a silica matrix are immobilized by post-modification of the inorganic support. These supports, however, present a high degree of leaching due to the poor stability of the immobilized phase. With Silia*Cat* Heterogeneous Catalysts, the ligand is directly cross linked in an organic-inorganic framework. This results a high degree of stability of the catalysts. Compared to homogeneous catalysts, Silia*Cat* exhibits a good reactivity and selectivity with one of the major advantages being that the catalyst is eliminated from the reaction mixture by a simple filtration. Forget your purification problems with our Silia*Cat* catalysts family.

The process for using Silia*Cat* Heterogeneous Catalysts is outlined in the scheme below.

What is Silia*Cat* Heterogeneous Catalyst?



Features and Benefits of Silia*Cat* Catalysts

Features & Benefits of Silia*Cat*

Features	Benefits
Inertness within entrapped molecules	High conversion and yield
Reagent concentrated at the surface of the material	Reliable and reproducible synthesis
Robustness	High thermal and mechanical stabilities
Rigid and porous structure	No swelling, solvent independent and air stable Conditions do not have to be inert
Leach-proof organoceramic matrix	No contamination of APIs
High and accurate catalyst loading	Less catalyst required over competitive products
High turnover number (*TON*)	Catalytic amount (*< 1 mol %*)
Reusability	Multi-uses possible
Ease of handling and purification	Free flowing, no static charge Easily removed by simple filtration
Ease of scalability	Scalable from mg up to multi-ton scale
Flexible formats	Amenable to use in Silia*Sep* & Silia*Prep* Cartridges
Available in bulk quantities	Can be delivered in large quantities and always in stock

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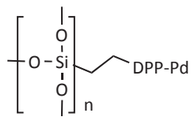
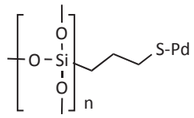
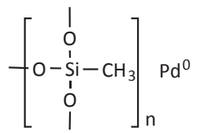
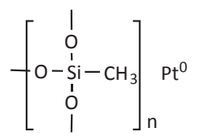
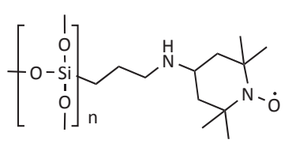
SiliaCat Heterogeneous Catalysts Product Range

SiliCycle, a leader in functionalized silica gels, has developed various catalysts at competitive prices.

SiliaCat Heterogeneous Catalysts Portfolio* **SiliaCat Heterogeneous Catalysts Portfolio**

SiliaCat Name	Product Number	Structure	Brief Description	Typical Applications	SiliaCat Typical Characteristics: Color	Endcapping	Molecular Loading	Typical Tap Density	Solvent Compatibility	Prolonged Storage	SiliaCat Name
SiliaCat DPP-Pd	R390-100		The significant costs associated with precious metal catalysts and their tendency to remain in organic products has generated interest for solutions that increase reactivity and can enable the recovery and reuse of these metals. SiliaCat DPP-Pd is a unique diphenylphosphine palladium (II) heterogeneous catalyst made from a leach-resistant organoceramic matrix.	Suzuki, Heck Sonogashira, Kumada, Stille	Yellow	Yes	≥ 0.2 mmol/g	415 g/L	All solvents, aqueous and organic	Keep dry	SiliaCat DPP-Pd
SiliaCat S-Pd	R510-100		The significant costs associated with precious metal catalysts and their tendency to remain in organic products has generated interest for solutions that increase reactivity and can enable the recovery and reuse of these metals. SiliaCat S-Pd is a unique thiol-based palladium (II) heterogeneous catalysts made from a leach-resistant organoceramic matrix.	Suzuki, Heck Sonogashira, Kumada, Stille	Red - Orange	Yes	≥ 0.3 mmol/g	550 g/L	All organic solvents	Keep dry	SiliaCat S-Pd
NEW PRODUCT SiliaCat Pd⁰	R815-100		SiliaCat Pd⁰ is a new series of patent-protected sol–gel-entrapped Pd nanocatalysts. It is made from highly dispersed Pd nanoparticles (*uniformly in the range 4.0–6.0 nm*) encapsulated within an organosilica matrix.	Suzuki, Heck Sonogashira, Kumada, Stille, Selective debenzylation, Selective hydrogenation	Dark brown to black	Yes	-	-	All solvents, aqueous and organic	Keep cool (< 8 °C) Under Argon	SiliaCat Pd⁰
NEW PRODUCT SiliaCat Pt⁰	R820-100		SiliaCat Pt⁰ is made of organosilica physically doped with nanostructured platinum (0), and is both stable and efficient. This catalyst was successfully prepared by a novel and simple sol-gel route. In the new procedure, Pt nanoparticles (*uniformly in the range 1.7–3.15 nm*) are encapsulated via an alcohol-free sol-gel process typical of enzyme sol-gel encapsulation.	Selective reduction of nitroarenes, Hydrosilylation	Dark brown to black	Yes	-	-	All solvents, aqueous and organic	Keep dry Under Argon	SiliaCat Pt⁰
SiliaCat TEMPO	R723-100		SiliaCat TEMPO is a new oxidizing catalyst made from a proprietary class of organosilica-entrapped radicals. This encapsulation process confers enhanced reactivity and properties. The leach-resistant organoceramic matrix makes SiliaCat TEMPO highly efficient and selective compared to homogeneous TEMPO reagents. It also has a superior performance compared to polymer-supported TEMPO and silica-supported TEMPO in terms of both selectivity and stability. With SiliaCat TEMPO, no activation is required prior to use and selective aldehyde vs acid oxidation is possible. *U.S.Patent: 6,797,773 B1,2004*	Oxidation of alcohols or Aldehydes	Orange	Yes	≥ 0.70 mmol/g	639 g/L	All solvents, aqueous and organic	Keep dry	SiliaCat TEMPO

Formats: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 5kg, 10kg, 25kg, ...

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Catalyst Screening Service

CONFIDENTIALITY ASSURED

Looking for the right Silia*Cat* Heterogeneous Catalyst to use? Contact us to take advantage of SiliCycle's expertise in catalysis. Our R&D team can find the optimal conditions for you.

Our Catalyst Screening Service is innovative because it provides a turn key solution to the pharmaceutical and manufacturing industries. Working with the substrates you identify, SiliCycle's chemists will quickly develop the most efficient catalysis process (*which catalyst and solvent to use, optimal concentrations, etc*).

We guarantee confidentiality, since in most cases our screening service requires us to work with APIs or other patented materials. This will ensure an easy technology transfer.

Make the call many major pharmaceutical companies have already made. Contact us to discuss how we can help you to reach your goals. Many screening services are available to fit your needs and budget.

Silia*Cat* - Regulatory Information

Silia*Cat* Heterogeneous Catalysts are being used more and more by GMP pharmaceutical, biotechnology, and fine chemical industries as well as contract research and manufacturing organizations. Many have run their own analysis proving Silia*Cat* can safely be used without compromising the purity of their compounds due to leaching.

Need specific regulatory files? SiliCycle can work with you to fulfill your requirements. We can provide custom regulatory documentations that include specific analytical tests in line with your needs.

SiliCycle is committed to high quality standards and strives to provide default-free products. In doing so, all products are manufactured in an ISO 9001:2008 compliant facility and submitted to a stringent quality control. Every lot must meet the quality specifications to be released for sale and a sample from every batch is kept for subsequent analysis. All products are shipped with the following information:

- Certificate of Analysis
  - Purity (*Leachables and extractables*)
  - Molecular loading
  - Surface Coverage
  - Volatile Content
- Material Safety Data Sheets (*MSDS*)
- BSE/TSE Declaration (*no animal origin*)
- Relevant Technical Information

Experimental Procedures and Optimization

Typical experimental procedures can be found for each catalytic reaction. Please note that these procedures are the starting suggestions meant to be starting points. Sometimes, optimization steps need to be undertaken to optimize yields and increase selectivity. Various parameters can be changed, one at a time or simultaneously, to improve results.

Number of mol % of Silia*Cat* Catalysts

For each new experiment, we suggest using a molar percent of Silia*Cat* with respect to the substrate. This quantity has to then be optimized in order to obtain a good catalytic activity with the lowest consumption of the Silia*Cat*. For initial experiments we suggest to use an higher mol % of Silia*Cat* Catalyst in respect to the substrate and then decrease the quantity if yield and kinetics are already in line with your needs. During development applications work at SiliCycle, we always start using 1 mol % of catalyst.

Solvent

Silia*Cat* can safely be used in a wide range of organic and aqueous solvents commonly used in laboratory and in process work, such as DMF, DMSO, THF, ACN, alcohols, ethers, chlorinated solvents, water, etc. The nature of the solvent does sometimes influence the catalytic efficiency, however. If yield is low or kinetics are too slow, changing solvent or adding a co-solvent should be considered.

Solution Concentration

At low substrate concentration, the activity of the catalyst will be directly proportional to the number of moles of substrate available. If you increase the concentration of the substrate, the activity will increase until the active sites become saturated. So the substrate concentration is a parameter that needs to be optimized to develop your catalytic conditions.

Temperature

A catalyst's purpose is to enhance the kinetics of a reaction, so we recommend running the experiments at room temperature. In the optimization step, the temperature could be adjusted, if it is needed.

Reaction Time

In the case that the TOF is low, and increasing the temperature to increase the kinetics is not possible, we recommend increasing the contact time with the catalyst to complete the reaction. Also, in this case, increasing of the amount of catalyst is an option.

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Silia*Cat*'s Compatibility with New Technologies

Silia*Cat* In Flow Chemistry and Microwave Assisted Experiments

Silia*Cat* can also be used in flow chemistry and under microwave radiation. In flow chemistry, simply place the Silia*Cat* inside the solid-phase reactor included in the flow system (*i.e. Syrris Asia® Solid Phase Chemistry Reactor*) and run the reaction. See page 78 for more details.

In microwave experiments, Silia*Cat* showed excellent catalytic efficiency in a short period of time. See following pages for the different applications developed.

Catalysis Definitions and Calculation

Silia*Cat* Heterogeneous Catalysts are sol-gel silica-supported catalysts that can be used to replace homogeneous catalysts. The process for using Silia*Cat* is outlined in the scheme page 21.

What is a Turnover Number (TON)?

In catalysis, the term turnover number has two meanings: the number of moles of substrate that a mole of catalyst can convert before becoming inactivated and is the amount of substrate converted per the amount of catalyst used.

In theory, the Ideal catalyst would have an infinite turnover number and would never be consumed. In practice, turnover numbers begin at 100 and can go up to a million, more so in some cases.

What is a Turnover Frequency (TOF)?

A catalyst's turnover frequency number, or turnover number per time unit, characterizes its level of activity. So the TOF is the total number of moles transformed into the desired product by one mole of active site per hour. The larger the TOF, the more active the catalyst.

TOF = TON/hour

How to Calculate the Amount of Silia*Cat* Needed Based on Mol %?

One mol % (1 mol %) means 0.01 molar equivalent. If 3 mmol of the substrate is used, then 0.03 mmol of Silia*Cat* catalyst is required. To determine the weight of the catalyst needed, simply divide this value by the loading of the catalyst. For example, Silia*Cat* DPP-Pd typical loading is 0.2 mmol/g, so 0.15 g is needed.

Solvent Molar Concentration

A 1.2 M solvent concentration means:

1.2 mmol of substrate per mL of solvent
(*or 1.2 mol of substrate per L of solvent*)

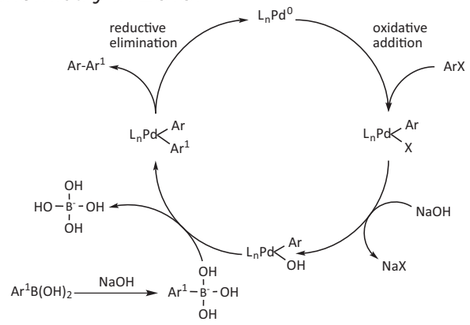
Volume of solvent needed = $\frac{\text{mmol of substrate used}}{\text{molar concentration desired}}$

For example, if 3 mmol of the substrate is used, then, 2.5 mL of solvent is necessary to reach a 1.2 M concentration.

Suzuki Coupling Using Pd-based Silia*Cat*

The Suzuki coupling (*also called Suzuki-Miyaura reaction*) is the reaction between a boronic acid and a halide catalyzed by a palladium (0) catalyst widely used in organic synthesis. At first, only aryl and vinyl substrates could undergo Suzuki coupling. Now, catalysts are becoming so powerful that the substrate scope has broadened to include: alkyl-, alkenyl- & alkynyl- halides, triflates and organoboranes, trifluoroborates or borate esters.

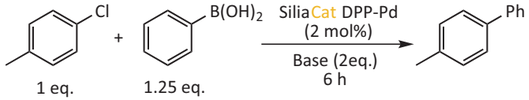
Its discovery was awarded the Nobel Prize in Chemistry in 2010.



Solvent and Base Effects

The choice of solvent and base play an important part in the Suzuki reaction. Different solvents and bases were tested to find the most suitable combination. Total conversion was obtained in both ethanol and propanol. With THF, dioxane, toluene and DMF, the kinetics were lower.

For the base, potassium carbonate (K_2CO_3) is the best. However, in some cases, Na_2CO_3 and NaOAc can also be used.



Solvent and Base Effects

Solvent	Temp. (°C)	Conversion / Selectivity (%)					
		K_2CO_3	Na_2CO_3	KOAc	NaOAc	K_2HPO_4	Et_3N
MeOH	64	74 / 95	69 / 99	63 / 98	63 / 98	73 / 100	72 / 93
EtOH	77	100 / 98	100 / 97	82 / 99	85 / 100	79 / 100	77 / 93
EtOH/H_2O (15%)	77	100 / 100	82 / 100	78 / 100	88 / 100	86 / 98	89 / 95
1-PrOH	90	100 / 95	70 / 97	90 / 99	91 / 99	15 / 100	20 / 95
2-PrOH	77	100 / 100	43 / 93	90 / 99	72 / 100	50 / 100	20 / 100
THF	64	30 / 93	15 / -	45 / 89	35 / 94	37 / 95	5 / -
MeTHF	77	40 / 95	33 / 100	39 / 100	56 / 100	30 / 97	4 / -
Dioxane	90	50 / 90	30 / 93	56 / 93	35 / 94	20 / 90	No reaction
Toluene	90	47 / 98	23 / 87	49 / 96	10 / 90	65 / 95	No reaction
DMF	90	50 / 100	30 / 100	15 / 100	17 / 100	7 / 100	No reaction

Catalyst Concentration Effect

Decreasing the mol % of the catalyst lowers the kinetics of the reaction, but the total conversion can still be achieved. In this example, the addition of water significantly improves catalyst activity. Even if the catalyst amount is divided by 10, the TOF is still increased by a factor of five.

(*Cdns: Silia*Cat* DPP-Pd, $PhB(OH)_2$ (1.1 eq.), K_2CO_3 (1.5 eq.) RT*).

Catalyst Concentration Effect

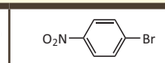
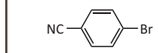
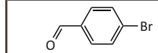
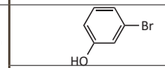
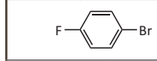
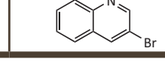
mol %	Solvent (M)	Time (h)	Conv. (%)	TON	TOF
0.2	EtOH (0.05)	0.5	100	500	1,000
0.1	EtOH (0.05)	1	100	1,000	1,000
0.01	EtOH/H_2O (0.08)	2	100	10,000	5,000
0.002	EtOH/H_2O (0.08)	16	100	50,000	3,125

Pd-Based Silia*Cat*'s Catalytic Performance Comparison and Reusability

All Silia*Cat* Pd-based catalysts can be used for Suzuki coupling. The table below presents the best conditions for bromo- substrates. It can be seen that even with half the catalyst amount, Silia*Cat* Pd^0 is the more active catalyst.

For substrates with electron-withdrawing groups, Silia*Cat* catalysts can be reused more than 5 times with a minimal loss of activity and leaching. For substrates containing an electron-donating group, Silia*Cat* catalysts can be used up to 3 times with only a small effect on activity.

Pd-Based Silia*Cat*'s Catalytic Performance Comparison and Reusability

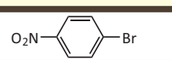
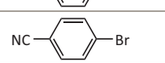
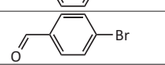
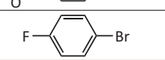
	Substrate (R)	Silia*Cat* Performance Comparison [Conversion / Selectivity (%)]			Reusability [Conversion / Selectivity (%)] Pd & Si Leaching (ppm)[1]			
		DPP-Pd (1 mol %)[a-b]	S-Pd (1 mol %)[c]	Pd^0 (0.5 mol %)[d]	Run 2	Run 3	Run 4	Run 5
Electron-Withdrawing		100 / 100 Pd: 0.1, Si: 2	100 / 99	100 / 99	100 / 100 Pd: 0.05, Si: 1	100 / 100 Pd: 0.08, Si: 1.5	100 / 100 Pd: 0.1, Si: 3	99 / 98 Pd: 0.1, Si: 3.5
		100 / 97 Pd: 0.1, Si: 3	100 / 99	99 / 97	98 / 99 Pd: 0.1, Si: 8	98 / 99 Pd: 0.07, Si: 5	100 / 99 Pd: 0.1, Si: 6	99 / 98 Pd: 0.1, Si: 5
		100 / 97 Pd: 0.1, Si: 6	94 /88	95 / 98	99 / 90 Pd: 0.2, Si: 7	97 / 92 Pd: 0.2, Si: 8	99 / 98 Pd: 0.1, Si: 4	98 / 97 Pd: 0.1, Si: 5
Electron-Donating		100 / 99 Pd: 0.9, Si: 5	82 / 100	83 / 100	100 / 100 Pd: 0.6, Si: 9	100 / 98 Pd: 0.4, Si: 7	60 / 97 Pd: 0.05, Si: 6	-
		100 / 80 Pd: 0.07, Si: 3	94 / 100	98 / 99	99 / 99 Pd: 0.04, Si: 1.5	98 / 98 Pd: 0.1, Si: 2	81 / 94 Pd: 0.06, Si: 2	73 / 95 Pd: 0.03, Si: 7
		100 / 99 Pd: 2.1, Si: 10	72 / 95	97 / 95	88 / 90 Pd: 0.3, Si: 7	75 / 95 Pd: 4, Si: 9	87 / 99 Pd: 0.6, Si: 10	68 / 96 Pd: 04, Si: 11

[a] Corresponds to "Run 1" in the reusability study.
General exp. cond.: 1 eq. substrate, 1.2 eq. $PhB(OH)_2$, 2 eq. K_2CO_3; [b] MeOH (0.1 M), 2 h, 65°C; [c] $EtOH/H_2O$ (0.12 M) 4h, 77°C; [d] EtOH (0.12 M) 2h, 77°C.

[1] Using Silia*Cat* DPP-Pd as catalyst under the same conditions previously described. Run #1 is the result presented in the performance comparison section of the table.

The performance of the Silia*Cat* DPP-Pd and S-Pd catalysts for Suzuki coupling was also compared in microwave assisted experiments for brominated substrates. Both products exhibit a very high performance in microwave experiments. After only 5 minutes, 100% of the product is obtained in most experiments. Both also present high selectivity, with yields nearly reaching 100%. The products were also tested for chlorinated substrates as presented on the following page.

Catalytic Performance in Microwave

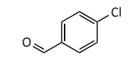
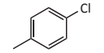
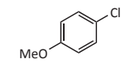
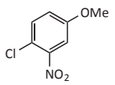
Substrate (R)	Conversion (%) / Yield (%)	
	Silia*Cat* DPP-Pd (0.5 mol %)[a-b]	Silia*Cat* S-Pd (0.5 mol %)[c]
	100 / 99.5	100 / 99.3
	100 / 99.4	100 / -
	100 / 88	100 / -
	98 / 97.3	72 / -

General exp. cond.: 1 eq. substrate, 1.1 eq. $PhB(OH)_2$, 1.5 eq. K_2CO_3; [a] MeOH (0.2 M), 5 min, 75°C, 150 W, 150 psi; [b] MeOH (0.2 M), 5 min, 75°C, 200 W, 200 psi, [c] 15 min.

Pd-based Silia*Cat*'s Catalytic Performance Comparison (con't)

The Silia*Cat* Pd-based catalysts can also be used for Suzuki coupling with chlorinated substrates in both conventional and microwave conditions. We have chosen to do this study with Silia*Cat* DPP-Pd.

Catalytic Performance of Chlorinated Substrates

Substrate (R)	Conversion /Yield (%)[a]	Substrate (R)	Conversion /Yield (%)[a]
	98 / 93		99 / -
	98 / 96		Bulk: 100 / 98 MW[b]: 100 / 95

[a]Exp. cond. in bulk: 1.5 mol % of Silia*Cat*, 1 eq. substrate, 1.5 eq. $PhB(OH)_2$, 2 eq. K_2CO_3, $EtOH/H_2O$ 15% (0.12M), 6 h, reflux. [b] Microwave: 1 mol % of Silia*Cat*, 15 min, 125°C.

Conclusion for Suzuki Coupling

In conclusion, Silia*Cat*, can be used successfuly for Suzuki coupling reactions with iodide, bromide and chloride aryl substrates in conventional or in microwaves conditions. The Silia*Cat* DPP-Pd gives better performance versus the Silia*Cat* S-Pd and nearly equivalent to the Silia*Cat* Pd^0 for the substrates presented.

Suzuki Coupling Typical Experimental Condition

Conventional Experimental Conditions

Reaction

All products except the catalyst are added to a round bottom flask equipped with a condenser and a magnetic stirrer. Bring mixture to reflux and, after 10 minutes (*when the solution is homogeneous*), add the required quantity of catalyst.

Work-up

Once the reaction is complete (*determined by TLC or GC-MS*), the catalyst is filtered at room temperature, rinsed twice with water and three times with the solvent used in the reaction, and finally dried and stored for future use. The reaction mixture obtained after filtration of the catalyst is evaporated, and the product is extracted using ethyl acetate (AcOEt) or diethyl ether (Et_2O) and washed twice with water. The organic phase is dried using magnesium sulfate ($MgSO_4$), and filtered, and the solvent is evaporated. The crude mixture is purified using flash chromatography, if needed. Also applicable to microwave conditions.

Microwave Conditions

Reaction

All products are added to a microwave tube equipped with a magnetic stirrer. Set microwave conditions to:

- Power: 150 W
- Pressure: 150 psi
- Temperature: 75 - 150°C
- Reaction Time: 5 - 15 min

Suzuki Coupling Typical Experimental Conditions

Products	Conventional Conditions			Microwave Conditions		
	Ar-Iodide	Ar-Bromide	Ar-Chloride	Ar-Iodide	Ar-Bromide	Ar-Chloride
Base [K_2CO_3]	1.5 eq.	1.5 eq.	2.0 eq.	1.5 eq.	1.5 eq.	2.0 eq.
Boronic Acid	1.2 eq.	1.2 eq.	1.5 eq.	1.2 eq.	1.2 eq.	1.5 eq.
Silia*Cat* Catalyst	≥ 0.5 mol %	≥ 0.5 mol %	≥ 1.0 mol %	≥ 0.5 mol %	≥ 0.5 mol %	≥ 1.0 mol %
Best Solvents (HPLC Grade)	MeOH (0.05 - 0.1 M)	$EtOH/H_2O$ (10:1, 0.12 M)	EtOH or TBA/H_2O (10:1.5, 0.12 M)	MeOH (0.2 M)	MeOH (0.2 M)	$EtOH/H_2O$ (10:1, 0.2 M)

***Note: molar concentration is related to the substrate.**

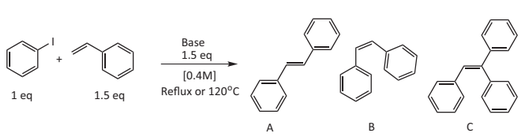
Heck Coupling Using Silia*Cat* DPP-Pd & S-Pd

The Heck reaction, also known as the Mizoroki-Heck reaction, is the coupling of a halide with an alkene in the presence of a base and a palladium catalyst. This coupling allows a substitution reaction on alkenes and is of great importance in pharmaceutical research. We determined that the best catalyst for this reaction is Silia*Cat* DPP-Pd. It showed good reactivity for aryl iodides, bromides and chlorides.

Note: Silia*Cat* Pd^0 results were not available at the time of printing. Contact us for details.

Base and Solvent Effects

The Heck coupling between iodobenzene and styrene was used to evaluate the influence of solvent and base. The best combinations are KOAc/DMF, Et_3N/MeCN and Pr_3N/neat. Using these systems, high yields and great selectivity in favor of product A were obtained.

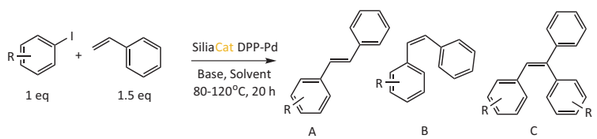


Base and Solvent Effects (Silia*Cat* DPP-Pd)

Silia*Cat* (mol %)	Base	Solvent (0.4 M)	Time (h)	Conversion A / B / C (%)
0.5	KOAc	DMF	24	100 (90 / 9.5 / 0.5)
0.5	Na_2CO_3	DMF	24	67 (62 / 47 / 0)
0.1	Et_3N	MeCN	24	93 (77 / 6 / 11)
0.1	Et_3N	H_2O	24	75 (70 / 5 / 0)
0.1	Pr_3N	(neat)	20	100 (95 / 5 / 0)

Catalytic Performance and Comparison vs Homogeneous Catalyst

Silia*Cat* DPP-Pd is a very efficient catalyst for the Heck coupling and allows greater selectivity over homogeneous Pd catalyst (*TPP is required*). In addition to higher yield of the desired product, the catalyst left minimal residual Pd, TPP or TPPO in solution that would have otherwise required the use of a metal scavenger, chromatography or trituration to remove.

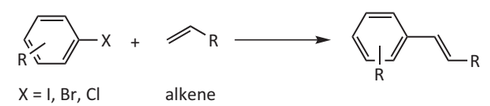


Catalytic Performance and Comparaison vs Homogeneous

Substrate R	Substrate X	Silia*Cat* DPP-Pd (mol %)	Base	Solvent (0.4 M)	Conversion A / B / C (%)	Phosphine Leaching (ppm)
4-CN	Br	0.25	NaOAc	DMF	100 (95 / 5 / -)	-
4-NO_2	Br	0.25	NaOAc	DMF	99 (97 / 2 / -)	-
2-CH_3	Br	0.25	Et_3N	MeCN	71 (67 / 5 / -)	-
4-OMe	I	0.25	Et_3N	MeCN	75 (60 / 15 / -)	-
H	I	0.1	Et_3N	MeCN	100 (98 / 2 / -)	0
H	I	1.0 $Pd(OAc)_2$ PPh_3	Et_3N	MeCN	100 (70 / 22 / 8)	6,030

Substrate Scope, Leaching and Microwave Compatibility

Silia*Cat* catalysts are efficient in the Heck coupling with different substrates. In all cases, conversion and selectivities were excellent. Leaching results were all below FDA regulations, and no further metal removal was needed. Microwave technology allows faster kinetics with good yields.



X = I, Br, Cl alkene

Substrate Scope, Leaching and Microwave (MW) Compatibility

Rn	Mode	Silia*Cat* DPP-Pd mol %	Time	Temp.	Conv./Sel.(%)	Leaching (ppm)	Silia*Cat* S-Pd mol %	Time	Temp.	Conv./Sel.(%)	Leaching (ppm)
1	Batch	0.5	24 h	120°C	100 / 97	-	0.5	24 h	120°C	98 / 92	-
	MW	0.2	10 m	125°C	93 / 85	P: 0.3, Pd: 0.02, Si: 0.8	0.2	15 m	125°C	97 / 93	Pd: 3.8, Si: 1.9
2	Batch	0.2	24 h	135°C	100 / 98	-	0.25	24 h	120°C	85 / 75	-
	MW	0.2 0.5[1]	10 m 30 m[1]	125°C 150°C[1]	92 / 81 99 / 93[1]	- P: 0.7, Pd: 0.02, Si: 1.6	0.2	15 m	125°C	87 / 76	Pd: 0.3, Si: 0.8

[1] Et_3N in water

Heck Coupling Typical Experimental Procedure

Conventional Experimental Conditions

Reaction

All products except the catalyst are added to a round bottom flask equipped with a condenser and a magnetic stirrer. Bring mixture to reflux (MeCN) or to 120°C (DMF or NMP) and after 10 minutes (*when solution is homogeneous*) add the desired quantity of catalyst.

Work-up

Once the reaction is complete (*determined by TLC or GC-MS*), follow the same work-up procedure as for Suzuki coupling conventional experimental conditions as they are applicable to microwave conditions.

Microwave Conditions

Reaction

All products are added to a microwave tube equipped with a magnetic stirrer. Set microwave conditions to:

- Power: 100 W (I-) or 200 W (Br-, Cl-)
- Pressure: 150 psi (I-) or 200 psi (Br-, Cl-)
- Temperature: 100°C (I-) or 125°C (Br-, Cl-)
- Reaction Time: 10 min (I-) or 15 min (Br-, Cl-)

Heck Coupling Typical Experimental Conditions

Products	Conventional Conditions for 1 eq of: Ar-Iodide	Ar-Bromide	Ar-Chloride	Microwave Conditions for 1 eq of: Ar-Iodide	Ar-Bromide	Ar-Chloride
Base	1.5 eq. [Et_3N or NaOAc]	1.5 eq. [NaOAc]	1.5/0.5 eq. [$Ca(OH)_2$/TBAB]	1.5 eq. [Et_3N or NaOAc]	1.5 eq. [K_2CO_3]	2.0 eq. [K_2CO_3]
Olefin	1.2 eq.	1.2 - 2.0 eq.	1.2 - 2.0 eq.	1.2 - 2.0 eq.	1.2 - 2.0 eq.	1.2 - 2.0 eq.
Silia*Cat* Catalyst	≥ 0.5 mol %	≥ 0.5 mol %	≥ 1.0 mol %	≥ 0.2 mol %	≥ 0.2 mol %	≥ 1.0 mol %
Best Solvents (HPLC Grade)	MeCN (1.2 M) DMF (0.75 M)	DMF (0.75 - 1.5 M)	NMP/H_2O (1:1, 1.67 M)	MeOH (0.2 M)	MeOH (0.2 M)	EtOH/H_2O (10:1, 0.2 M)

*Note: molar concentration is related to the substrate.

Sonogashira Coupling Using Silia*Cat* Catalysts

The Sonogashira coupling reaction of aryl halides and terminal acetylenes is an effective method for the formation of substituted acetylenes. This reaction is frequently utilized as a key step in natural product chemistry and for the synthesis of acetylene compounds, which have several applications.

Catalyst Concentration and Solvent Effects

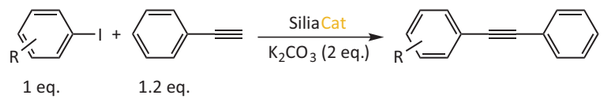
Sonogashira coupling between iodonitrobenzene and phenylacetylene was achieved easily and without the need for co-catalysts to activate the alkyne, making the use of Silia*Cat* an efficient method for the formation of substituted acetylenes. All catalysts screened presented excellent efficiency, even in low amounts.

Catalyst Concentration and Solvent Effects

Silia*Cat* DPP-Pd					Silia*Cat* S-Pd					Silia*Cat* Pd^0				
mol %	Solvent (M)	Time (min)	Conv. (%)	TON (TOF)	mol %	Solvent (M)	Time (min)	Conv. (%)	TON (TOF)	mol %	Solvent (M)	Time (min)	Conv. (%)	TON (TOF)
0.5	EtOH/H_2O (0.07)	30	100	200 (400)	0.5	EtOH/H_2O (0.07)	5	100	200 (2,500)	0.1	EtOH (0.1)	2 h	100	1,000 (500)
0.5	MeOH/H_2O (0.07)	5	100	200 (2500)	0.5	MeOH/H_2O (0.07)	1 h	100	200 (200)	0.1	EtOH (0.05)	30	100	1,000 (2,000)
0.1	EtOH/H_2O (0.07)	1 h	100	1,000 (1,000)	0.1	EtOH/H_2O (0.07)	1 h	100	1,000 (1,000)					
0.1	MeOH/H_2O (0.07)	15	100	1,000 (4,000)										
0.01	EtOH/H_2O (0.13)	3 h	100	10,000 (4,000)										
0.002	EtOH/H_2O (0.13)	8 h	100	50,000										

Iodo-Substrate Scope and Microwave Compatibility

Sonogashira couplings between iodoaryls and phenylacetylene are achieved with ease and without the need for co-catalysts to activate the alkyne.This shows that Silia*Cat* is an efficient tool for the formation of substituted acetylenes.



1 eq. 1.2 eq.

Iodo- Substrate Scope and Microwave Compatibility

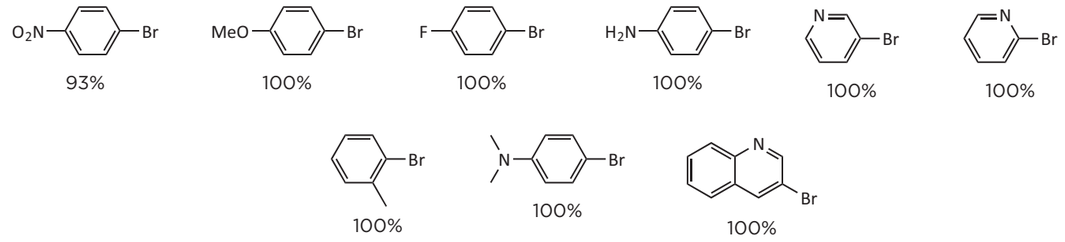
R	Mode	Silia*Cat* DPP-Pd			Silia*Cat* S-Pd			Silia*Cat* Pd^0		
		mol %	Conditions	Conv. / Sel. (%)	mol %	Conditions	Conv. / Sel. (%)	mol %	Conditions	Conv. / Sel.(%)
4-NO_2	Batch	1	EtOH (0.08 M) 77°C, 4 h	100 / 100	1	EtOH (0.08 M) 77°C, 4 h	100 / 100	1	EtOH (0.08 M) 77°C, 4 h	100 / 100
	MW	0.6	MeOH/H_2O (0.2 M) 100°C, 2 min	100 / -	0.6	MeOH/H_2O (0.2 M) 100°C, 2 min	100 / -	0.1	MeOH (0.1 M) 75°C, 5 min	100 / -
4-OMe	Batch	1	EtOH (0.08 M) 77°C, 4 h	99 / 98	1	EtOH (0.08 M) 77°C, 4 h	100 / 100	1	EtOH (0.08 M) 77°C, 4 h	99 / 98
4-CH_3	Batch	1	EtOH (0.08 M) 77°C, 4 h	100 / 100	1	EtOH (0.08 M) 77°C, 4 h	100 / 99	1	EtOH (0.08 M) 77°C, 4 h	100 / 100
	MW	0.5	MeOH/H_2O (0.2 M) 100°C, 2 min	90 / -	1	MeOH/H_2O (0.2 M) 100°C, 2 min	88 / -	0.1	MeOH (0.2 M) 75°C, 5 min	100 / -

Bromo- Substrate Scope and Microwave Compatibility

Silia*Cat* DPP-Pd and Pd^0 are also efficient catalysts for use with bromo substrates. A few examples of the Sonogashira coupling between various bromoaryls substrates (1 eq.) and phenylacetylene (1.25 eq.) using K_2CO_3 (2 eq.) in MeOH (0.2 M) are shown below.

Conversions obtained with 1 mol % of Silia*Cat* DPP-Pd under microwave irradiation are presented below. Conventional methodology is also possible, but kinetics are significantly lower (*a few hours compared to 15 minutes*).

Bromo- Substrate Scope Conversion (%) Results using Silia*Cat* DPP-Pd



Sonogashira Coupling Typical Experimental Procedure

Conventional Experimental Conditions

Reaction

All products except the catalyst are added to a round bottom flask equipped with a condenser and a magnetic stirrer. Bring mixture to reflux, and after 10 minutes (*when the solution is homogeneous*) add the required quantity of catalyst.

Work-up

Once the reaction is complete (*determined by TLC or GC-MS*), follow the same work-up procedure as for Suzuki coupling conventional experimental conditions as they are applicable to microwave conditions.

Microwave Conditions

Reaction

All products are added to a microwave tube equipped with a magnetic stirrer. Set microwave conditions to:

- Power: 150 W (I-) or 200 W (Br-)
- Pressure: 150 psi (I-) or 200 psi (Br-)
- Temperature: 100°C (I-) or 100 - 150°C (Br-)
- Reaction Time: 5 - 15 min (I-) or 5 - 20 min (Br-)

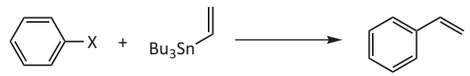
Sonogashira Coupling Typical Experimental Conditions

Products	Standard Conditions for 1 eq of:		Microwave Conditions for 1 eq of:	
	Ar-Iodide	Ar-Bromide	Ar-Iodide	Ar-Bromide
Base [K_2CO_3]	1.5 eq.	1.5 eq.	1.5 eq.	2.0 eq.
Alkyne	1.1 eq.	1.2 5 eq.	1.10 eq.	1.5 eq.
Silia*Cat* Catalyst	≥ 0.5 mol %	≥ 1.0 mol %	≥ 0.5 mol %	≥ 1.0 mol %
Best Solvents (HPLC Grade)	For room temperature reaction: MeOH (0.02 M) For reflux reaction: MeOH (0.05 - 0.13 M, typ.: 0.07 M) or EtOH/H_2O (10:1, 0.1 M)		MeOH (0.2M)	MeOH/H_2O (10:1, 0.2 M)

*Note: molar concentration is related to the substrate.

Stille Coupling Using Silia*Cat* Pd Catalysts

The Stille coupling is a versatile reaction for C-C bond formation. It is a coupling between a halide and an organotin compound. This reaction is widely used in synthesis, but a major drawback is the toxicity of the tin compounds involved. In Stille couplings, a Pd^0 or Pd^{II} catalyst is required, and it must be compatible with a wide variety of functional groups (*very few limitation on the R-group*). SiliCycle has developed catalysts that are highly efficient for Stille couplings, as shown below.



Note: Silia*Cat* Pd^0 results were not available at the time of printing.

Catalyst Concentration and Solvent Effects

Increasing the amount of the catalyst, for the same solvent and at a constant substrate concentration, improves kinetics (*see table below*). With a mol % of 0.25, the reaction was not completed in 22 h. With a mol % of 2.0, the reaction was completed in 17 h.

As a general rule, if the solvent and the concentration of the substrate are kept constant, increasing the amount of the catalyst, thus increasing the member of the active sites, will speed up the kinetics of the reaction.

This table also shows the importance of the solvent. At low catalyst concentration, 0.25 mol % in dioxane, the reaction was not completed in 22 h. However, under the same conditions but with toluene as the solvent, the reaction was completed in 16 h. In dioxane, the same activity is observed for a concentration of 2.0 mol %. The solvent is responsible for diffusion of the substrate to the active sites, so the better the diffusion, the higher the kinetics will be.

In all experiments, determining the optimal quantity of Silia*Cat* in respect to the solvent should be done.

Catalyst Concentration and Solvent Effects

Silia*Cat* DPP-Pd (mol %)	Solvent (M)	Time (h)	Conversion (%)
2.0	Dioxane (0.1 M)	17	99
0.5	Dioxane (0.1 M)	20	100
0.25	Dioxane (0.1 M)	22	74
0.25	Toluene (0.1 M)	16	99

Silia*Cat* DPP-Pd Reusability and Leaching

The minimal leaching and the robustness of the organoceramic matrix are important factors that allow Silia*Cat* DPP-Pd to be reused several times.

Silia*Cat* DPP-Pd Reusability and Leaching

Reusability	Conversion (%)	Pd Leaching (ppm)
1st	100	3.0
2nd	100	1.7
3rd	100	2.3
4th	100	2.3

Catalytic Activity and Additive CsF Influence

Reactions were performed at reflux until the GC/MS analysis showed maximum conversion. Anhydrous conditions are not required.

Catalytic Activity and Additive CsF Influence

Substrate (R)	Halide (X)	Silia*Cat* DPP-Pd (mol %)	Additive (eq.)	Solvent (M)	Time (h)	Conversion (%)
4-CN	Br	2	-	Dioxane (0.1 M)	18	87
4-F	Br	10	-	Dioxane (0.1 M)	24	99
4-F	Br	10	CsF (2)	Toluene (0.1 M)	24	100
H	Br	10	-	Toluene (0.1 M)	24	100
4-CH_3	Br	10	CsF (2)	Dioxane (0.1 M)	24	100
4-OCH_3	Br	10	CsF (2)	Dioxane (0.1 M)	24	100
H	I	10	CsF (2)	Toluene (0.1 M)	24	100
4-NO_2	I	2	-	Dioxane (0.1 M)	18	88

Note: R'$SnBu_3$ was vinyl (1.1 eq.)

Silia*Cat* DPP-Pd vs Competitive Catalysts

Comparative analysis with other Pd catalysts available on the market demonstrates the Silia*Cat* DPP-Pd to be comparable or better in standard Stille conditions. Table at right shows conversion %.

Silia*Cat* DPP-Pd vs Competitive Catalysts

Silia*Cat* DPP-Pd	Escat 1351	Encat 30	Royer Catalyst	$Pd(PPh_3)_4$	$Pd(OAc)_2$
99	44	95	90	72	20

Stille Coupling Typical Experimental Procedure

Conventional Experimental Conditions

Reaction

All products except the catalyst are added to a round bottom flask equipped with a condenser and a magnetic stirrer. Bring mixture to reflux, and after 10 minutes (*when the solution is homogeneous*) add the required quantity of catalyst.

Work-up

Once the reaction is complete (*determined by TLC or GC-MS*), follow the same work-up procedure as for Suzuki coupling standard conditions.

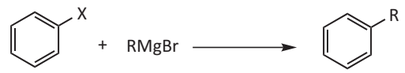
Experimental Conditions - Stille Coupling

Products	Standard Conditions for 1 eq of: Ar-Iodide & Ar-Bromide
Base [K_2CO_3]	1.0- 2.0 eq. (usually 1.1 eq.)
Additive (CsF)	If needed, add 2.0 eq. for higher conversion
Silia*Cat* Catalyst	0.25 - 10.0 mol % (typ.: 2 mol % for -I and 2 - 10 mol % for -Br)
Best Solvents (HPLC Grade)	Dioxane (0.1 M) or Toluene (0.1 M)

*__Note__: molar concentration is related to the substrate.

Kumada Coupling Using Silia*Cat* Pd Catalysts

The Kumada coupling is the direct cross-coupling between an alkyl or an aryl Grignard and a halocarbon. It can be catalyzed by a Pd or a Ni catalyst.

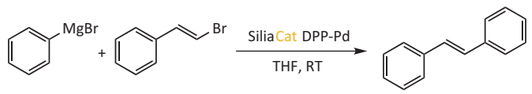


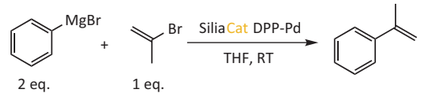
Note: Silia*Cat* Pd^0 results were not available at the time of printing.

Catalyst Concentration Effect

At a constant concentration of substrate, an increase of the amount of Silia*Cat* from 0.1 (or 0.2) to 1.0 will increase the kinetics (*completed in only 15 minutes*).

By increasing the concentration of the catalyst, thus thereby increasing the number of active sites, conversion of the substrate will be favored.





Catalyst Concentration Effect

Silia*Cat* DPP-Pd (mol %)	Solvent (M)	Time (min)	Conversion (%)
1.0	THF (0.07 M)	15	96
0.5	THF (0.07 M)	15	95
0.2	THF (0.08 M)	2 h	94

Catalyst Concentration Effect

Silia*Cat* DPP-Pd (mol %)	Solvent (M)	Time (min)	Conversion (%)
1.0	THF (0.08 M)	15	98
0.5	THF (0.08 M)	90	96
0.2	THF (0.08 M)	4 h	98

Catalyst Reusability and Leaching

Minimal leaching and the robustness of the organoceramic matrix are important factors that allow it to be reused several times.

Silia*Cat* Reusability and Leaching

Reusability	Conversion (%)	Leaching (ppm)	
		Pd	Si
1st	98	0.20	1.5
2nd	95	0.20	2.3
3rd	94	0.50	1.7
4th	77	0.02	1.9

Catalytic Activity and Leaching

Silia*Cat* DPP-Pd showed good reactivity for aryl iodides and bromides. Inert conditions are required for Kumada couplings due to the presence of Grignard reagent.

Catalytic Activity and Leaching

Substrate (R) / Halide (X)	R-MgBr (2 eq.)	Solvent (M)	Time (h)	Conversion (%)	Leaching (ppm)	
					Pd	Si
4-OCH_3 / Br	Ph-MgBr	THF (0.05 M)	18	98	0.3	0.2
4-OCH_3 / Br	i-Bu-MgBr	THF (0.05 M)	18	95	-	-
4-CH_3 / Br	Ph-MgBr	THF (0.05 M)	18	96	-	-
4-CH_3 / Br	i-Bu-MgBr	THF (0.05 M)	18	98	-	-
4-F / Br	Ph-MgBr	THF (0.08 M)	24	94	< 0.01	1.5
H / I	Ph-MgBr	THF (0.08 M)	24	99	-	-
4-OCH_3 / I	Ph-MgBr	THF (0.08 M)	24	94	-	-
4-CH_3 / I	Ph-MgBr	THF (0.08 M)	24	95	< 0.01	1.0

Kumada Coupling Typical Experimental Procedure

Conventional Experimental Conditions

Reaction

All products under inert conditions (*catalyst, solvent, substrates, and Grignard reagent*) are added to a Schlenk or a dry round bottom flask equipped with a magnetic stirrer. The mixture was stirred at room temperature until the TLC or GC-MS analysis confirmed reaction completion (18-24h).

Work-up

Once the reaction is completed, inert conditions are not necessary. Follow same work-up procedure as for Suzuki coupling conventional experimental conditions.

Experimental Conditions - Kumada Coupling

Products	Standard Conditions for 1 eq of: Ar-Iodide & Ar-Bromide
R-MgBr	2.0 eq.
Silia*Cat* Catalyst	2.0 - 10.0 mol % (usually 5 mol %)
Best Solvents (HPLC Grade)	Tetrahydrofuran (0.05 - 0.08 M) (usually 0.08 M)

*Note: molar concentration is related to the substrate. Reaction need to be done at room temperature under inert atmosphere.

Selective Hydrogenation of Nitroarenes Using Silia*Cat* Pt^0

Functionalized anilines are important intermediates in various industries such as pharmaceuticals, polymers, and dyes. Simple aromatic amines are generally obtained by catalytic hydrogenation of nitroarene compounds with various heterogeneous commercial catalysts (*supported nickel, copper, cobalt*) including Pt/C. Yet, the selective reduction of a nitro group with H_2 when other reducible groups are present in the same molecule is generally not feasible with these catalytic materials and requires the use of advanced heterogeneous catalysts. Silia*Cat* Pt^0 exhibits chemoselective catalytic activity for the hydrogenation reaction of a series of substituted nitro compounds under remarkably mild conditions, namely at room temperature with 1 bar H_2 in a simple hydrogen balloon, using a modest 0.5 mol % catalyst amount.

Solvent and Catalyst Concentration Effects

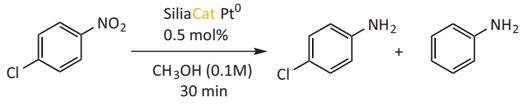
The best results were obtained using methanol as solvent at 0.1 M concentration with respect to substrate. Even if the use of EtOAc results in high selectivities, the reaction times are generally much longer. Complete conversion is obtained after 1 hour in hexane using 0.5 or 0.1 mol % catalyst, but the selectivity to 4-chloroaniline was generally low.

Solvent and Catalyst Concentration Effects

Silia*Cat* Pt^0 (mol %)	Time (h)	Solvent (M)	Yield (%)	
			Product	Aniline
1.0	0.5	MeOH (0.1 M)	92	8
0.5	0.5	MeOH (0.1 M)	87	13
0.2	1	MeOH (0.1 M)	84	13
0.1	2	MeOH (0.1 M)	90	10
0.5	4	EtOAc (0.1 M)	55	0.5
1.0	4	EtOAc (0.1 M)	75	1
1.0	1	THF (0.1 M)	45	17

Silia*Cat* Pt^0 Reusability and Leaching

The reusability test of Silia*Cat* Pt^0 was studied using 4-chloronitrobenzene as substrate under the optimal reaction conditions identified above. Reusing the catalyst in 7 consecutive cycles did not result in any loss of catalytic activity and leaching of Pt and Si (*assessed by ICP-MS*) was minimal. Complete substrate conversion was obtained even after the seventh cycle, with 99% selectivity. The selectivity of the reaction even improves with each subsequent cycle going from 84% in the first run up to 99% in run 7. The positive-feedback phenomenon of effective selectivity in consecutive reaction cycles is probably attributed to the silica matrix alkylation.



Silia*Cat* Pt^0 Reusability and Leaching

Reusability	Yield (%)		Leaching (ppm)	
	Product	Aniline	Pt	Si
1	84	12	0.20	1.20
2	89	11	0.04	0.40
3	90	10	0.02	0.08
4	92	8	0.17	0.10
5	98	2	0.01	0.10
6	99	1	0.01	0.12
7	99	1	0.01	0.08

Silia*Cat* Pt^0 vs Competitive Catalysts

Other commercially available Pt heterogeneous catalysts [Pt/C, Pt/SiO_2 and Reaxa Pt(0)EnCat40] were tested in the selective reduction of 4-chloro-nitrobenzene. In comparison to other Pt(0) heterogeneous catalysts, the Silia*Cat* Pt^0 catalyst proved to be much more reactive, with complete conversion after 0.5 h with just 0.5 mol %. Furthermore, selectivity was significantly higher with only 4% aniline formed as by-product. No secondary product was observed in solution.

Silia*Cat* Pt^0 vs Competitive Catalysts

Catalyst	Pt/C			Pt/SiO_2			Reaxa Pt(0)EnCat40 wet			Reaxa Pt(0)EnCat40 dry		
Mol %	5	1	0.5	5	1	0.5	5	1	0.5	5	1	0.5
Time (h)	1	1	1	1	2	2	1	2	2	0.5	2	2
Product (%)	82	65	43	84	88	48	75	78	72	87	90	86
Aniline (%)	14	4	0	13	10	2	18	14	12	13	10	13

Exp. conditions: 2 mol substrate in 20 mL MeOH under hydrogen balloon at room temperature.

Substrate Scope and Selectivity

The Hydrogenation of different nitro compounds, including those nitro compounds containing different functionalities, was attempted to demonstrate the selectivity of Silia*Cat* Pt^0 catalyst in a wide range of reactions. The material was tested under hydrogen balloon, at room temperature conditions in methanol solvent with 0.5 – 1 mol % Pt catalyst.

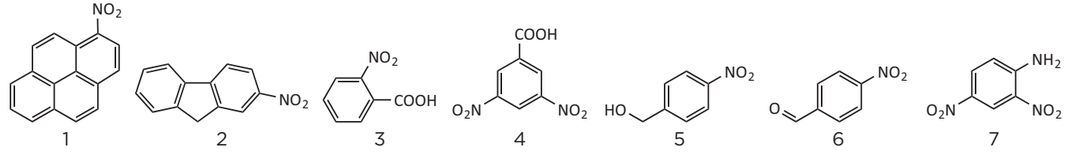
Note: look at our publication in *Adv. Synth. Catal.*, **2011**, *353*, 1306-1316 for more examples.

Substrate Scope and Selectivity

Substrate	Silia*Cat* Pt^0 (mol %)	Solvent (M)	Time (h)	Conversion (%)	Selectivity (%)
Structure #1	0.5	MeOH (0.05 M)	1	100	98 (5% pyrene)
Structure #2	0.5	MeOH (0.05 M)	1	98	100
Structure #3	0.5	MeOH (0.1 M)	1	100	100
Structure #4	0.5 / 1.0	MeOH (0.1 M)	2	100 / 100[1]	98 / 100[1]
Structure #5	0.5	MeOH (0.1 M)	1	100	100
Structure #6	0.5	MeOH (0.1 M)	1	100	95
Structure #7	0.5	MeOH (0.07 M)	2	100	100

[1] If 0.5 mol % was used only one NH_2 group was reduced. If 1 mol % was used, both nitro groups were reduced.

Substrate Structures



Conclusion of Selective Hydrogenation of Nitroarenes

The hydrogenation of different nitro compounds and the selective hydrogenation of different nitro compounds in the presence of different functionalities, including reducible carbonyl, amide, ester, amine and halide groups was achieved with Silia*Cat* Pt^0 catalyst in methanol at room temperature and under 1 bar H_2 pressure. Given the broad applicability of Pt-based catalysts to widely different chemical reactions, it is envisaged that these catalysts, now commercially available, will be used in numerous fields of chemical synthesis as well as in energy generation applications.

Selective Hydrogenation of Nitroarenes Typical Experimental Procedure

Conventional Experimental Conditions

Reaction

Typical reactions are performed on a 2 mmol scale. The substrate is dissolved in 20 mL of MeOH and then treated with 0.1 – 1 mol % of Silia*Cat* Pt^0 catalyst. The mixture is degassed twice, replacing the vacuum by hydrogen each time. The reaction mixture, connected to a balloon of hydrogen, is stirred at room temperature until it shows maximum conversion.

Work-up

Once the reaction is complete (*determined by TLC or GC-MS*), the catalyst is filtered off and washed with EtOH or MeOH. The filtrate is concentrated to give a crude product, and the conversion to the desired product is determined by GC/MS analysis.

Reusability

To reuse the catalyst, after completion of the reaction remove the catalyst by filtration, rinse with MeOH/THF solvents and dry under vacuum.

Selective Debenzylation Using Silia*Cat* Pd^0

The selective debenzylation of aryl benzyl ethers, benzyl esters, and benzyl amines, while leaving other sensitive groups intact, can be carried out in high yield under remarkably mild conditions (*namely at room temperature under 1 bar H_2 in a simple hydrogen balloon, using a modest 0.5 mol % catalyst amount*) using Silia*Cat* Pd^0 (*note that Silia*Cat* Pt^0 can also be used but reaction times are longer and concentrations are higher*).

Selective and smooth deprotection is critical. The commonly used method makes use of catalytic hydrogenolysis to protect benzylic groups with H_2 under pressure and in the presence of a heterogeneous catalyst such as Pd/C or Raney Ni. Often, however, the deprotection reaction conditions are not compatible with other functional groups, such as nitro, unsaturated bonds, and halogen groups.

Silia*Cat* hydrogenation catalysts offer a number of additional advantages over traditional Pd/C. They are non pyrophoric, and have a higher density and lower catalytic consumption (*<1 mol % vs 5 - 10% for Pd/C*) due to fast kinetics and high turnover.

Note: refer you to our publication called “Selective Debenzylation of Benzyl Protected Groups with Silia*Cat* Pd^0 under Mild Conditions” in *ChemCatChem,* **2011**, *3*, 1–5.

Solvent Effect

Solvent choice is critical for any debenzylation reaction. Therefore, in order to optimize the reaction conditions, 1-(benzyloxy)-4-methoxybenzene was used as our substrate of choice. A series of commonly employed solvents (*THF, methanol, ethanol, ethyl acetate, and hexane*) were screened under a hydrogen balloon at room temperature and at different solvent concentrations. The best results were achieved with methanol and ethanol (*HPLC grade*).

Solvent Effect

Silia*Cat* Pd^0 (mol %)	Time (h)	Solvent (M)	Conversion (%)
2	16	EtOH (0.1 M)	17
2	16	MeOH (0.1 M)	15
2	4	EtOH (0.07 M)	100
2	0.5	MeOH (0.07 M)	100
2	20	THF (0.07 M)	10
2	20	THF (0.07 M)	15
2	20	EtOAc (0.07 M)	20
2	20	Hexane (0.07 M)	21

Catalyst Concentration Effect

The molar concentration of the solvent with respect to the substrate is crucial with higher concentrations, slowing or even preventing reaction. The best results were achieved by using a methanol concentration of 0.07 M and 0.5 – 1 mol % Silia*Cat* Pd^0, with complete conversion obtained after 1 – 2 hours.

Catalyst Concentration Effect

Silia*Cat* Pd^0 (mol %)	Time (h)	Solvent (M)	Conversion (%)
2	0.5	MeOH (0.07 M)	100
1	1	MeOH (0.07 M)	100
0.5	2	MeOH (0.07 M)	100

Silia*Cat* Pd^0 Reusability and Leaching

Catalyst stability and reusability are crucial features of any catalyst seeking commercial applications. The Silia*Cat* Pd^0 was thus reused six consecutive times in the O-debenzylation reaction of 1-(benzyloxy)-4-methoxybenzene under the standard mild conditions developed in our laboratory (*reaction shown on previous page using 1 mol % of catalyst*).

After six runs, the catalyst exhibits only a slight loss in activity compared with that of a catalyst run three times. However, the activity remained approximately constant, and it was enough to expand the reaction time to 1 h and 30 min to gain complete debenzylation of the substrate.

Silia*Cat* Pd^0 Reusability and Leaching

Reusability	Time (h)	Conversion (%)	Leaching (ppm)	
			Pd	Si
1st	1	100	0.7	2.5
2nd	1	100	0.3	1.3
3rd	1	95	-	-
	1.5	100	0.3	2.3
4th	1	95	-	-
	1.5	100	0.2	1.4
5th	1	94	-	-
	1.5	99	0.2	0.8
6th	1	94	-	-
	1.5	100	0.1	0.5

Silia*Cat* Pd⁰ vs a Competitive Catalyst

Using the same reaction as that used to demonstrate the reusability of Silia*Cat* Pd⁰ in the O-debenzylation reaction of 1-(benzyloxy)-4-methoxybenzene, we also tested the commercial catalyst Pd0 EnCat, a polyurea-entrapped catalyst.

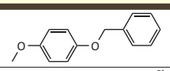
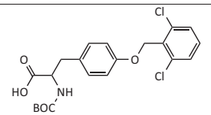
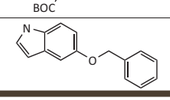
Silia*Cat* Pd⁰ vs a Competitive Catalyst

Catalyst (mol %)	Time (h)	Conversion (%)	Selectivity (%)
Silia*Cat* Pd⁰ (0.5)	1 / 2	95 / 100	- / 100
Silia*Cat* Pd⁰ (1.0)	0.5 / 1	75 / 100	- / 100
Pd0 EnCat (10)	16	100	100

Substrate Scope and Selectivity

Silia*Cat* Pd⁰ is an efficient catalyst for the selective debenzylation of different aryl benzyl ethers, benzyl amino-acids, and benzylprotected sugars leaving other sensitive groups intact. Refer to our publication in *ChemCatChem,* **2011**, *3*, 1–5 for more examples.

Substrate Scope & Selectivity

Substrate	Silia*Cat* Pd⁰ (mol %)	Time (h)	Conversion [Yield] (%)	Leaching (ppm) Pd	Leaching (ppm) Si
	1	1	100 [99.7]	3.4	1.3
	1	1	100 [98.6]	1.7	5.0
	1	20	100 [98.0]	0.4	7.0

Conclusion of Selective Debenzylation

In conclusion, the Silia*Cat* Pd⁰ catalyst is suitable for the selective debenzylation of numerous substrates under mild conditions with a modest 1 – 2 mol % catalyst amount. Benzyl-protected sugars, amino acids, ethers, and esters are smoothly debenzylated under 0.1 MPa H_2 at room temperature.

Selective Debenzylation Typical Experimental Procedure

Conventional Experimental Conditions

Reaction

Typical reactions were performed on a 1 mmol scale. The substrate was dissolved in 15 mL of MeOH or EtOH (0.07 M) and 1 or 2 mol % of the Silia*Cat* Pd⁰ catalyst was added. The mixture was degassed twice and each time replacing the vacuum by hydrogen. The reaction mixture, connected to a balloon filled with hydrogen, was stirred at room temperature until GC/MS analysis showed maximum conversion.

Work-up

Once the reaction is complete as deemed by TLC or GC-MS, the catalyst was filtered off and washed with EtOH or MeOH. The filtrate was concentrated to give a crude product. The conversion in the desired product was determined by GC/MS analysis and by ^{1}H NMR.

Reusability

To reuse the catalyst, after completion of the reaction, remove the catalyst by filtration, rinse with MeOH/THF solvents and dry under vacuum.

Hydrosilylation Using Silia*Cat* Pt⁰

Hydrosilylation reactions (*or catalytic hydrosilation*) are a widely used method to prepare organosilicon products. The reaction consists of the addition of *Si*-H bonds on unsaturated bonds like alkenes, alkynes or ketones, where catalysts are often required (usually H_2PtCl_6). Silia*Cat* Pt⁰ can be used for hydrosilylation reactions. Some examples are shown to the right.

1 eq 1.3 eq

Hydrosilylation using Silia*Cat* Pt⁰

Substrate	Time (h)	Temp. (°C)	Conversion (%)	Selectivity (%)
1-octene	5	22 / 60	88 / 99	98 / 99
1-decene	5	22 / 60	100 / 100	97 / 98
1-octadecene	5	22 / 60	95 / 98	56 / 83
4-vinyl-benzamine	5 / 24	60	47 / 80	96 / 96
3,3-diethoxy-prop-1-ene	5	22 / 60	94 / 100	93 / 81

Hydrosilylation Typical Experimental Procedure

Conventional Experimental Conditions

Reaction

A 100 ml two neck dry round bottom flask equipped with a condenser and a rubber septum is filled with 1 mol % Silia*Cat* Pt⁰ and was degassed two times for 15 minutes kept under argon conditions. The anhydrous solvent, the silane (*95% pure*) and the olefin (*previously degassed for 15 minutes under argon*) were added using a syringe. The reaction mixture was either stirred at room temperature or heated at 60°C until the GC/MS analysis showed maximum conversion

Work-up

Once the reaction is complete the catalyst was filtered off and washed with toluene. The filtrate was concentrated to give a crude product. The conversion in the desired product was determined by GC/MS.

Note: Unless otherwise indicated, all manipulations were carried out under argon conditions. In general, reactions were performed on a 2 mmol scale in 15 ml anhydrous toluene.

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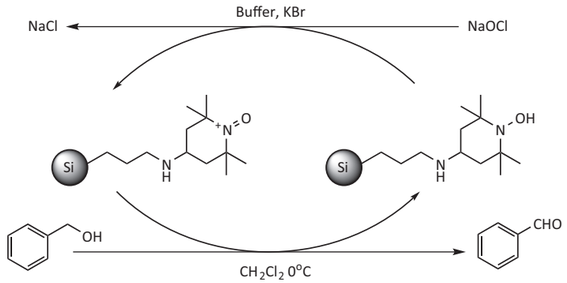
Oxidation Using Silia*Cat* TEMPO

Aldehydes and ketones, either as starting materials, synthetic intermediates, or final products, are of great interest in synthetic chemistry. Such carbonyl-containing products can lead to carbon–carbon (*i.e. Wittig, Aldol, alkylation*) or carbon–nitrogen bond formation. Over the years, chemists have discovered various oxidizing agents such as pyridinium chlorochromate (PCC), MnO2, Dess-Martin periodinane, or Swern oxidation conditions. Although all these methods lead to the aldehyde (*limited oxidation of the aldehyde to the carboxylic acid*), they have drawbacks such as the hazards and toxicity associated with residual metal contamination.

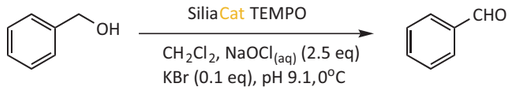
Development of environmentally friendly methods such as selective catalytic oxidation of alcohol substrates to aldehydes and ketones can have significant impact on modern methods of chemical synthesis. Silia*Cat* TEMPO is the oxidation solution of choice.



Catalytic Performance and Leaching

Silia*Cat* TEMPO was investigated in the Montanari-Anelli conditions. The catalytic cycle involves regeneration of the oxidative species with NaOCl (*commercially available bleach*) in presence of KBr as co-catalyst to form the stronger anion OBr-.

Unless otherwise stated, the reaction shown below was used for the demonstration.



Catalytic Performance and Leaching

Silia*Cat* (mol %)	Time (h)	Conversion (%)	TON	Si Leaching (ppm)
0.1	1	95	950	-
0.01	2	83	8,300	3
0.01	3	95	9,500	1.6
0.01	4	97	9,650	1.5
0.02	2	96	4,800	-
0.02	3	100	5,000	2

Silia*Cat* TEMPO can be used with as low as 0.01 mol % quantity to provide the desired aldehyde in short reaction times. ICP analysis confirms that the material is leach-resistant ([Si] ≤ 3 ppm).

Silia*Cat* TEMPO Reusability

Minimal leaching and the robustness of Silia*Cat* TEMPO's organoceramic matrix allow it to be reused several times for further uses.

Silia*Cat* TEMPO Reusability

Reusability	Time (min)	Conversion (%)	Reusability	Time (min)	Conversion (%)	Reusability	Time (min)	Conversion (%)
1st	30	100	...	...	...	9th	30 / 60	97 / 100
2nd	30	100	8th	30 / 60	95 / 100	10th	30 / 60	90 / 100

[a]Silia*Cat* TEMPO is recycled by post-reaction filtration, DCM washes and air drying.

Influence of Co-Catalyst KBr and Temperature

SiliCycle investigated wether it was necessary to use a co-catalyst (KBr) for the reaction to proceed effectively. As shown in the table, although KBr is not required for the reaction, it does have a significant impact on the kinetics. The reaction can still proceed to completion without KBr but requires longer time and/or more Silia*Cat* TEMPO. It was also demonstrated that the reaction can be carried out at room temperature without KBr.

Influence of KBr and Temperature

Silia*Cat* (mol %)	KBr (eq.)	Temp. (°C)	Time (min)	Conversion (%)
0.1	0.1	0	60	95
0.1	0	0	60	80
0.1	0	0	210	100
0.2	0	0	105	96
0.2	0	22	60	76
0.2	0	22	90	87

Influence of Solvents, pH and NaOCl

As shown on the right, the reaction can be carried out at pH 9.0 or at pH 7.5 in DCM with high conversion yields. The catalytic conditions are selective towards the aldehyde, rather than the carboxylic acid, even with 10 equiv of NaOCl. At pH 7 in water, the reaction is slower, but this can be overcome by using more $NaOCl_{(aq)}$. At pH 9, the conversion is high, but too much bleach and the long reaction time in the aqueous media will lead to the corresponding carboxylic acid. The reaction can also be pursued in other organic solvents.

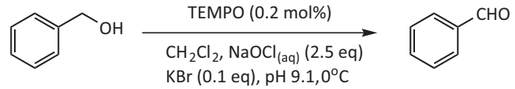
Influence of Solvent, pH and $NaOCl_{(aq)}$

Silia*Cat* (mol %)	$NaOCl_{(aq)}$ (eq.)	Solvent	pH	Time (min)	Conversion (%)
0.2	2.50	DCM	9.0	60	98
0.2	10.00	DCM	9.0	90	98
0.2	1.25	DCM	7.5	60 / 90	83 / 86
0.2	2.50	DCM	7.5	60 / 90	94 / 98
0.2	1.25	H_2O	7.5	60 / 90	57 / 65
0.2	2.50	H_2O	7.5	60 / 90	87 / 88
0.7	1.20	H_2O	9.0	60 / 150	83 / 89
0.8	5.00	H_2O	9.0	60 / 18 h	60 (19)/ 7 (89)[1]
0.2	1.25	EtOAc	9.0	60 / 90	95 / 96

[1] In parenthesis = conversion to carboxylic acid.

Silia*Cat* TEMPO vs Homogeneous TEMPOs

Comparative analysis versus homogeneous TEMPOs demonstrates the Silia*Cat* TEMPO to be comparable or better at neutral pH and significantly superior in basic conditions.



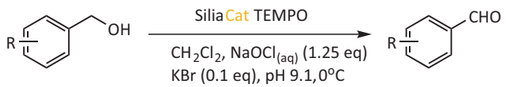
Silia*Cat* TEMPO vs Homogeneous TEMPOs

pH	Silia*Cat* TEMPO	4-MeO-TEMPO	4-Oxo-TEMPO
7.5	91	99	45
9.0	98	55 (40)[1]	73

[1] In parenthesis = conversion to carboxylic acid.

Substrate Scope with Silia*Cat* TEMPO

Silia*Cat* TEMPO is efficient with different substrates and can be used with phase a transfer agents such as Aliquat 336. When an electron-rich benzylic alcohol cannot be oxidized successfully with NaOCl, conditions involving I_2 in toluene, at room temperature, will yield the desired product.



Substrate Scope with Silia*Cat* TEMPO

Substrate (R)	Catalyst (mol %)	Time (min)	Conversion (%)
3-NO_2	0.4	90	100
4-NO_2	0.4	90	98
4-OCH_3	0.4	90	36
4-OCH_3	0.4 (0.05 eq. Aliquat 336)	60	79
4-Cl	0.4	90	95
3-phenyl-1-propanol	0.4	60	97
1-phenyl-3-propanol	0.4	180	95
4-OCH_3	8.2	16 h	99[1]
3-OCH_3	7.8	16 h	96[1]
Piperonal	10.0	20 h	100[1]

[1]Exp. Cond.: I_2 (1.8 eq.), $NaHCO_3$(aq), pH 8, toluene, 22°C.

Conclusion of Oxidation

In conclusion, the Silia*Cat* TEMPO is an effective oxidizing catalyst presenting unique advantages such as high activity, robustness, leach-proof properties and selectivity toward the oxidation of alcohols into aldehydes and ketones, both very valuable products in organic chemistry.

Oxidation Typical Experimental Procedure

Oxidation of Alcohols or Aldehydes to Carboxylic Acid

Note: changing the solvent to water, increasing temperature and the amount of bleach will all favor the acid formation.

Conventional Experimental Conditions

Reaction

Under mechanical agitation, a 0.4M solution of alcohol in water and a 0.5 M aqueous solution of KBr were cooled at 0°C in an ice bath. The desired amount of Silia*Cat* TEMPO was added, followed by an aqueous solution of NaOCl (*from 10-13% bleach*) buffered at pH 9 (*using* $NaHCO_3$) or pH 6.7 (*using* NaH_2PO_4/Na_2HPO_4). NaOCl was added slowly over a 10 minute period as the reaction is exothermic. The mixture was warmed to room temperature (20°C) and stirred between 1,300-1,500 rpm. The temperature can be increased to 35°C if necessary.

Work-up

Once the reaction was complete (*determined by TLC or GC-MS*), the catalyst was filtered at room temperature, and the pH was adjusted to 12 with aqueous NaOH (2N). The aqueous phase was separated, acidified with HCl 6N and extracted with CH_2Cl_2. The organic phase was dried over $MgSO_4$ and evaporated. The residue was purified by crystallization or column chromatography on silica gel.

- 1.2 - 5 eq. of $NaOCl_{(aq)}$ (*typically start with 3 eq. and, if necessary, add another 2 eq. of* NaOCl *via an addition funnel after all of alcohol is consumed*)
- 0.1 eq. of potassium bromide (KBr) (*prepared as a 0.5 M solution*)
- pH 9 is achieved using a $NaHCO_3$ buffer or a pH of 6.7 is achieved using a sodium phosphate buffer (*1:1 mixture of 0.67 M* NaH_2PO_4 *and 0.67 M* Na_2HPO_4)
- 0.01 - 1 mol % of Silia*Cat* TEMPO (*typically 1 mol %*)
- The best solvents are H_2O, ACN/H_2O or DCM/H_2O, typically at 0.4 M (*molar concentration with respect to the substrate*)

Oxidation of Primary or Secondary Alcohols

Under Montanari-Anelli Conditions (using NaOCl)

Conventional Experimental Conditions

Reaction

Under mechanical agitation, a 0.4M solution of the alcohol in dichloromethane is mixed with a 0.5M aqueous solution of KBr and cooled at 0°C in an ice bath. The desired amount of Silia*Cat* TEMPO is then added, followed by an aqueous solution of NaOCl (*from commercially available 10-13% bleach*), then the solution is buffered at pH 9 (*using* $NaHCO_3$). NaOCl solution is added slowly over a 10 minute period as the reaction is exothermic. The mixture is then stirred between 1,300-1,500 rpm.

Work-up

Once the reaction is complete (*determined by TLC or GC-MS*), the catalyst is filtered at room temperature, and the organic phase is dried over $MgSO_4$ and evaporated. Crude mixture is purified using flash chromatography, if needed.

Under Miller Conditions (using I_2 co-catalyst)

Conventional Experimental Conditions

Reaction

Under mechanical agitation, a 0.4M solution of alcohol in toluene is mixed at room temperature (20°C) with a 0.3 M aqueous solution of $NaHCO_3$. Solid iodine is then added in one portion to the mixture, followed by the desired amount of Silia*Cat* TEMPO.

Work-up

Once the reaction is complete (*determined by TLC or GC-MS*), the catalyst is filtered at room temperature. The mixture should then be cooled to 5°C, diluted with ethyl acetate, and quenched with a 0.8M aqueous solution of Na_2SO_3. The uncolored organic phase is then washed with a saturated aqueous solution of $NaHCO_3$ followed by brine and dried over $MgSO_4$. After filtration and evaporation of the solvents, the crude mixture can be purified using flash chromatography.

- For Montanari-Anelli conditions: 1.2 - 5 eq. of $NaOCl_{(aq)}$ (*typically 2.5 eq.*) and 0.1 eq. of KBr (*prepared as a 0.5 M solution*)
- For Miller conditions: 1.8 eq. of solid iodine (I_2)
- 0.001 - 1 mol % of Silia*Cat* TEMPO (*typically 1 mol %*)
- The best solvents are DCM, EtOAc or ACN/H_2O (*HPLC grade*), typically at 0.4 M (*molar concentration is with respect to the substrate*)

SiliCycle Publications

Silia*Cat* TEMPO Oxydation

Topics in Catalysis, **2010**, *53*, 1110-1113
Organic Process Research & Development, **2010**, *14*, 245–251
Chemistry Today, **2009**, *27*, 13-16
Organic Process Research and Developement, **2007**, *11*, 766-768

Hydrogenation of nitroarenes with Silia*Cat* Pt^0

Advanced Synthesis & Catalysis, **2011**, *353*, 1306–1316
Catal. Sci. Technol., **2011**, Advance Article, DOI: 10.1039/C1CY00097G

Suzuki coupling with Silia*Cat*

Catal. Sci. Technol., **2011**, Advance Article, DOI: 10.1039/C1CY00119A
Topics in Catalysis, **2010**, *53*, 1059-1062

Selective debenzylation with Silia*Cat* Pd^0

ChemCatChem, **2011**, *3*, 1146-1150

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Silia*Bond* Pyridinium Chlorochromate (*R24030B*) and Silia*Bond* Pyridinium Dichromate (*R24530B*)

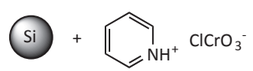
Loading 20.0% w/w	Endcapping: No	Category: Oxidant	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

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

is commonly used for the 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]

Pyridinium Chlorochromate (*Si*-PCC)

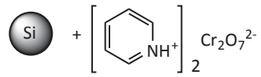


Silia*Bond* Pyridinium Dichromate (*Si*-PDC)

may be used as an alternative to *Si*-PCC in nucleoside and carbohydrate oxidation, particularly for fragile molecules.[10] Silia*Bond* PDC can also be used in conjunction with tertbutylhydroperoxide for a variety of oxidative transformations.[11]

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.[12]

Pyridinium Dichromate (*Si*-PDC)



Solvent compatibility

- Anhydrous CH_2Cl_2

Prolonged storage

- Keep cool (< *8°C*) and dry

[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
[10] *J. Chem. Soc.* Perkin Trans. I, **1982**, 1967
[11] *J. Chem. Soc.* Chem. Commun., 7, **1993** 651
[12] Tetrahedron, 35, **1979**, 1789

Silia*Bond* Potassium Permanganate (*R23030B*)

Loading 20.0% w/w	Endcapping: No	Category: Oxidant	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

Potassium permanganate

is a strong oxidant that will oxidize methyl groups and alcohols to carboxylic acids. Silia*Bond* Potassium Permanganate 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 Silia*Bond* Potassium Permanganate, the manganese salt by-products stay adsorbed onto the silica.

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

Solvent compatibility

- Anhydrous CH_2Cl_2

Prolonged storage

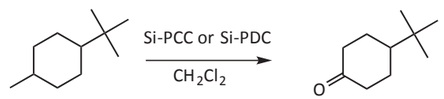
- Keep dry

Potassium permanganate

Oxidation of Alcohols to Aldehydes and Ketones

General procedure

Silia*Bond* PDC or Silia*Bond* PCC (*2 eq.*) and acetic acid (*4 mmol*) were addeed to a solution of the alcohol % mmol) in CH_2Cl_2 (*7.5 mL*). The resulting mixture was stirred for 6 h at room temperature. Ether (*15 mL*) was added, and after stirring for another 2 min, the solution was filtered and the solids were washed with ether (*4 x 9 mL*). Concentration under vacuum afforded the required product.



Oxidation of Alcohols Results

Silia*Bond* Oxidant	Conditions	Conversion[a]
Silia*Bond* PCC	6 h, room temperature	100%
Silia*Bond* PDC	6 h, room temperature	100%

[a] Determined from the isolated product

TEL.: 1 418 874.0054 FAX: 1 418 874.0355 TOLL-FREE: 1 877.SILICYCLE (NORTH AMERICA ONLY) WWW.SILICYCLE.COM INFO@SILICYCLE.COM

SiliaBond®
Reagents

SILICYCLE inc.
UltraPure SILICA GELS

Silia*Bond* Reagents

Amide Coupling Reagents

The amide bond is the defining molecular structure of proteins and peptides. In addition, a report estimates that as many as 25% of all synthetic pharmaceutical drugs contain an amide group.[1] Therefore, there is an ongoing scientific endeavor to develop efficient amidation methodologies.[2] 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.

The use of a reagent linked to an insoluble material has become a widely used tool since the introduction of the solid-phase synthesis concept.[3] Solid-phase reagents are valuable for amide coupling with a carboxylic acid because of the decrease of unwanted side products. Other advantages to 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 both batch reactions and flow chemistry.

[1] *J. Comb. Chem.* **1999**, *1*, 55.

[2] *Tetrahedron* **2005**, *61*, 10827.

[3] *J. Am Chem Soc.* **1963**, *85*, 2149.

Silia*Bond* Carbodiimide (*R70530B*)

Loading: 1.0 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

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

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.

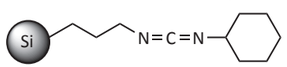
Solvent compatibility

- Aprotic Solvent

Prolonged storage

- Keep cool (< *8* °C) and dry, store under argon

Carbodiimide (*Si-DCC*)



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

Loading: 0.8 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

Silia*Bond* Ethyl-Dimethylaminopropyl Carbodiimide (*Si-EDC*)

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 potentially 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.

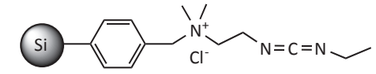
Solvent compatibility

- Aprotic Solvent

Prolonged storage

- Keep cool (< *8* °C) and dry, store under argon

Ethyl-Dimethylaminopropyl Carbodiimide (*Si-EDC*)



Silia*Bond* Dichlorotriazine (*R52230B*)

Loading: 0.7 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

Silia*Bond* Dichlorotriazine (*Si-DCT*)

2,4,6-Trichloro[1,3,5]triazine (*cyanuric chloride*) has been used as a versatile reagent in alkyl chloride and acid chloride synthesis. This triazine has been especially useful as a coupling reagent for amide selective formation.[1] However, cyanuric chloride is toxic, corrosive, and a severe eye, skin and respiratory tract irritant. By anchoring cyanuric chloride on a silica matrix, it is now possible to use this valuable reagent without worrying about its toxicity profile. Silia*Bond* DCT reacts in a similar manner as cyanuric chloride. In addition, excess reagent and by-product elimination is reduced to a simple filtration, which is particularly useful for products where toxicity is a concern such as in the synthesis of active pharmaceutical ingredients (*API*).

[1] *J. Org. Chem.* **1997**, *62*, 982.

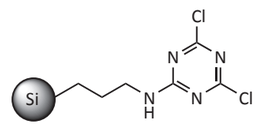
Solvent compatibility

- Aprotic Solvent

Prolonged storage

- Keep cool (< *8* °C) and dry, store under argon

Dichlorotriazine (*Si-DCT*)



Silia*Bond* HOBt (*R70730B*)

Loading: 0.7 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

Silia*Bond* HOBt (*Si-HOBt*) NEW PRODUCT

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 condition as in homogeneous solution. Moreover, this supported reagent can be reused a few times without adversely affecting its performance.

Solvent compatibility

- Aprotic Solvent

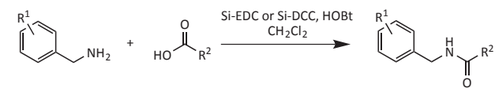
Prolonged storage

- Keep cool (*< 8 °C*) and dry, store under argon

HOBt (*Si-HOBt*)

Synthesis of Capsaicin Analogues

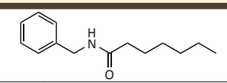
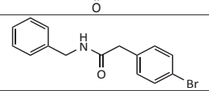
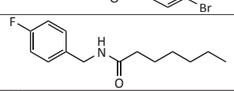
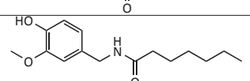
Capsaicin's potential clinical use as an analgesic and peripheral anti-inflammatory effects, as well as the discovery of an ultra-potent analogue (*resiniferatoxin*) has attracted significant interest in finding capsaicin synthesis routes.



General Procedure

The acid (*0.5 mmol*) was placed in an oven-dried reaction vial with anhydrous CH_2Cl_2 (*10 mL*) under N_2. The HOBt (*1.0 mmol*) and the Silia*Bond* Carbodiimide or Silia*Bond* EDC were added to the solution, which was then stirred briefly (*5 min*). The amine (*0.5 mmol*) was then added to the reaction tube, and the mixture was then stirred for 16 h at room temperature. Finally, the reaction was followed by GC-MS.

Capsaicin Analogues Reaction Results

Entry	Product	Yield[a] (Purity[b]) *Si*-DCC	Yield[a] (Purity[b]) *Si*-EDC
1		99% (> 98%)	81% (> 98%)
2		98% (> 98%)	88% (95%)
3		99% (> 98%)	99% (> 98%)
4		98%[c]	98%[c]

[a]Yield calculated in crude product, [b]Purity determined by GC-MS, [c]Yield determined by GC-MS

Amine Protection Using Benzylcarbamate Group

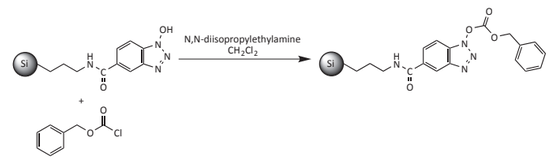
Benzylcarbamate groups are one of the most used amine protecting functions because of the easy deprotection by hydrogenenolysis. Silia*Bond* HOBt, as a key reactive, facilitates the protection manipulation and can be reused a few times without loss of reactivity.

General Procedure

Silia*Bond* HOBt (*1 g or 1 eq.*) was introduced in a flask (*oven-dried*) containing anhydrous CH_2Cl_2. Benzylchloroformate (*4 eq.*) was added to the suspension, followed by N,N-diisopropylethylamine 4 eq. The reaction mixture was stirred for 60 minutes at room temperature. Then, the suspension mixture was filtered, and washed with CH_2Cl_2 (*2 x 10 mL*), and the Silia*Bond* HOBt was oven-dried.

The dried, activated Silia*Bond* HOBt was placed in a flask containing anhydrous CH_2Cl_2 under N_2. To this suspension, 0.8 eq. of amine was added, and the reaction mixture was stirred for 4 to 16 h at room temperature. The reaction suspension was filtered and washed with CH_2Cl_2 (*2 x 10 mL*).

Activation Reaction



Activation and recycling Results

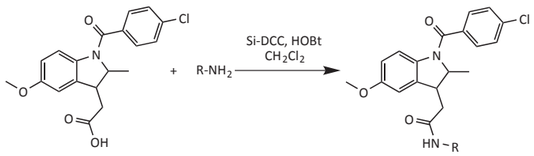
Entry	Yield[a]
Activation	96%
1st Recycling	86%
2st Recycling	95%
3st Recycling	96%

[a]Conversion determined by GC-MS

Synthesis of Amide Derivatives of Indomethacin

A report[1] has shown that indomethacin primary and secondary amide analogues are potent compounds for human COX-2 specific inhibition. Silia*Bond* Carbodiimide can be used as a key reagent in its synthesis.

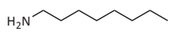
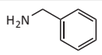
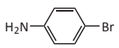
[1] *J. Med. Chem.* **2000**, 2860.



General Procedure

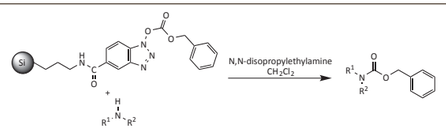
The indomethacin (*0.56 mmol*) was placed in an oven-dried reaction vial in anhydrous dichloromethane (*5 mL*) under N_2. HOBt (*0.95 mmol*) and the Silia*Bond* Carbodiimide (*1.12 mmol*) were added, and the mixture was stirred briefly (*5 minutes*). Then, the amine (*0.56 mmol*) was added to the vial, and the reaction was stirred at room temperature for 16 h. Then, the crude product was directly purified on a short plug of silica gel (*hexane/EtOAc 1/1*) to yield pure amide.

Amide Derivatives of Indomethacin Results

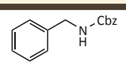
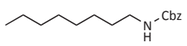
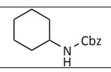
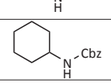
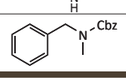
Entry	Amine	Yield[a]
1		90%
2		82%
3		94%
4		78%

[a]Conversion determined by GC-MS

Amine Protection Reaction



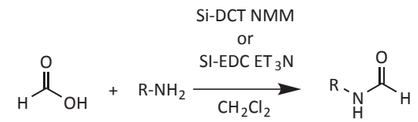
Amine Protection Results

Entry	Product	Conversion[a]
1		98% (4 h)
2		94% (4 h) 96% (16 h) 86% (16 h)[b]
3		81% (16 h)
4		93% (4 h) 98% (16 h)
5		98% (4 h)
6		93% (16 h)

[a]Conversion determined by GC-MS, [b]Polymer HOBt

Synthesis of Formylated Amino Acids

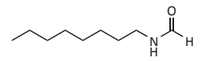
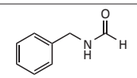
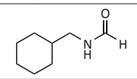
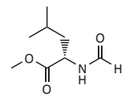
N-formylamino acid esters are useful derivatives for preparing selected N-formylamino acids, incorporating polyfunctional amino acids into peptides, and for other useful starting material preparation. Formylated amino acids have been prepared in high yields by using Silia*Bond* Dichlorotriazine (*DCT*) and Silia*Bond* Ethyl-Dimethylaminopropyl Carbodiimide (*EDC*).



General Procedure

Formic acid (*0.90 mmol*) was placed in an oven-dried reaction vial in anhydrous CH_2Cl_2 (*10 mL*) under N_2. To this solution was added triethylamine (*0.90 mmol*) and either the Silia*Bond* EDC (*2.25 mmol*) or N-methylmorpholine (*0.90 mmol*) and Silia*Bond* DCT (*2.25 mmol*). Then, the mixture was stirred briefly (*5 minutes*). The amine (*0.45 mmol*) was then added to the vial and the reaction was stirred at room temperature for 16 h. Conversion to the desired formamide was followed by GC-MS. Upon completion, the Silia*Bond* EDC or Silia*Bond* DCT was filtered and washed with 2 x 10 mL of CH_2Cl_2. Evaporation of the solvent yielded the desired product.

Synthesis of Formylated Amino Acids Results

Entry	Product	Conversion[a] *Si*-DCT	Conversion[a] *Si*-EDC
1		99%	93%
2		99%	100%
3		99%	99%
4		98%	95%

[a]Conversion determined by GC-MS

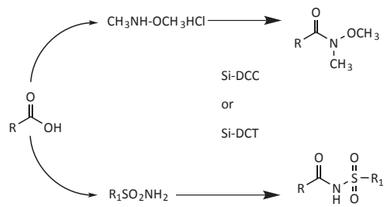
Weinreb and Acylsulfonamide Synthesis

Weinreb synthesis is a reaction often used in medicinal chemistry to produce amides. These functional groups are present in natural products and can be reliably reacted to form new carbon-carbon bonds or converted to other functions. In normal conditions, Weinreb synthesis can tolerate a large variety of functional groups such as N-protected amines, sulfonates, alpha-beta saturation and silyl ethers.

Weinreb Synthesis Results

Acid	Amine	Yield (Purity)[a]	
		Si-DCC	*Si*-DCT
Benzoic Acid	N,O-Dimethylhydroxyamine Hydrochloride	99% (96%)	96% (94%)
t-Cinnamic Acid		87% (95%)	82% (70%)
2-Nitrobenzoic Acid		> 99% (93%)	92% (79%)

[a]Yield and purity determined by GC-MS



Acylsulfonamide Synthesis Results

Acid	Sulfonamide	Yield (Purity)[a]	
		Si-DCC	*Si*-DCT
Benzoic Acid	Benzenesulfonamide	96% (71%)	98% (90%)
	Methanesulfonamide	79% (53%)	71% (82%)

[a]Yield and Purity determined by GC-MS

SiliaBond Cyanoborohydride for Reductive Aminations

Reductive amination involves the conversion of a carbonyl group, most of the time a ketone or an aldehyde, to an amine by 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 than ketones and are stable under moderately acidic conditions.

General Procedure

To 1 mmol of SiliaBond Cyanoborohydride 5 mL of solvent, 0.5 mmol of aldehydes or ketones and 0.6 mmol of amines were added. The reaction mixture was stirred at room temperature for 16 h. Each solution was then analysed by GC-MS.

SiliaBond Cyanoborohydride (*R66730B*)

Loading: 1.0 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

SiliaBond Cyanoborohydride (*Si-CBH*)

SiliaBond 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. Cyanide contamination of the product is a concern, however, 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. To see if any cyanide ion was leaching from the silica, 1 g of SiliaBond Cyanoborohydride was washed 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 workup required only a filtration, and HCN and NaCN were not liberated during workup.

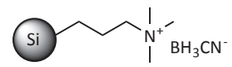
Solvent compatibility

- All solvents, aqueous and organic

Prolonged storage

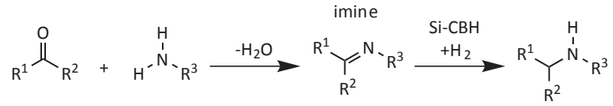
- Keep cool (< 8 °C) and dry, store under argon

Cyanoborohydride (*Si-CBH*)

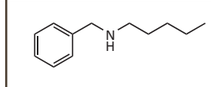
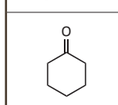
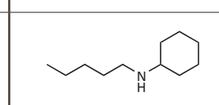
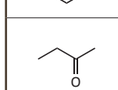
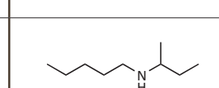
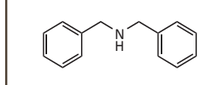
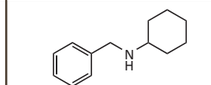
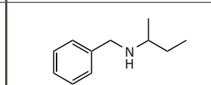
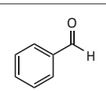
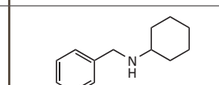
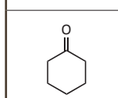
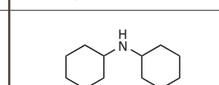
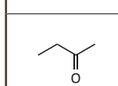
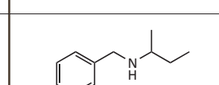


SiliaBond Cyanoborohydride for Reductive Aminations

Reduction of Primary Amines

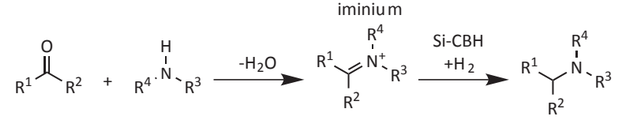


Reduction of Primary Amine Results

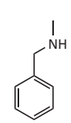
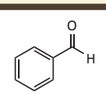
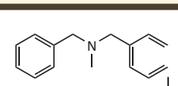
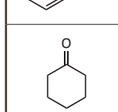
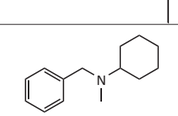
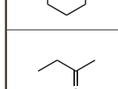
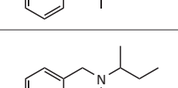
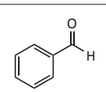
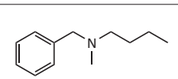
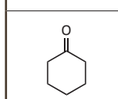
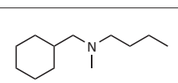
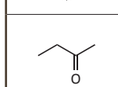
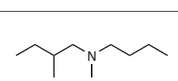
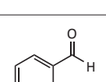
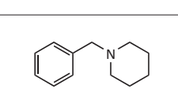
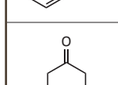
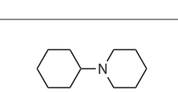
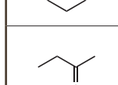
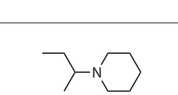
1°Amine	Carbonyl	Conditions (RT 16 h) Product	Acetonitrile Conversion Product (%)[a]	Acetonitrile Imine (%)[b]	Ethanol Conversion Product (%)[a]	Ethanol Imine (%)[b]	Methylene Chloride Conversion Product (%)[a]	Methylene Chloride Imine (%)[b]
			27	25	64	11	69	12
			97	0	95	5	92	8
			92	0	84	7	78	9
			61	20	71	23	73	24
			92	2	83	17	81	13
			88	3	90	7	91	6
			66	21	97	0	100	0
			91	5	93	5	93	6
			90	0	92	6	86	7

[a]Conversion determined by GC-MS, [b]Unreacted imine was determined by GC-MS

Reduction of Secondary Amines



Reduction of Secondary Amine Results

2°Amine	Carbonyl	Conditions (RT 16 h) Product	Acetonitrile Conversion Product (%)[a]	Acetonitrile SM[c] (%)[b]	Ethanol Conversion Product (%)[a]	Ethanol SM[c] (%)[b]	Methylene Chloride Conversion Product (%)[a]	Methylene Chloride SM[c] (%)[b]
			90	2	71	0	91	0
			92	5	79	17	93	3
			79	8	79	21	93	2
			94	6	67	0	79	0
			77	23	77	20	87	3
			70	25	61	26	44	2
			97	3	80	0	83	1
			85	15	69	19	88	6
			81	9	70	21	55	2

[a]Conversion determined by GC-MS, [b]Unreacted imine was determined by GC-MS, [c]Starting Material

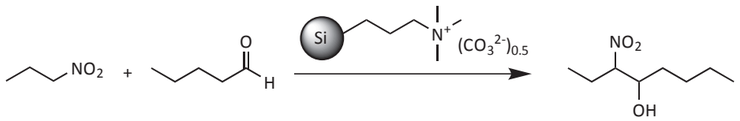
TEL.: 1 418 874.0054 FAX: 1 418 874.0355 TOLL-FREE: 1 877.SILICYCLE (NORTH AMERICA ONLY) WWW.SILICYCLE.COM INFO@SILICYCLE.COM

Silia*Bond* Carbonate for Henry Reactions

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, alpha-nitro ketones by oxidation and ß-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.

General Procedure

1-nitropropane (*1 eq.*) was added to a solution containing THF (*5 mL*) and valeraldehyde (*1 eq.*). Silia*Bond* Carbonate (*0.1 eq.*) was added, and the mixture was stirred at room temperature for 6 h. The reaction mixture was then filtered and washed with THF and the crude product was evaporated. Finally, pure product was obtained after flash chromatography purification using a mix of hexane/ethylacetate (*80/20*).



Henry Reaction Results

Entry	Solvent	Reaction Conditions	Conversion[a]
1	THF	0.1 eq. Si-CO_3 room temperature, 6 h	92% (83%)[b]
2	CH_2Cl_2		76%
3	Ethanol		90%
4	Propanol		95%
5	None		92%
6	THF	0.1 eq. Si-CO_3 µwave 100 W, 100°C 10 min	89%

[a]Conversion determined by GC-MS, [b]Purity determined from the isolated product

Silia*Bond* Carbonate (*R66030B*)

Loading: 0.7 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

Silia*Bond* Carbonate (*Si-CO_3*)

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.

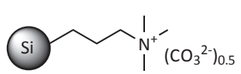
Solvent compatibility

- Aprotic solvents

Prolonged storage

- Keep dry

Carbonate (*Si-CO_3*)



Silia*Bond* DMAP for Baylis-Hillman and Acylation Reactions

Baylis-Hillman Reaction

Coupling of activated alkenes, generally alpha, 1-beta-unsaturated, with aldehydes is named the Baylis-Hillman reaction. This reaction is well known for the formation of carbon-carbon bonds under soft conditions and its compatibility with several functional groups. Furthermore, an organic base can be used to catalyze this reaction with similar success to using transition metals.

Acylation Reaction

It is well-known that DMAP used as a catalyst increases speed and yield of alcohol and phenol acylations over acetic and bezoic anhydrides.

Silia*Bond* DMAP (*R75530B*)

Loading: 0.8 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

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

Silia*Bond* DMAP is the heterogenous 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.

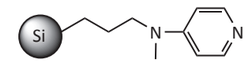
Solvent compatibility

- All organic solvents

Prolonged storage

- Keep cool (*< 8 °C*) and dry, store under argon

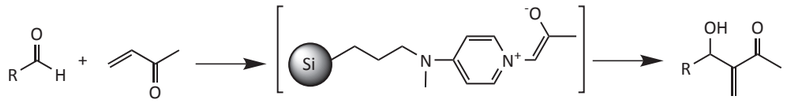
DMAP (*Si-DMAP*)



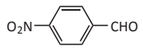
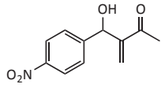
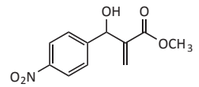
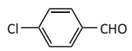
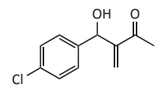
SiliaBond DMAP for Baylis-Hillman Reaction

General procedure

Aldehyde (*1 mmol*) was placed in a flask, and THF, SiliaBond DMAP (*0.10 mmol*), water and enone (*2 mmol*) were added. The mixture was stirred at room temperature for 6 to 96 h.



Baylis-Hillman Reaction Results

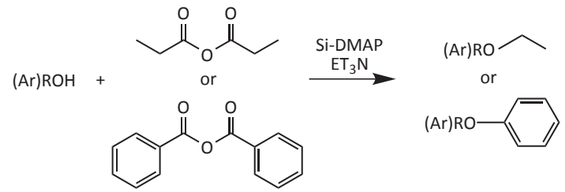
Aldehyde	Enone	Conditions	Product	Yield[a] *Si*-DMAP	Yield[a] Polymer
		THF/H_2O (3/1) room temperature, 6 h 10% *Si*-DMAP		81%	37%
		DMF/H_2O (3/1) room temperature, 90 min 10% *Si*-DMAP		75%	14%
		CH_2Cl_2 room temperature, 24 h 10% *Si*-DMAP		74%	37%
		No solvent room temperature, 96 h 24% *Si*-DMAP		71%	58%
		THF/H_2O (3/1) room temperature, 96 h 19% *Si*-DMAP		63%	15%

[a]Yield determined from the isolated product

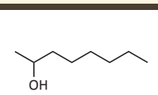
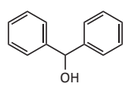
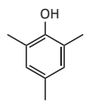
SiliaBond DMAP for Acylation Reactions (*Acylation of 1-phenyl-1-propanol*)

General procedure

A mixture of 2 mmol of alcohol, acetic anhydride (*1.3 eq.*), triethylamine (*1.5 eq.*), and 5% SiliaBond DMAP in 5 mL CH_2Cl_2 was stirred at room temperature for 90 min. The reaction was quenched by the addition of 0.5 mL of methanol, diluted with Et_2O, and washed twice with saturated aqueous $NaHCO_3$ and once with brine. After drying over Na_2SO_4, the solution was filtered and evaporated to give a colorless oil in quantitative yield.



Acylation Results

Alcohol	Catalyst	Anhydride	Reaction Conditions	Conversion[a]
	None	1.4 eq. Ac_2O	18 h, CH_2Cl_2, room temperature	25%
	5% *Si*-DMAP	1.2 eq. Ac_2O	2 h, CH_2Cl_2, room temperature	> 98%
	None	1.3 eq. Bz_2O	24 h, CH_2Cl_2, room temperature	11%
	5% *Si*-DMAP	1.3 eq. Bz_2O	24 h, CH_2Cl_2, room tremperature	91%
	None	1.3 eq. Ac_2O	18 h, CH_2Cl_2, room temperature	50%
	5% *Si*-DMAP	1.3 eq. Ac_2O	40 min, CH_2Cl_2, room temperature	> 98%
	None	1.3 eq. Bz_2O	18 h, CH_2Cl_2, room temperature	29%
	5% *Si*-DMAP	1.3 eq. Bz_2O	2 h, CH_2Cl_2, room temperature	91%
	None	1.3 eq. Ac_2O	19 h, CH_2Cl_2, room temperature	18%
	5% *Si*-DMAP	1.3 eq. Ac_2O	40 min, CH_2Cl_2, room temperature	> 98%
	None	1.3 eq. Bz_2O	24 h, CH_2Cl_2, room temperature	6%
	5% *Si*-DMAP	1.3 eq. Bz_2O	24 h, CH_2Cl_2, room temperature	88%
	None	1.3 eq. Ac_2O	3 h, CH_2Cl_2, room temperature	89%
	5% *Si*-DMAP	1.3 eq. Ac_2O	25 min, CH_2Cl_2, room temperature	> 99%
	None	1.3 eq. Bz_2O	4 h, CH_2Cl_2, room temperature	63%
	5% *Si*-DMAP	1.3 eq. Bz_2O	4 h, CH_2Cl_2, room temperature	94%
	None	1.3 eq. Ac_2O	24 h, PhH, 80°C	49%[b]
	5% *Si*-DMAP	1.3 eq. Ac_2O	24 h, PhH, 80°C	80%[b]

[a]Conversion determined from the isolated product, [b]Determined by GC-FID

Silia*Bond* Tosic Acid in Fischer-Speier Esterifications

The Fischer-Speier reaction is a classic organic process where a carboxylic acid is reacted with an alcohol in the presence of an acidic catalyst to form an ester. All carboxylic acids and only primary and secondary aliphatic alcohols can be use in this reaction. The most commonly used catalysts for this reaction are highly toxic such as H_2SO_4, tosic acid and scandium triflate. Also, a large excess of one of the reagents is used to push the equilibrium towards the product.

Silia*Bond* Tosic Acid (*R60530B*)

Loading: 0.8 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

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

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. It will not dissolve in methanol or any other solvents.
Silia*Bond* Tosic Acid used as an acid catalyst for Fischer-Speier estherification provides excellent conversion.

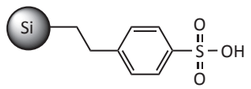
Solvent compatibility

- All solvents, aqueous and organic

Prolonged storage

- Keep dry

Tosic Acid (*Si-SCX*)



Silia*Bond* Tosic Acid for Fischer-Speier Esterifications

General procedure

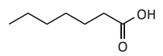
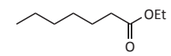
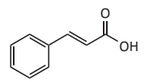
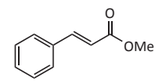
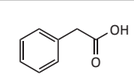
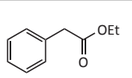
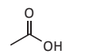
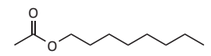
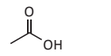
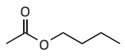
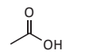
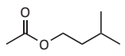
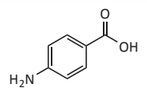
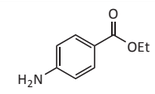
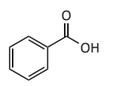
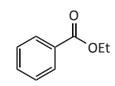
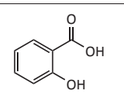
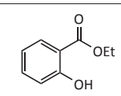
Method A

1.5 mmol of carboxylic acid was added to a mixture of alcohol (*10 mL*) and Silia*Bond* Tosic Acid (*0.1 eq.*). The reaction mixture was maintained at reflux under magnetic agitation for 16 h.

Method B

In a 250 mL round-bottom flask with a magnetic stirrer and a Dean-Stark apparatus, 16.3 mmol of a carboxylic acid was added to alcohol (*4 eq.*) and Silia*Bond* Tosic Acid (*0.1 eq.*). The mixture was then heated to reflux for 20 to 24 h under magnetic agitation.

Fischer-Speier Esterification Results

Alcohol	Carboxylic Acid	Method	Ester	Conversion[a]
Ethanol		A		100%
Methanol		A		98%
Ethanol		A		100%
1-Octanol		A		100%
1-Butanol		A		100% (99%)[b]
3-Methylbutanol		A		100%
Ethanol		A (72 h)		40%[c]
Ethanol		B		94%[c]
Methanol		B		89%[c]

[a]Conversion determined by GC-MS, [b]*Si*-SCX reused 3 times, [c]Conversion determined from the isolated product

Silia*Bond* TBD for Williamson Etherifications

The Williamson etherification is a standard reaction to synthesize asymmetric ethers from alcoholates, prepared from primary and secondary alcohols or phenols with base, in the presence of primary alkyl halides. Because of the high reactivity of alcoholates, they need to be produced during the reaction by strong bases.

Silia*Bond* TBD (*R68530B*)

Loading: 0.9 mmol/g	Endcapping: Yes	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

Silia*Bond* TBD (*Si-TBD*)

Silia*Bond* TBD is a silica-bound bicyclic guanidine moiety that is sufficiently basic to deprotonate moderately acidic hydrogens. It is most commonly used in Williamson ether synthesis.

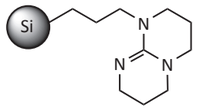
Solvent compatibility

- All solvents, aqueous and organic

Prolonged storage

- Keep cool (< 8°C) and dry, store under argon

TBD (*Si-TBD*)

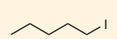
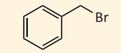
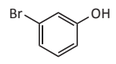
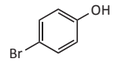
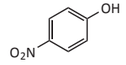
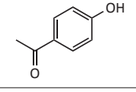
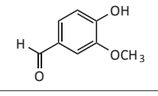
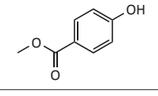
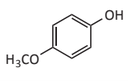


Silia*Bond* TBD for Williamson Etherifications

General procedure

0.15 mmol of alcohol was added to 4 mL of acetonitrile and Silia*Bond* TBD (*0.3 eq.*). The solution was stirred for 1 h at room temperature. Next, 0.12 mmol of the alkyl halide was transferred to the reaction mixture, which was again stirred for 16 h at 60°C. Finally, the mixture was filtered and washed with 2 mL of acetonitrile. Conversion was measured by GC-MS.

Williamson Etherification Results

Alcohol	Alkyl Halide 	Alkyl Halide 
	83%	89%
	89%	88%
	81%	88%
	80%	80%
	59%	86%
	79%	88%
	87%	94%
	78%	86%
	76%	75%

Silia*Bond* Aluminum Chloride Used as a Catalyst for Friedel-Crafts Alkylations and Acylations

For decades, sulfonated linear alkylbenzenes (*LABs*) have been among the most prolific detergents. LAB synthesis is carried out by Friedel-Crafts alkylation of benzene by linear olefins using hydrogen fluoride or aluminum chloride as catalyst. The use of these catalysts presents severe problems, however. For example, aluminum chloride is difficult to separate after reaction and produces a large amount of waste effluent.

Silia*Bond* Aluminium Chloride (*R74530B*)

Loading: 1.6 mmol/g	Endcapping: No	Category: Reagent	Format: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 10kg, 25kg, ..., Multi-Ton

Description

Silia*Bond* Aluminum Chloride (*Si-AlCl$_x$*)

Silia*Bond* Aluminum Chloride 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]

- *It is a milder Lewis acid. $AlCl_3$ is so reactive that it often lacks selectivity and causes the formation of unwanted by-products.*
- *The steric bulk of the silica reduces over alkylation and increases shelf life.*

  Execution of the reaction is easier. The reagent is removed by a simple filtration, avoiding the destructive water quench which produces large amounts of hazardous waste.

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.

Solvent compatibility

- All anhydrous organic solvents

Prolonged storage

- Keep cool (*< 8°C*) and dry, store under argon

Aluminum Chloride (*Si-AlCl$_x$*)

[1] *Acc. Chem. Res.*, **2002**, *35*, 791

[2] *Org. Process Res. Dev.*, **1998**, *2*, 221

[3] *J. Catal.*, **2000**, *195*, 237

[4] *J. Catal.*, **2000**, *195*, 412

[5] *Chem. Rev.*, **2003**, *103*, 4307

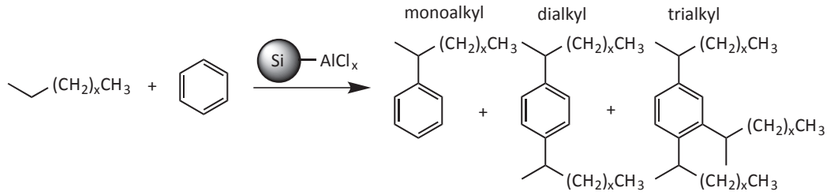
[6] *Tetrahedron*, **2003**, *59*, 1781

Silia*Bond* Aluminum Chloride as a Catalyst for Friedel-Crafts Alkylations

General procedure

Silia*Bond* Aluminum Chloride (*0.03 eq.*) is stirred into anhydrous benzene (*Typical reaction solvent volume: 5 mL/g of Silia*Bond* Aluminium Chloride*). Add the alkene (*1.0 eq.*) slowly (*a small exothermic reaction should be observed*).

After the addition is completed, remove the catalyst by filtration.



Friedel-Crafts Alkylation Results

Alkene	Catalyst	Alkene Conversion[a]	Selectivity Towards Alkylbenzene		
			% Mono	% Di	% Tri
1-Hexene	$AlCl_3$	100%	58.6	31.1	10.3
1-Hexene	*Si*-$AlCl_x$	100%	71.0	28.0	1.0
1-Decene	$AlCl_3$	100%	68.5	22.5	9.0
1-Decene	*Si*-$AlCl_x$	100%	80.0	20.0	0

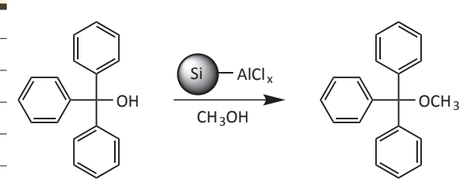
[a]Conversion determined by GC-MS

Silia*Bond* Aluminum Chloride as Catalyst for Friedel-Crafts Acylation

Friedel-Crafts Acylation Results

Alcohol	Catalyst	Conversion[a]
Triphenylmethanol	*Si*-$AlCl_x$	95%
	Polymer-$AlCl_3$	81%
Tert-Butyl Alcohol	*Si*-$AlCl_x$	60%
	Polymer-$AlCl_3$	0%
Benzyl Alcohol	*Si*-$AlCl_x$	40%
	Polymer-$AlCl_3$	0%

[a]Conversion determined by ^{1}H NMR



SiliaBond Reagents and Scavengers for Typical Coupling Reactions

Coupling Reactions	
Reaction	**Reagent / Scavenger**
Amide Coupling	
with acid chlorides and amines	SiliaBond Carbodiimide
with acids and amines	SiliaBond Dichlorotriazine SiliaBond Amine (scavenger) - removes excess acid chloride SiliaBond Isocyanate/SiliaBond Tosic Acid - remove excess of amine
using HOBt or pentafluorophenol	SiliaBond Carbonate - removes excess of HOBt
Buchwald Amination	SiliaMetS Metal Scavengers - remove palladium SiliaBond Tosic Acid
Heck Coupling	SiliaCat DPP-Pd, SiliaCat S-Pd and SiliaCat Pd^0 (*catalyst*) SiliaMetS Metal Scavengers - remove palladium
Sonogashira Coupling	SiliaCat DPP-Pd, SiliaCat S-Pd and SiliaCat Pd^0 (*catalyst*) SiliaMetS Metal Scavengers - remove palladium, copper
Stille Coupling	SiliaCat DPP-Pd, SiliaCat S-Pd and SiliaCat Pd^0 (*catalyst*) SiliaMetS Metal Scavengers - remove palladium, tin
Suzuki Coupling	SiliaCat DPP-Pd, SiliaCat S-Pd and SiliaCat Pd^0 (*catalyst*) SiliaBond Carbonate - removes excess of boronic acid SiliaMetS Metal Scavengers - remove palladium
Kumada Coupling	SiliaCat DPP-Pd, SiliaCat S-Pd and SiliaCat Pd^0 (*catalyst*) SiliaMetS Metal Scavengers - remove palladium
Negishi Coupling	SiliaCat DPP-Pd, SiliaCat S-Pd and SiliaCat Pd^0 (*catalyst*) SiliaMetS Metal Scavengers - remove metal residue

SiliaBond Reagents and Scavengers for Common Organic Reactions

Common Organic Reactions	
Reaction	**Reagent/Scavenger**
Acylation/Esterification	SiliaBond DMAP SiliaBond TBD
Deprotection of Aromatic Ether	SiliaBond Tosic Acid
Ether formation	SiliaBond Aluminium Chloride (*catalyst*) SiliaBond Tosic Acid SiliaMetS Metal Scavengers - remove metal catalyst
Fmoc, Bsmoc Deprotection of Amino Acid	SiliaBond Piperazine (*reagent / scavenger*) - Fmoc deprotection
Friedel-Crafts Alkylation	SiliaBond Aluminium Chloride
Fries Rearrangement	SiliaBond Tosic Acid
Knoevenagel Condensation	SiliaBond Amine SiliaBond Dimethylamine SiliaBond TBD SiliaBond Piperidine SiliaBond Piperazine
Michael Addition	SiliaBond Dimethylamine SiliaBond TBD SiliaMetS Metal Scavengers - remove metal catalyst
Oxidation	
alcohols to acids	SiliaBond Potassium Permanganate
alcohols to ketones or aldehydes	SiliaCat TEMPO SiliaBond Pyridinium Chlorochromate (PCC) SiliaBond Pyridinium Dichromate (PDC)
alkanes	SiliaBond Dimethylamine
Reduction	
with borohydride reducing agents	SiliaBond Tosic Acid - removes excess and spent borohydride
Reductive Amination	SiliaBond Cyanoborohydride SiliaBond Tosic Acid - removes excess of amine
Sulfonamide Synthesis	SiliaBond Dichlorotriazine SiliaBond EDC SiliaBond Amine - removes excess of sulfonyl chloride
Tosylate Formation	SiliaBond Tosyl Chloride
Urea Synthesis	SiliaBond Amine - removes excess of isocyanate
Williamson Ether Synthesis	SiliaBond TBD
Grubbs Metathesis	SiliaMetS Metal Scavengers - remove ruthenium
Sharpless Dihydroxylation	SiliaMetS Metal Scavengers - remove osmium
Catalytic Hydrogenation	SiliaCat DPP-Pd, SiliaCat S-Pd and SiliaCat Pd^0 (*catalyst*)/ SiliaMetS Metal Scavengers - remove metal catalysts
Cyanation	SiliaCat DPP-Pd, SiliaCat S-Pd and SiliaCat Pd^0 (*catalyst*)/ SiliaMetS Metal Scavengers - remove metal catalysts
Hydrogenation	SiliaCat Pt^0 (*catalyst*)
Debenzylation of Benzyl protected Groups	SiliaCat Pd^0 (*catalyst*)
Hydrosilylation	SiliaCat Pt^0 (*catalyst*)

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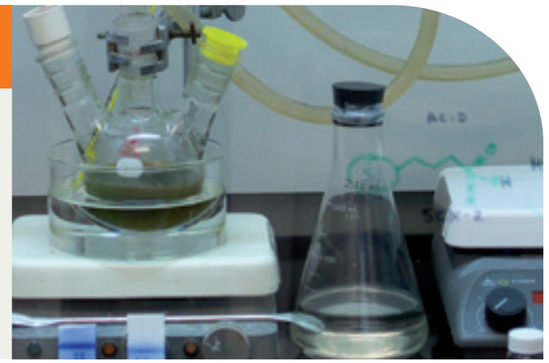
Compatibility With New Technologies

SILICYCLE Inc.
UltraPure SILICA GELS

Flow Chemistry Applications

Using silica-supported products in flow chemistry applications will ensure the following:

- Increase in R&D and manufacturing productivity
- Separation of the catalyst from the products does not require any filtration (*or further handling*)
- Flow-through processes are more reliable and safer than in batch
- Silia*Bond*, Silia*Cat* and Silia*MetS* can be used without degradation



Importance of Flow Chemistry

Flow chemistry is a relatively new technique that 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 catalysts in flow chemistry is even more recent. Supported catalysts are available on different supports such as polymers, charcoal, alumina and silica. They offer many advantages over the traditional homogeneous catalysts, including ease of handling and purification. Silica presents many advantages such as no swelling, good mechanical and thermal stabilities and ease of scalability. SiliCycle has developed innovative silica-based catalysts (Silia*Cat*), reagents (Silia*Bond*) and metal scavengers (Silia*MetS*) that can be used in flow chemistry.

Acylation Reactions Using Silia*Bond* DMAP

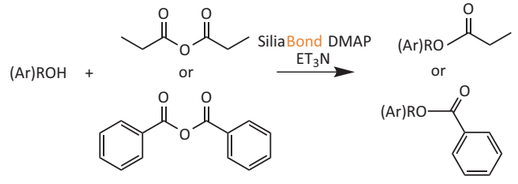
Acylation reactions can generate esters using activated carboxylic acids (*acids chlorides*) and alcohols, even hindered tertiary alcohols.

General Procedure (*conventional - batch*)

Typical reaction: acetylation of 1-phenyl-1-propanol. A mixture of 6 mmol of substrate, 1.5 eq. of acetic anhydride, 1.5 eq. of triethylamine and 5 mol % Silia*Bond* DMAP in 15 ml of CH_2Cl_2 was stirred at room temperature for 90 minutes. The reaction was quenched by the addition of 0.5 mL of methanol, diluted with 25 mL Et_2O, and washed twice with saturated aqueous $NaHCO_3$ and brine. After drying over Na_2SO_4, the solution was filtered and evaporated to give a colorless oil in a quantitative yield.

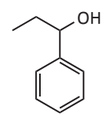
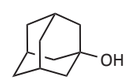
General Procedure (*flow*)

Typical reaction: acetylation of 1-phenyl-1-propanol. A mixture of 4 mmol of substrate, 1.5 eq. of acetic anhydride, 1.5 eq. of triethylamine in 10 mL of CH_2Cl_2 was stirred at room temperature for 5 minutes. Two fractions of 5 mL solution were introduced into the reactor charged with the 9 mol % Silia*Bond* DMAP (0.45 g). Upon completion of the reaction, the mixture was analyzed by GC-MS to determine the conversion.



Acylation Reactions Using Silia*Bond* DMAP (*con't*)

Acylation Reaction Results

Substrate	Reagent	Catalyst (eq.)	Time (h)	Flow Conditions: Flow (µL/min)	Flow Conditions: Vol. Reactor (mL)	Flow Conditions: Res. Time (min)	Conversion (%) (Yield %)
2-Octanol	Ac_2O	5	2	Conventional (Batch)			98 (99)
		9	1.67 0.93	100.0 200.0	0.7	7.0 3.5	100 (99) 98 (99)
	Bz_2O	10	24	Conventional (Batch)			91
		9	6.67 13.3	25.0 12.5	0.7	28 56	93 (95) 95 (97)
	Ac_2O	5	1.5	Conventional (Batch)			98 (99)
		9	3.30 1.67 0.83	50.0 100.0 200.0	0.7	14.0 7.0 3.5	97 (99) 97 (99) 97 (99)
	Bz_2O	5	24	Conventional (Batch)			88
		9	1.67 3.38 6.67	100.0 50.0 25.0	2.38	24 48 96	88 (98) 94 (99) 97 (99)
	Ac_2O	6	24	Conventional (Batch)			67
		9	3.33 6.67 16.67	50.0 25.0 10.0	2.38	48 96 239	27 (97) 40 (97) 61 (95)

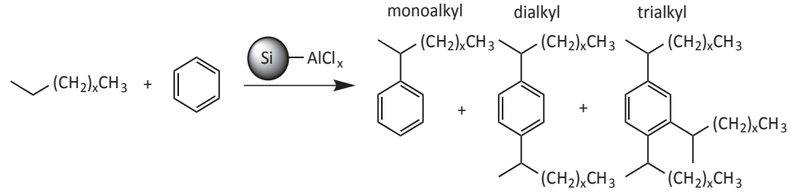
Friedel-Craft Alkylations Using Silia*Bond* Aluminum Chloride

General Procedure (*conventional - batch*)

1 eq. of 1-decene was added slowly (*over 30 min*) to a mixture of anhydrous benzene (*20 eq.*) and 0.02 eq. of Silia*Bond* Aluminum Chloride (*1.67 mmol/g*). After the addition, the catalyst was removed by filtration and the crude product was analyzed by GC/MS.

General Procedure (*flow*)

A mixture of 1 eq. of 1-decene and 20 eq. of anhydrous benzene was pumped in a reactor charged with 0.2 eq. of Silia*Bond* Aluminum Chloride. After completion of the reaction the mixture was analyzed by GC/MS.



Friedel-Craft Alkylation Results

Ratio 1-Decene vs Benzene	Catalyst (eq.)	Time (min)	Flow Conditions: Flow (µL/min)	Flow Conditions: Vol. Reactor (mL)	Flow Conditions: Res. Time (min)	Conversion & Selectivity (%): Conv.	Mono	Di	Tri
1 : 20	0.2	30	Conventional (Batch)			100	85	15	0
	0.2	20	250	0.76	3	100	89	11	0

Knoevenagel Condensations using Silia*Bond* Piperidine

The Knoevenagel condensation between carbonyl compounds and methylene malonic esters produce several important products, including nitriles used in anionic polymerization and unsaturated ester intermediates employed in the synthesis of several therapeutic drugs. Alkali metal hydroxides, pyridine and piperidine are the traditional catalysts used in these reactions

General Procedure (*conventional - batch*)

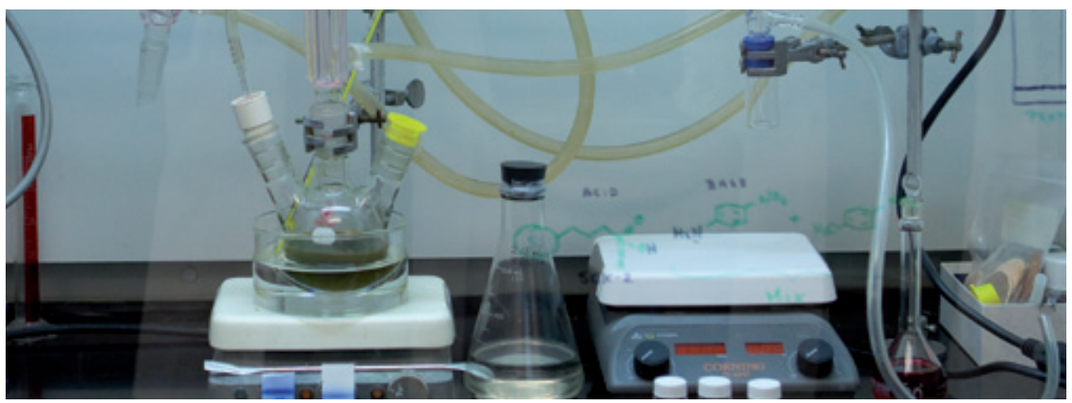
A mixture of 2 mmol of benzaldehyde, 1.5 eq. of ethylcyanoacetate and 10 mol % of Silia*Bond* Piperidine in 15 mL of toluene were stirred at 110°C for 20 h. The reaction mixture was filtered, and the solvent was evaporated. The crude product obtained was analyzed by GC/MS.

General Procedure (*flow*)

The reactor was charged with 10 mol % of Silia*Bond* Piperidine (*1.36 g*) and heated at 110°C using toluene as solvent. A mixture of 15 mmol of benzaldehyde, 1.5 eq. of ethylcyanoacetate in 110 mL of toluene was stirred at room temperature for 5 minutes. The mixture was then introduced in a glass bottle directly connected to the pump. Upon completion of the reaction, the reaction mixture was evaporated and the crude product analyzed by GC/MS to determine the conversion ratio.

Knoevenagel Condensation Reaction Results

Entry	Catalyst (mol %)	Time (h)	Flow Conditions			Conversion (%) (Yield %)
			Flow (µL/min)	Vol. Reactor (mL)	Residence Time (min)	
1	10	20	Conventional (Batch)			80 (98)
2	55	1.67	50	0.7	14	82 (99)
3	4	25	50	0.7	14	75 (99)
4	10	19	100	2.4	24	90 (100)



Deprotection of Methoxymethyl Groups using Silia*Bond* SCX

MOM groups are used as a protecting group for alcohols. The group can be removed using an acid. In this application Silia*Bond* Tosic Acid (SCX) has been used to deprotect alcohols previously protected by chloromethyl ether.

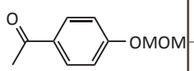
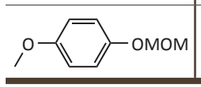
General Procedure (*conventional - batch*)

A mixture of 2.5 mmol of 1-(4-(MOM)phenyl)ethanone and 0.05 eq. of Silia*Bond* Tosic Acid (0.8 mmol/g) in 10 mL of toluene/H_2O (10:0.5) was stirred at 65°C for 4 h. The reaction mixture was filtered and the solvent was evaporated. The crude product obtained was analyzed by GC/MS.

General Procedure (*flow*)

The reactor was filled with the desired amount of Silia*Bond* Tosic Acid and heated at room temperature or at 65°C using toluene as solvent. A solution of 12.5 mmol of 1-(4-(MOM)phenyl)ethanone in 50 mL of toluene was introduced in a glass bottle connected directly to a pump. A second glass bottle, connected to another pump, was filled with solvent. The flow for the two pumps was different: 100 µL/min for the first pump and 20 µL/min for the second pump. Upon completion of the reaction, the mixture was evaporated and the crude product was analyzed by GC/MS.

Deprotection of Methoxymethyl (*MOM*) Group using Silia*Bond* SCX Results

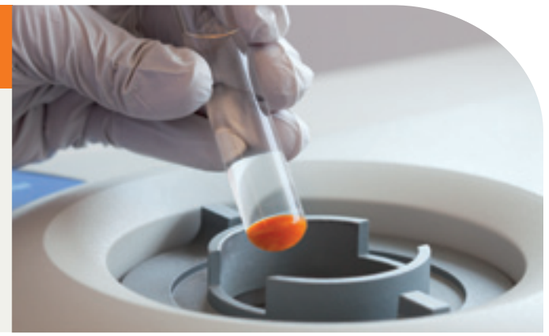
Substrate	Catalyst (eq.)	Time (h)	Solvent	Flow Conditions			Conversion (%) (Yield %)
				Flow (µL/min)	Vol. Reactor (mL)	Res. Time (min)	
	0.5	2	Toluene/MeOH (0.25M)	Conventional (Batch)			100 (90)
	0.05	4	Toluene/MeOH (0.25M)	Conventional (Batch)			83 (93)
	0.44	1.67	Toluene/MeOH (0.25M)	100	2.4	24	100 (100)
	0.1	8.34	Toluene/MeOH (0.25M)	120	2.4	17.5	99 (100)
	0.5	3.33	CH_2Cl_2 (0.1 M)[a]	50	2.4	48	91 (98)
	0.5	1.67	CH_2Cl_2 (0.1 M)[a]	100	2.4	24	90 (97)
	0.35	1.67	Toluene/MeOH (0.25M)	100	2.4	24	88 (99)

[a] at RT.

Microwave Applications

Using silica-supported products in microwave applications will ensure the following:

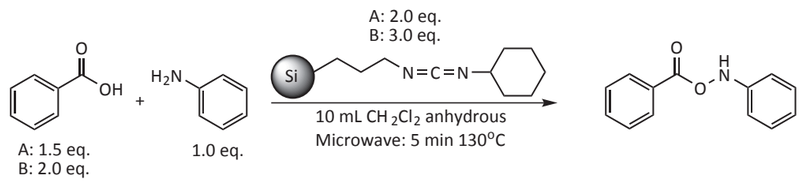
- Faster kinetics: only a few minutes per reaction
- Higher yields and excellent purities
- Compatibility with many solvents
- Silia*Bond*, Silia*Cat* and Silia*MetS* can be used without degradation
- Wide variety of reactions and applications



Importance of Microwave Assisted Synthesis

In recent years, microwave synthesizers have taken organic chemistry by storm. Fast kinetics, higher yields, excellent purities, wide compatibility of solvents and their applicability to a variety of reactions and applications, make them very important tools in the laboratory. After their introduction, chemists started to use supported reagents for solution-phase synthesis. The polymer-supported reagents commonly used, although very useful, have drawbacks in microwave synthesizers, namely swelling and heat instability. The high temperatures generated inside these synthesizers put stress on the resins. Also, because of the small reaction volumes, the swelling of the resins can be problematic. Silica-based products on the other hand, do not suffer from such shortcomings. They are heat resistant and they do not swell. In the following pages, we present different reactions (*amide synthesis, reductive amination, Henry reaction*) using Silia*Bond* Reagents as well as an electrophile and nucleophile that demonstrate the effectiveness of these reagents for microwave applications.

Amide Couplings using Silia*Bond* Carbodiimide

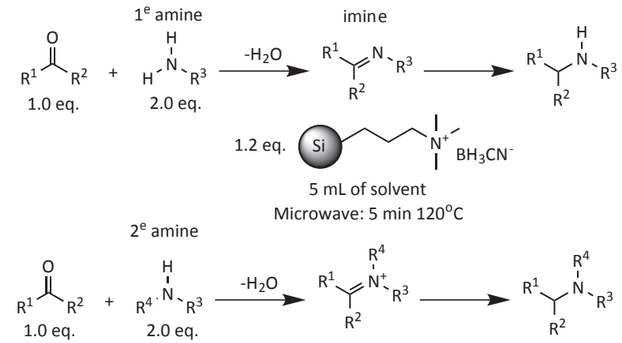


Amide Coupling Yield[a] (*Purity*)[b] in %		
Method	Microwave	Bulk (RT, 24 h)
A	73.3 (88.0)	52.7 (99.5)
B	94.9 (95.0)	80.1 (98.1)

[a]Determined from GC-FID,
[b]Refers to the isolated product

Reductive Aminations using Silia*Bond* Cyanoborohydride

General Procedure



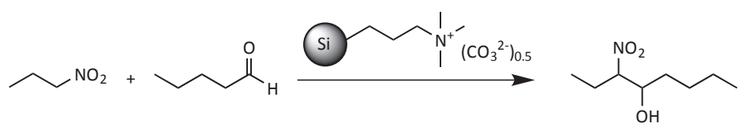
Acylsulfonamide Synthesis Conversion (%) Results				
Amine	Carbonyl	Microwave[a] 5 min	Bulk (RT, 2.5 eq. *Si*-CBH)[a] 1 h	24 h
Piperidine	Benzaldehyde	> 99	80	> 99
N-Benzylmethylamine	Benzaldehyde	> 99	97	> 99
3-Phenyl-1-propylamine	Cyclohexanone	> 99	88	87

[a]Conversion determined by GC-FID

Henry Reactions using Silia*Bond* Carbonate

General Procedure

1-nitropropane (*1 eq.*) was added to a solution containing THF (*5 mL*) and valeraldehyde (*1 eq.*). To this reaction mixture, Silia*Bond* Carbonate (*0.1 eq.*) was added and maintained at 100°C for 10 min in the microwave. The reaction mixture was filtered and washed with THF, and the crude product was evaporated. Finally, pure product was obtained after flash chromatography purification using a mix of hexane/ethylacetate (*80/20*).



Conversion: 89%

Drug Purification & Analysis

SiliaMetS®
Metal Scavengers

SILICYCLE inc.
UltraPure SILICA GELS

Metal Scavenging with Silia*MetS*®

Silia*Bond* Metal Scavengers BECOME Silia*MetS* Metal Scavengers!

Same Efficient & High Quality Metal Scavenger Products, Brand New Look!

SiliCycle has developed a new look and a new brand for our Metal Scavengers. These products, known as Silia*Bond* Metal Scavengers (*i.e.: Silia Bond Thiol*), are now named Silia*MetS* (*i.e, Silia MetS Thiol*) with a new color code. We have updated our branding to give more visibility to our metal removal solutions. This new branding will help differentiate these products from other functionalized silica gels available (*reagents and other bonded phases*).

Although we changed our branding from Silia*Bond* Metal scavengers to Silia*MetS*, no change has been made to the products themself; you will still be purchasing the same quality products that you have been enjoying for years.

SiliCycle is THE world leader and THE pioneer in metal scavenging solutions. Reasons to choose us:

- Over 12 years of know-how in silica-grafting and metal scavenging technology
- Strong, extensive, and confidential technical support and scientists to help you
- Broadest portfolio of scavengers (*wide variety of ligands*) and applications developed
- Wide range of formats for all purification scales; from laboratory to plant scale purifications
- Cited in many external publications (*and patents*) used by satisfied customers

Introduction

In recent years, the time pressure associated with quickly bringing candidate drugs to market has increased the number of transition metal-catalyzed reactions progressing from lead optimization to early scale-up. The removal of post-reaction metal residues has become a major issue in the pharmaceutical industry. Purification of APIs from residual metal catalyst by traditional methods (*chromatography, activated carbon, distillation, etc.*) often leads to problems such as high costs, time loss, low efficiency, and reduced API yields. To overcome these limitations, SiliCycle has developed **Silia*MetS* Metal Scavengers**, a range of products that have significantly changed how chemists purify APIs.

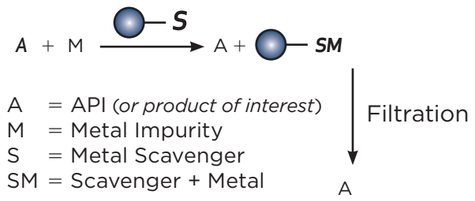
Silica-based metal scavengers have been proven to be the purification method of choice used by several companies from various industries. Take a look at the section "Customer Success Stories with **Silia*MetS***" to read about our satisfied customers. With the silica matrix advantages over polymers (*no swelling, more general solvent compatibility, higher mechanical and thermal stability, easily scalable applications and availability of different formats, including SPE, flash cartridges, and bulk*) and SiliCycle's expertise in grafting technology, **Silia*MetS*** are the solution of choice for metal removal without contamination of drug candidates. **Silia*MetS*** are highly selective and offer a cost-effective alternative for metal removal.

This section includes useful information and tips on **Silia*MetS*** (*properties and selection chart*) uses, experimental procedures, and results.

What are Silia*MetS* Metal Scavengers?

Silia*MetS* Metal Scavengers are functionalized silica gels designed to react and bind excess metal complexes. The process for using scavengers is outlined in the scheme below.

What is a Supported Metal Scavenger?



To be effective, the Metal Scavengers need the ability and inherent functionality to remove metals in their various oxidation states from the reaction mixture. For example, upon completion of a palladium metal-catalyzed reaction, the metal residue contained in the reaction can exist in both Pd (0) and Pd (II).

Silia*MetS* - Regulatory Information

For many years, Silia*MetS* Metal Scavengers have been used in pilot plants by GMP pharmaceutical, biotechnology, and fine chemical industries as well as contract research and manufacturing organizations. They have run their own analysis proving Silia*MetS* Metal Scavengers can safely be used without compromising the purity of the material by leaching of the silica-supported product.

Thus, SiliCycle is committed to high quality standards and always striges to provide defect-free products. In doing so, all products are manufactured in an ISO 9001:2008 compliant facility and subjected to a stringent quality control. Every lot needs to meet the quality specifications and a sample from every batch is kept for subsequent analysis. All products are shipped with the following information:

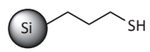
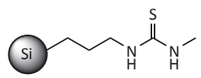
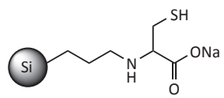
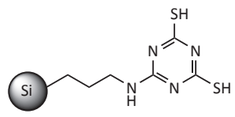
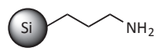
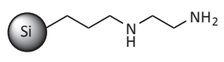
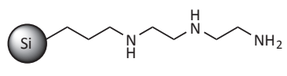
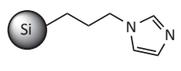
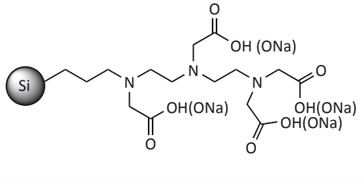
- Certificate of Analysis
  - Purity (*Leachables and extractables*)
  - Molecular loading
  - Surface Coverage
  - Volatile Content
- Material Safety Data Sheets (*MSDS*)
- BSE/TSE Declaration (*no animal origin*)
- Relevant Technical Information

Need specific regulatory files? SiliCycle can work with you to fill your requirements and provide you custom regulatory documentation including specific analytical tests in line with your needs.

Silia*MetS* Product Range

SiliCycle, a leader in functionalized silica gels, has developed a wide range of scavengers to remove a variety of metals at competitive prices.

Silia*MetS* Metal Scavengers Portfolio

Silia*MetS*	Product Number	Structure	Brief Description	Metals Removed	Silia*MetS* Typical Characteristics: Color	Endcapping	Molecular Loading	Typical Tap Density	Solvent Compatibility	Prolonged Storage	Silia*MetS*
Silia*MetS* Thiol	R51030B		**Silia*MetS*** Thiol is our most versatile and robust metal scavenger for a variety of metals under a wide range of conditions. It has been used in pharmaceutical processes up to production scale.	Ag, Hg, Os, Pd^{2+}, Pd^{0} & Ru Cu, Ir, Pd, Rh^{1+}, Rh^{2+}, Rh^{3+}, Sc, Sn	White	Yes	1.20 mmol/g	682 g/L	All solvents, aqueous and organic	Keep dry	**Silia*MetS*** Thiol
Silia*MetS* Thiourea	R69530B		**Silia*MetS*** Thiourea is a versatile metal scavenger for all forms of palladium and is widely used in the pharmaceutical industry. Once complexed with a transition metal, it has been reported to be an effective catalyst.	Pd^{2+}, Pd^{0} Ag, Cu, Fe, Os, Rh^{1+}, Rh^{2+}, Rh^{3+}, Sc, Sn	Off-white	Yes	1.20 mmol/g	767 g/L	All solvents, aqueous and organic	Keep dry	**Silia*MetS*** Thiourea
Silia*MetS* Cysteine	R80530B		**Silia*MetS*** Cysteine is the silica bound equivalent of the amino acid cysteine. It is a versatile scavenger for a variety of metals and the preferred metal scavenger for tin residues. By attaching the molecule to the backbone via the amino group, the thiol group remains free and accessible for higher metal scavenging efficiency.	Cd, Fe, Ir, Os, Ru, Sc & Sn Ca, Cr, Cs, Cu, La, Mg, Pd^{2+}, Pd^{0}, Pt, Rh^{+1}, Rh^{+2} & Zn	Orange	Yes	0.30 mmol/g	665 g/L	All organic solvents	Keep dry under argon	**Silia*MetS*** Cysteine
Silia*MetS* DMT	R79030B		**Silia*MetS*** DMT is the silica-bound equivalent of 2,4,6-trimercaptotriazine (*trithiocyanuric acid, TMT*). It is a versatile metal scavenger for a variety of metals and the preferred metal scavenger for ruthenium catalysts and hindered Pd complexes (*i.e. Pd(dppf)*Cl_2).	Ir, Ni, Os, Pd^{2+}, Pd^{0}, Pt, Rh^{+1}, Rh^{+2}, Rh^{+3} & Ru Cd, Co, Cu, Fe, Sc & Zn	Light brown	Yes	0.50 mmol/g	732 g/L	All solvents, aqueous and organic	Keep dry	**Silia*MetS*** DMT
Silia*Bond* Amine	R52030B		Better known for their electrophile scavenging efficiency, and their base reagent quality, **Silia*MetS*** Amine, Diamine and Triamine are also proven scavengers for metals. They are very useful for scavenging Pd, Pt, Cr, W and Zn.	Cd, Cr, Pt, Rh^{+1} & Rh^{+2} Co, Cu, Fe, Hg, Pb, Pd^{+2}, W & Zn	Off-white	Yes	1.20 mmol/g	700 g/L	All solvents, aqueous and organic	Keep cool (<8°C) and dry	**Silia*Bond*** Amine
Silia*MetS* Diamine	R49030B			Cr, Pd^{2+}, Pd^{0}, & Pt Cd, Co, Cu, Fe, Hg, Ni, Pb, Ru, W & Zn	Off-white	Yes	1.20 mmol/g	728 g/L	All solvents, aqueous and organic	Keep cool (<8°C) and dry	**Silia*MetS*** Diamine
Silia*MetS* Triamine	R48030B			Cr, Pb, Pd^{2+}, Pd^{0} & Pt Co, Cu, Fe, Ni, Ru, W & Zn	Off-white	Yes	1.20 mmol/g	736 g/L	All solvents, aqueous and organic	Keep cool (<8°C) and dry	**Silia*MetS*** Triamine
Silia*MetS* Imidazole	R79230B		**Silia*MetS*** Imidazole is a versatile metal scavenger for a variety of metals including, Cd, Co, Cu, Fe, Ni, Pd, Os, and Rh, under a wide range of conditions and the preferred metal scavenger for iron catalysts.	Cd, Co, Cu, Fe, Ni, Os, W & Zn Cr, Pd^{2+}, Pd^{0}, Rh^{1+} & Rh^{2+}	Off-white	Yes	1.20 mmol/g	681 g/L	All solvents, aqueous and organic	Keep dry	**Silia*MetS*** Imidazole
Silia*MetS* TAAcOH	R69030B		**Silia*MetS*** TAAcOH & TAAcONa (*Si-Triaminetetraacetic Acid or Sodium Salt*) are supported versions of EDTA in their free and sodium salt forms. These two products are effective metal scavengers for Ca, Mg, Li, Ir, Cs, Os, Sn, Pd, Ni and Cu.	Co, Ni, Os & Sc Cr, Cs, Fe, Pd^{2+}, Pd^{0}, Rh^{1+}, Rh^{2+} & Sn	Off-white	No	0.40 mmol/g	635 g/L	All solvents, aqueous and organic	Keep dry	**Silia*MetS*** TAAcOH
Silia*MetS* TAAcONa	R69230B		**Silia*MetS*** TAAcOH is effective for metals in low or zero oxydation states, compared to **Silia*MetS*** TAAcONa which is useful for metals in higher oxydation states (*2+ or higher*).	Cd, Cs, Cu, Fe, Ir, La, Li, Mg, Ni, Os, Rh^{3+}, Sc, & Sn Cr, Pd^{2+}, Pd^{0}, Rh^{1+}, Rh^{2+} & Zn	Off-white	No	0.40 mmol/g	712 g/L	All solvents, aqueous and organic	Keep dry	**Silia*MetS*** TAAcONa

All **Silia*MetS*** are made of standard flash silica gel, namely 40 - 63 microns, 60 Å.

Preferred SiliaMetS Metal Scavengers for these metals ***Also Scavenges these metals***

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Features & Benefits of Silia*MetS* Metal Scavengers

Silia*MetS* Metal Scavengers are functionalized silica gels designed to react and bind excess metal complexes. The process for using scavengers is outlined in the scheme on page 87.

Features & Benefits of Silia*MetS*

Features	Benefits
No leaching	No API contamination by the metal scavenger
Very High Purity	Each Silia*MetS* product manufactured is submitted to very rigorous quality control in order to provide customers with default-free products and ensure 100% satisfaction
High Selectivity	Total recovery of the API
Wide Range of Metal Species (*various oxidation state*)	Efficient for a wide range of metal catalysts
Fast Kinetics	Even at room temperature
Cost Efficient	Low cost per gram of metal scavenged Less solvent used
Solvent Compatibility	Can be used in any solvent, aqueous (*pH 2 to 12*) and organic
New Technologies Compatibility	Suitable for use in microwave synthesizers and flow chemistry
Excellent Stability (*Thermally and Mechanically*)	Works well with overhead stirring Can withstand very high temperatures
Ease of Use & Scalable	No swelling or static charge Remove easily by a simple filtration Scalable from mg up to multi-ton scale
Various Formats	Amenable to use in Silia*Sep* & Silia*Prep* Cartridges
Controlled Loading	Consistent and accurate loading insure lot-to-lot reproducibility
Available in Bulk Quantities	Available in large quantities and always in stock

Metal Scavenging Screening Service

CONFIDENTIALITY ASSURED

Having a problem removing any residual metal catalyst? Contact us to take advantage of SiliCycle's expertise in metal removal. Our R&D team can find the optimal conditions for you.

Metal Scavenger Screening Services are innovative as they provide an on-hand solution to the pharmaceutical and manufacturing industries. Working with the product that needs to be free of residual metals and the restricted conditions that can be used with the compound (*i.e, solvent, temperature*), SiliCycle's **Metal Scavenger Screening Service** will quickly develop the most efficient metal scavenging process providing both time and cost savings. Confidentiality is assured, as in most cases the solution involves working with API and other patented materials, and easy technology transfer is guaranteed.

Take the step many major pharmaceutical companies have, and contact us to discuss how we can help you to reach your metal purity goals.

Many screening services adapted to your needs & budgets are available.

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Silia*MetS* - Typical Experimental Procedures

Screening in Batch Reactor Mode (*bulk*)

To select the best scavenger for initial screening experiments, do the following steps for each Silia*MetS* Metal Scavengers included in the kit. Use 4-8 molar equivalents of each Silia*MetS* in respect to the residual metal concentration.

1. Dissolve the crude product to be treated in a suitable solvent (*or use directly the crude reaction mixture*) and prepare vials containing the same solution volume.
2. Directly, add each Silia*MetS* included in the kit to these vials.
   Note: no pre-wetting of the Silia*MetS* is required. See “Determing the Optimal Amount of Silia*MetS* to use” at page 96.
3. For initial tests, stir the solution for at least one hour at room temperature.
4. Scavenging progress can be followed by normal analytical techniques. The scavenging progress can be estimated by looking at the color of the solution as demonstrated in the figure (*right*). When the scavenging is almost complete, the solution is less colored and Silia*MetS* becomes colored. In some occasional cases, if all the samples are still coloured, try one or all of the following: let them react for a longer period of time; add more equivalents of the Silia*MetS*, increase the temperature of the reaction.

5. At the end of the scavenging, filter off the Silia*MetS* using a fritted funnel or filtration device.
6. Wash the Silia*MetS* with additional solvent for total recovery of the API (*or compound of interest*) and concentrate the solution under vacuum.
7. Analyze the residual metal concentration of each vial to identify the most efficient Silia*MetS* Metal Scavenger

   Note: you can choose more than one scavenger.
8. If you are satisfied with the scavenging efficiency of the best Silia*MetS*, direct scale-up is possible. Otherwise, scavenging optimization can be done with Silia*MetS* identified in #7 (*see next section*).

Screening with Silia*MetS* Fixed Bed Mode (*SPE or Flash Cartridges*)

Silia*MetS* fixed bed formats are a great alternative for metal removal and are directly scalable. Initial screening investigations can be done using Silia*Prep* 2g/6mL SPE cartridges.

1. Condition the cartridge with 3-4 cartridge volume using the same solvent as the solution to be treated.
2. Add the solution containing the API and the metal to the top of the cartridge and let it pass through the cartridge under gravity.

   Note: if needed, a slight positive pressure on the top of the cartridge or a light vacuum can be applied to speed up the flow rate.

3. As shown to the right, a dark coloured band will be observed on the top of the silica bed most of the time.
4. If the residual solution is still coloured, multiple passes through the same cartridge can be done.
5. Once the scavenging is completed, wash the cartridge using at least 3 column volumes of solvent to insure total API (*or compound of interest*) recovery.

   Note: in some cases, additional washing may be required.

Silia*MetS* Compatible with New Technologies

Silia*MetS* In Flow Chemistry

Metal scavenging can also be achieved using Silia*MetS* in flow chemistry applications. Simply place Silia*MetS* inside the solid-phase reactors provided with your flow system (*like Syrris Asia® Solid Phase Chemistry Reactors*) and let the solution to be purified flow through these reactors. Multiple reactors can be placed in series and reactors can be heated to obtain optimum scavenging results.

Silia*MetS* In Microwave

Metal removal using Silia*MetS* can also be done under microwave irradiation to provide excellent scavenging efficiency in just minutes. Simply mix the scavenger and the API dissolved in a suitable solvent a microwave tube and set-up the system with the appropriate parameters. Usually, 5 minutes is enough to scavenge all residual metals.

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Experiment Optimization with Silia*MetS*

If, upon completion of the screening procedure, the scavenging is not complete or you wish to either reduce the number of equivalents or the reaction time, optimization steps can be undertaken.

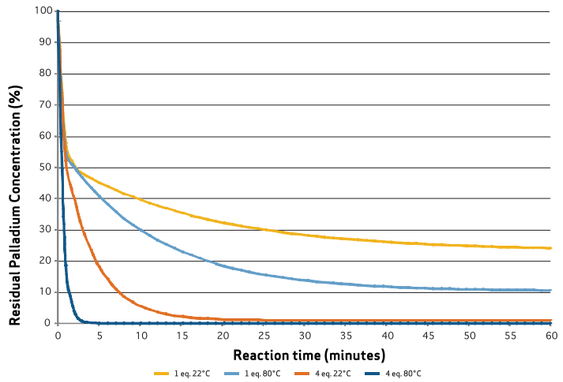
Various parameters can be changed one at a time or simultaneously to improve the metal removal efficiency.

Note: you can mix multiple Silia*MetS* to get superior efficiency.

Number of Silia*MetS* Equivalents

For initial screening experiments we suggest 4-8 molar equivalents be used in respect to the residual metal concentration of each Silia*MetS*. Once the preferred scavenger is identified, further optimization can be done to reduce the number of equivalents used (*typically down to 2-4 equivalents*).

*Graph represents residual concentration (%) of $Pd(OAc)_2$ with Silia*MetS* Thiol in DMF.*



Subsequent Treatments with Silia*MetS*

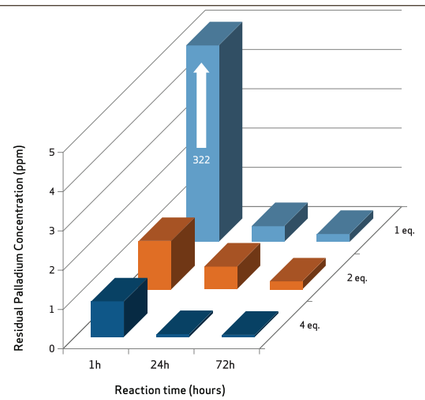
In some cases (*equilibrium process or the presence of multiple species*), multiple treatments with Silia*MetS* is suitable instead of a single treatment with a larger amount.

For optimal results, filtration between each treatment can allow for a higher scavenging efficiency.

Reaction Time

In some cases, where increasing the temperature is impossible, longer contact time with the scavenger can allow higher scavenging efficiency.

*Conditions: $Pd(OAc)_2$, THF, Silia*MetS* Thiol, RT.*

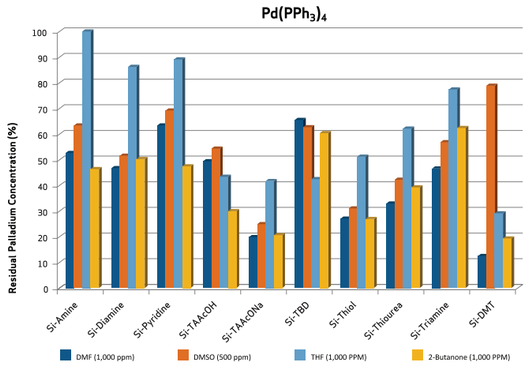


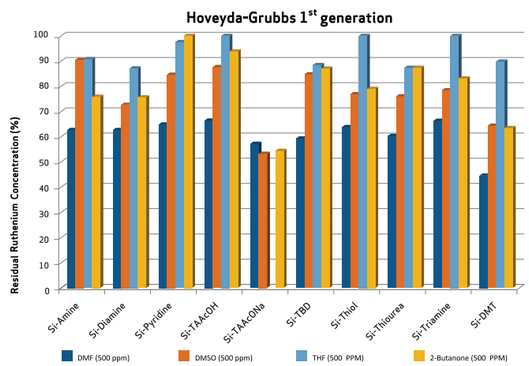
Temperature

In initial screening, we suggest the scavenging experiments be run at room temperature. Usually, metal scavenging is completed after one hour or so. However, when shorter scavenging times are required, higher scavenging rates can be achieved by increasing the temperature. Silia*MetS* can be safely used at elevated temperature without degradation and can be added either at room temperature or directly to a warm solution.

Solvent

Silia*MetS* can safely be used in a wide range of organic and aqueous solvents commonly used in laboratory and in process, such as DMF, DMSO, THF, 2-butanone, alcohols, ethers, chlorinated solvent, etc. As demonstrated in the graphs below, the nature of the solvent does sometimes influence scavenging efficiency. If scavenging or kinetics are too slow, changing solvent or adding a co-solvent should be considered.





Silia*MetS* Format (*Mode Used*)

One advantage of Silia*MetS* is their compatibility with various technologies. They can be used in batch, in fixed bed (*SPE or Flash cartridges*), in flow chemistry, or in microwave. Scavenging efficiency can be improved by changing the mode used.

Mixing Rate

Silia*MetS* are mechanically stable and offer excellent scavenging efficiency in batch processes agitated by overhead and magnetic stirrers, as well as orbital shaking under low to moderate agitation rates.

If required, mixing rates can be increased to get better scavenging results. With faster stirring, you improve Silia*MetS* dispersion in solution.

pH of the Aqueous Solution

When the scavenging is done in aqueous solutions, it is possible to use Silia*MetS* in a pH range of 2 to 12. Depending on the nature of the Silia*MetS*, pH can modify the functional groups present on the scavengers by charging them. Scavenging can be affected (*i.e, amine groups in acidic conditions*).

Determining the Optimal Amount of Silia*MetS*

To get an effective metal removal, the amount of Silia*MetS* Metal Scavenger used is very important. You can determine how much scavenger will be needed by one of two ways:

- from the residual concentration (*more accurate method*)
- from the amount of metal catalyst used (*when the residual metal concentration is unknown*)

From residual metal concentration (*ppm*)

Knowing that the palladium (*Pd*) level in 800 g of material is 500 ppm (*the oxidation state does not affect the calculation*).

Data needed:

- Loading of the scavenger (*Silia*MetS* Thiol*): 1.2 mmol/g
- Metal molecular weight: Ex. Pd = 106.42 g/mol
- Amount of product to be treated : Ex. 800 g
- Residual concentration of metal: Ex. 500 ppm of Pd

1. Determine the amount of palladium to be scavenged

$$\text{Amount of Pd in mg} = \frac{\text{Residual metal concentration} \times \text{Qty of product to be treated}}{1{,}000}$$

$$\text{Amount of Pd in mg} = \frac{500 \text{ ppm} \times 800 \text{ g of product}}{1{,}000} = 400 \text{ mg of Pd in 800 g of product}$$

$$\text{Conversion in mmol of Pd} = \frac{\text{Amount of Pd in mg}}{\text{Metal molecular weight}}$$

$$\text{Conversion in mmol of Pd} = \frac{400 \text{ mg}}{106.42 \text{ g/mol}} = 3.76 \text{ mmol of Pd}$$

2. Calculate the amount of scavenger (*Silia*MetS* Thiol*) to use (*1 equivalent*)

$$\text{Amount of Silia}\textit{MetS}\text{ Thiol to use} = \frac{\text{Number of mmol of metal concentration}}{\text{Silia}\textit{MetS}\text{ Thiol loading}}$$

$$\text{Amount of Silia}\textit{MetS}\text{ Thiol to use} = \frac{3.76 \text{ mmol of Pd}}{1.2 \text{ mmol/g}} = 3.13 \text{ g of Silia}\textit{MetS}\text{ Thiol for 1 eq.}$$

To scavenge 400 mg of palladium, 3.13 g of Silia*MetS* Thiol is needed if using only one equivalent. However, it is highly recommend that a minimum of 4 equivalents be used at first. In this case, the amount of Silia*MetS* Thiol will be 4 times higher (*4 x 3.13 g = 12.52 g*).

Sometimes, the metal residual concentration is unknown. In such a case, the amount (*g*) of palladium to be scavenged can be replaced by the amount of metal catalyst used for the reaction:

From amount of metal catalyst used

Data needed:

- Amount of metal catalyst used: Ex. 10 g of $Pd(PPh_3)_4$
- Metal catalyst molecular weight: $Pd(PPh_3)_4$ = 1,155.56 g/mol

1. Determine the amount of palladium to be scavenged

$$\text{Amount of Pd in mmol} = \frac{\text{Qty of catalyst used for the reaction used} \times 1{,}000}{\text{Metal catalyst molecular weight}}$$

$$\text{Amount of Pd in mmol} = \frac{10 \text{ g of } Pd(PPh_3)_4 \times 1{,}000}{1{,}155.56 \text{ g/mol}} = 8.65 \text{ mmol of Pd (}\textit{max to be scavenged}\text{)}$$

The amount of Silia*MetS* Thiol to be used can then be determined as stated above (*see point 2. above*). In this particular case, one equivalent of Silia*MetS* Thiol corresponds to 7.20 g.

Silia*MetS* Selection Guide

When selecting a metal scavenger, every parameter must be considered: metal catalyst, solvent, residual reagents, by-products, structure of the API (*or molecule of interest*) and temperature. The following table, shown below, helps customers in selecting the most efficient scavenger for a specific metal and application. However, since some parameters may affect the efficiency of the scavenging, we highly recommend performing a preliminary screening experiment using the Silia*MetS* Metal Scavenger Kit.

SiliCycle also offers a confidential Metal Scavengers Screening Service. Contact us to take advantage of our expertise in metal removal. See page 93 to learn more about this service.

Silia*MetS* Metal Scavenger Selection Table

Scavenger	Ag	Ca	Cd	Co	Cr	Cs	Cu	Fe	Hg	Ir	La	Li	Mg	Ni	Os	Pb	Pd (II)	Pd (0)	Pt	Rh (1)	Rh (II)	Rh (III)	Ru (II)	Sc	Sn	W	Zn
Si-Thiol	■						□		■	□					■	□	■	■		□	□	□			□		
Si-Thiourea	□						□	□							□		■	■		□	□	□		□	□		
Si-Cysteine		□	■		□	□	□	■		■	□		□		■		□	□	□	□	□		■	■	■		□
Si-DMT			□	□			□	□		■				■	■		■	■	■	■	■	■	■	□			□
Si-Amine			■	□	■		□	□	□							□	□		■	■	■					□	□
Si-Diamine			□	□	■		□	□	□					□		□	■	■	■				□			□	□
Si-Triamine				□	■		□	□						□		■	■	■	■				□			□	□
Si-Imidazole			■	■	□		■	■						■	■		□	□		□	□					■	■
Si-TAAcOH				■	□	□		□						■	■		□	□		□	□			■	□		
Si-TAAcONa		■	■		□	■	■	■		■	■	■	■	■	■		□	□		□	□		□	■	■		□

■ Preferred scavengers □ Scavenges

SiliaMetS Selection Guide (*con't*)

SiliaMetS Metal Scavengers Selection Guide (*Only Catalyst in Solution*)

SiliaMetS	Catalyst, Solvent & Conditions (*% of catalyst scavenged*)																SiliaMetS
	$Pd(OAc)_2$ DMF 4 eq., 4 h, 22°C	$Pd_2(allyl)_2Cl_2$ DMF 4 eq., 4 h, 80°C	$Pd_2(dba)_3$ DMF 4 eq., 4 h, 22°C	$Pd(PPh_3)_4$ DMF 4 eq., 4 h, 80°C	$PdCl_2(dppf)$ DMF 4 eq., 4 h, 22°C	Grubbs 1st Gen. DMF 8 eq., 16 h, 80°C	Grubbs 2nd Gen. DMF 8 eq., 16 h, 80°C	Hoveyda-Grubbs 1st DMF 8 eq., 16 h, 80°C	Hoveyda-Grubbs 2nd DMF 8 eq., 16 h, 80°C	TPAP DCM 4 eq., 16 h, 22°C	$Ni(acac)_2$ DMF 4 eq., 4 h, 22°C	Wilkinson's Cat. DMF 4 eq., 4 h, 80°C	$[Rh(OAc)_2]_2$ DMF 4 eq., 4 h, 80°C	H_2PtCl_6 DMF 4 eq., 4 h, 80°C	$Pb(OAc)_2.3H_2O$ DMF 4 eq., 4 h, 22°C	$Zn(OAc)_2.2H_2O$ DMF 4 eq., 4 h, 22°C	
SiliaMetS Thiol	> 99	> 99	98	98		96	99	93		96 [4 eq.]		> 99 [16h]	97	80 [16 h]	97	> 99	Thiol
SiliaMetS Thiourea	> 99	> 99	98	91		98	96	98		> 99		99	97			97 [80°C]	Thiourea
SiliaMetS Cysteine	not screened	not screened	not screened	98	not screened	not screened	not screened	not screened	not screened	not screened	92	88	not screened	99		> 99	Cysteine
SiliaMetS DMT	98	> 99 [22°C]	> 99	> 99	Pd: 94, Fe: 92	> 99 [4 eq.]	99 [4 eq.]	98 [4 eq.]	99 [4 eq.]	> 99 [4 eq.]	97	> 99	> 99	> 99	99	94	DMT
SiliaBond Amine	98	> 99	97			97				> 99		> 99	> 99			> 99	Amine
SiliaMetS Diamine	> 99	> 99	> 99	90		99	94	98	90	97 [4 eq.]	99	> 99	> 99 [22°C]	> 99	81	> 99	Diamine
SiliaMetS Triamine	> 99	90	> 99	80		95		95	95	> 99	93	97	97 [22°C]	97	> 99 [80°C]	> 99	Triamine
SiliaMetS Imidazole	not screened	not screened	not screened	not screened		not screened	not screened	not screened		not screened	91 [80°C]	90	97 [22°C]	not screened		> 99	Imidazole
SiliaMetS TAAcOH	98	93	97 [80°C]							> 99 [4 eq.]	> 99	97	96 [16 h]				TAAcOH
SiliaMetS TAAcONa	97		80 [80°C]							> 99 [4 eq.]	> 99	88	> 99 [16 h]		90	> 99	TAAcONa

Note: other catalysts results are available on request (*metal screened but not shown: calcium, cobalt, cesium, copper, iron, iridium, lanthane, tin, & tunsgten. Contact us!*)

SiliaMetS Metal Scavengers Selection Guide (*Catalysts Scavenging in a Reaction*)

SiliaMetS	Catalyst, Solvent, Condition & Reaction									SiliaMetS
	$PdCl_2(PPh_3)_2$, CuI (in DME)	$Pd(OAc)_2$, P(o-tol)$_3$ (in i-PrOH, H_2O)	$RhCl(PPh_3)_3$ (in Toluene)	$FeCl_3.6H_2O$	CuCN (in DMF)	ridium Crabtree's Cat. (in DCM)	$LaCl_3.LiCl$ (in DMF)	$PhCH_2ZnCl$ (in THF)		
	8 eq., 4 h, 22°C Sonogashira Coupling	5 eq., 4 h, 40°C Suzuki Coupling	65 eq., 4 h, 22°C Wilkinson Hydrogenation	5 eq., 4 h, 22°C Michael Addition	10 eq., 4 h, 22°C Rosemund von-Braun Cyanation	4 eq., 4 h, 22°C Alkene Hydrogenation	1 eq., 4 h, 22°C 1,2-Addition on Ketone	4 eq., 4 h, 80°C Negishi Coupling		
SiliaMetS Thiol	Pd: 89, Cu: 29	98			94					Thiol
SiliaMetS Thiourea	Pd: 72, Cu: 80	92	81	82	> 99					Thiourea
SiliaMetS Cysteine		84	88	> 99	> 99	86	Li: 75, La: > 99	91		Cysteine
SiliaMetS DMT	Pd: 98, Cu: > 99	> 99	94	98	> 99			84		DMT
SiliaBond Amine		80	93	98	98			94		Amine
SiliaMetS Diamine		80		> 99	> 99			95		Diamine
SiliaMetS Triamine				98	> 99			91		Triamine
SiliaMetS Imidazole		88	92	98	95			94		Imidazole
SiliaMetS TAAcOH			81	98	80					TAAcOH
SiliaMetS TAAcONa			88	> 99	> 99	80	Li: 95, La: > 99	94		TAAcONa

Scavenging > 99 % | Scavenging 95 - 99 % | Scavenging 90 - 94 % | Scavenging 80 - 89 %

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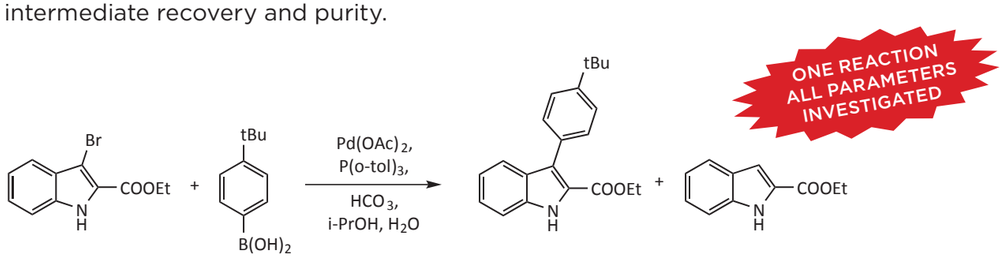
Silia*MetS* - A GlaxoSmithKline Case Study[1]

A metal scavenging study was performed following the synthesis of a key synthetic intermediate obtained by the Suzuki-Miyaura coupling presented below. Various parameters were investigated including the efficiency of Silia*MetS* in different formats, scavenging kinetics, intermediate recovery and purity.



Scavenging Efficiency, Recovery & Purity

Small-Scale Scavenging (*Synthesis Scale ~ 5 g*)

Table below shows the most efficient Silia*MetS* Metal Scavenger products for the treatment of the reaction mixture after work-up in both bulk and fixed mode bed (*pre-packed SPE cartridges*).

Silia*MetS* Scavenging Efficiency & Intermediate Recovery Results

Silia*MetS*	Batch Reactor Mode (*Bulk*)		Fixed Mode (*SPE*)	Intermediate Recovery
	5 eq., 4 h, 22°C	5 eq., 4 h, 40°C	6 mL / 1 g	
Silia*MetS* Thiol	95%	> 99%	98%	> 99%
Silia*MetS* Thiourea	83%	93%	99%	98%
Silia*MetS* Cysteine	84%	91%	97%	> 99%
Silia*MetS* DMT	97%	> 99%	> 99%	98%
Initial Pd Concentration:	179 ppm in MTBE		76 ppm in Toluene	-

Scavenging Conclusion

Addition of only 5 equivalents of Silia*MetS* products for 4 hours at the end of the reaction reduces the residual metal concentration to single digit ppm.

Recovery & Purity Conclusion

Palladium was completely removed, while the organic compound was not sequestrated by Silia*MetS* products. No impurities were released.

[1] *Org. Proc. Res. & Dev.*, **2008**, *12*, 896

Larger Scale Scavenging (*Synthesis Scale ~ 55 g*)

Silia*MetS* Metal Scavengers in pre-packed Silia*Sep* Flash Cartridges are a great alternative for metal removal at process development scale. These cartridges offer excellent scavenging efficiency as shown by results in t below. After the first run, almost all the palladium is captured. After three runs, less than 1 ppm remained in solution.

Silia*Sep* Scavenging Results

Run #	Scavenging
1	97%
2	99%
3	> 99%

Initial Pd Concentration: 700 ppm in AcOEt

Experimental Conditions:

Cartridge Size: 120 g of Silia*MetS* Thiol
Nb. Equivalent of Silia*MetS* Thiol: 25 eq.
Solution Volume: 1 liter
Flow Rate: 40 mL / min

Metal Scavenging in Flow Chemistry (*Preliminary Results*)

Flow chemistry is a relatively new technique that is gaining in popularity for large scale manufacturing because of the small investment needed to be able to produce large quantities in a short time. Silia*MetS* Metal Scavengers can also be used in flow chemistry to scavenge metals. A crude reaction mixture purified using a Syrris ASIA® Flow Chemistry System is presented below.

Silia*MetS* Thiol Scavenging Results in Flow Chemistry

Flow Rate	Solution Volume	Contact Time with Silia*MetS* Thiol	Time Needed to Treat the Solution	Scavenging Results
1.50 mL/min	100 mL	16 min	1h10	94.0%
1.00 mL/min	100 mL	24 min	1h40	94.3%
0.75 mL/min	50 mL	32 min	1h10	94.5%
0.50 mLmin	50 mL	48 min	1h40	95.0%

Initial Pd Concentration: 547 ppm in EtOAc

Experimental Conditions:
Scavenger Used: Silia*MetS* Thiol
Silia*MetS* Nb. Equivalent: 13.5 eq.
Reactors: 2 x 12 mL Reactors in Series
Total Solution Volume: 100 mL
Purification Scale: 12.5 g
Temperature: 22°C

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Variation of Phosphorous Ligand Nature & Scavenging

Even for the same metal, a variation in the scavenging efficiency can be observed depending on the nature of the products present in the solution to be treated. For example, the steric hindrance of a catalyst and the electronic effects of the phosphorous ligands, are factors influencing the removal of the metal. The same suzuki coupling shown on page 27 was performed using different phosphorous ligands; three monodentate and three bidentate ligands. For comparison purposes, scavenging screening was done by using the same two sets of conditions. No optimization was done to increase Silia*MetS* performance. By experience, using longer reaction times or higher temperatures will allow for better results.

Silia*MetS* Scavenging Results with Monodentate Ligands

Silia*MetS*	Triphenylphosphine [PPh_3]		Tri(o-tolyl)phosphine [$P(otol)_3$]		Tri-n-butylphosphine [$PnBu_3$]	
	4 eq., 4 h, 22°C	4 eq., 4 h, 60°C	4 eq., 4 h, 22°C	4 eq., 4 h, 60°C	4 eq., 4 h, 22°C	4 eq., 4 h, 60°C
Silia*MetS* Thiol	70%	97%	87%	96%	26%	85%
Silia*MetS* Thiourea	55%	86%	54%	82%	18%	41%
Silia*MetS* Cysteine	69%	76%	77%	90%	17%	44%
Silia*MetS* DMT	95%	97%	95%	> 99%	36%	87%
Initial Pd Concentration:	27 ppm in EtOAc		84 ppm in EtOAc		90 ppm in EtOAc	

Silia*MetS* Scavenging Results with Bidentate Ligands

Silia*MetS*	1,1'-bis(diphenylphosphino)ferrocene [dppf]		1,3-bis(diphenylphosphino)propane [dppp]		(+/-) BINAP	
	4 eq., 4 h, 22°C	4 eq., 4 h, 60°C	4 eq., 4 h, 22°C	4 eq., 4 h, 60°C	4 eq., 4 h, 22°C	4 eq., 4 h, 60°C
Silia*MetS* Thiol	50%	69%	75%	90%	31%	56%
Silia*MetS* Thiourea	3%	23%	40%	60%	33%	21%
Silia*MetS* Cysteine	29%	36%	47%	55%	19%	29%
Silia*MetS* DMT	14%	22%	95%	98%	41%	64%
Initial Pd Concentration:	63 ppm in EtOAc		93 ppm in EtOAc		16 ppm in EtOAc	

Scavenging Conclusion

In all cases, Silia*MetS* DMT and Thiol remained the better scavengers throughout the study, even though there is a variation in the nature of the ligand.

Ruthenium Scavenging with Silia*MetS*

Ruthenium-based catalysts are commonly used in organic synthesis, mainly in olefin metathesis reactions [ROM(P) and RCM]. Grubbs and Hoveyda-Grubbs catalysts are the most popular ruthenium-based complexes in this field of applications. Complete ruthenium removal can be tedious using conventional methods.

Silia*MetS* allow the maximal tolerated concentration of the residual ruthenium to be reached. A ruthenium scavenging study was conducted and various parameters were investigated in order to learn more about their influence on the scavengers' robustness as well as to establish the best experimental conditions.

Ruthenium Scavenging Results using Silia*MetS*

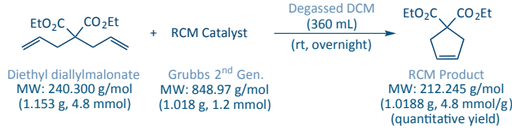
Silia*MetS*	Grubbs 1st Gen.		Grubbs 2nd Gen.		Hoveyda-Grubbs 1st Gen.		Hoveyda-Grubbs 2nd Gen.	
	Toluene[1]	DMF[2]	Toluene[1]	DMF[2]	Toluene[1]	DMF[2]	Toluene[1]	DMF[2]
Silia*MetS* Thiol	90%	96%	-	99%	97%	93%	-	-
Silia*MetS* Thiourea	-	98%	-	96%	97%	98%	-	-
Silia*MetS* DMT	95%	99%[2]	> 99%	99%[2]	> 99%[2]	98%[2]	98%[2]	99%[2]
Silia*Bond* Amine	95%	97%	92%	-	-	-	-	-
Silia*MetS* Diamine	99%	99%	91%	94%	> 99%	98%	-	90%
Silia*MetS* Triamine	-	95%	-	-	93%	95%	-	95%
Silia*MetS* TAAcOH	93%	-	-	-	-	-	-	-
Silia*MetS* TAAcONa	96%	-	96%	-	98%	-	-	-

Exp. Conditions: [1]8 eq. of Silia*MetS*, 16 h, 80°C; [2] Only 4 eq. of Silia*MetS*. Initial concentration: 500 ppm for all ruthenium-based catalysts.

Note: **Silia*MetS* Cysteine** and **Imidazole** were not screened in this study (*and are not currently available for this application*). Only **Silia*MetS*** results higher than 90% are presented in this table.

Silia*MetS* vs Other Purification Methods

The use of Silia*MetS* to remove ruthenium catalyst after a ring-closing metathesis (*RCM*) reaction is the most effective purification method. As demonstrated below, the main advantage is that no product is lost during the purification step.



Scavenging Results for Various Purification Methods*

Scavenging	Scavenger	Filtration over packed bed of ...[2]			Flash Purification	
	Silia*MetS* DMT[1]	Act. Carbon	Celite	Silica	Manual	Silia*Sep* Cart.
Ruthenium captation	93%	73%	24%	58%	70%	73%

[1] Using 4 eq., 16h, 22°C. [2] Solution is passed directly on a packed bed of various adsorbents, which was then washed with the same quantity of solvent.

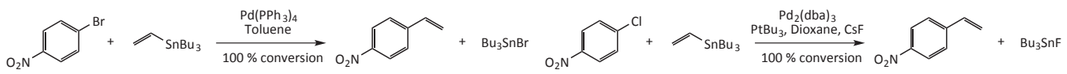
*Quantitative yield obtained for each purification method (*adjusted in function of the residual concentration of catalyst*). No impurities were generated in all cases using the different methods (*determined by NMR*).

Tin Scavenging with Silia*MetS*

Organotin compounds are versatile reagents commonly used in organic synthesis. The two main applications are in Stille couplings or radical reactions. The removal of tin residues can often be an issue due to the high toxicity of this metal.

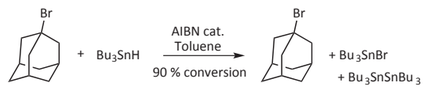
Traditional removal methods for this impurity are treatment with an aqueous solution of KF, NH_4OH or NaOH, or with bases such as DBU. However, the efficiency of these methods can vary and may be inapplicable for particular compounds.

Silia*MetS* Cysteine & TAAcONa can be used to efficiently remove tin residues from organic mixtures as demonstrated by the exemples below.



Stille Coupling #1

Stille Coupling #2



Radical Reduction

Tin Scavenging using Silia*MetS* Cysteine & TAAcONa

Reactions	Inital Concentration	Silia*MetS* Cysteine		Silia*MetS* TAAcONa		
		4 eq., 4 h, 22°C [2 treatments]	8 eq., 4 h, 22°C	4 eq., 4 h, 22°C [2 treatments]	8 eq., 4 h, 22°C	4 eq., 16 h, 22°C
Stille coupling #1[1]	3,385 ppm	99%	64%	96%	62%	-
Stille coupling #2[1]	981 ppm	90%	66%	66%	50%	-
Radical Reduction	4,090 ppm	92%	88%	90%	90%	90%

[1] Pd residues were completely removed after only one treatment with **Silia*MetS* Cysteine**.

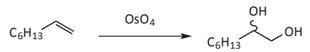
Osmium Scavenging with Silia*MetS*

Osmium products are very useful in organic synthesis. One of the most commonly used is osmium tetroxide (OsO_4), which is a very reliable and powerful reagent for the cis-dihydroxylation of alkenes. However, osmium compounds, in particular OsO_4, are highly poisonous, even at low exposure levels, and must be handled with appropriate precautions.

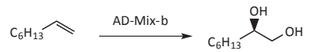
Therefore, it is important to efficiently remove residual osmium from products of interest.

A scavenging study on three organic reactions involving osmium reactants were performed. The metal scavenging efficiency of Silia*MetS* is highlighted in the table on the following page.

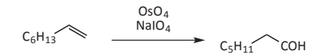
Osmium Scavenging with Silia*MetS* (*con't*)



Dihydroxylation Reaction
[OsO_4]



Sharpless Dihydroxylation
[Potassium osmate ($K_2OsO_2(OH)_4$]



Lemieux-Johnson Oxidation
[OsO_4]

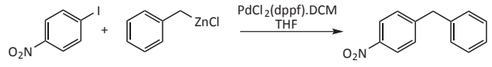
Osmium Scavenging using Silia*MetS*

Silia*MetS*	Dihydroxylation	Sharpless Dihydroxylation		Lemieux-Johnson Oxidation	
	4 eq., 4 h, 22°C	8 eq., 4 h, 22°C	8 eq., 16 h, 22°C	8 eq., 4 h, 22°C	8 eq., 16 h, 22°C
Silia*MetS* Thiol	87%	> 98%	> 98%	87%	92%
Silia*MetS* Cysteine	89%	> 98%	> 98%	87%	91%
Silia*MetS* DMT	92%	97%	> 98%	87%	91%
Silia*MetS* Imidazole	87%	> 98%	> 98%	89%	91 %
Initial Os Concentration:	132 ppm in EtOAc	25 ppm in EtOAc		21 ppm in EtOAc	

Note: > 98 % of scavenging means < 0.5 ppm of osmium.

Multiple Metal Scavenging with Silia*MetS*

Silia*MetS* can be used to remove multiple metals in the same reaction with excellent efficiency. The Negishi coupling presented below was performed to show that Silia*MetS* can be used to simultaneously remove residual zinc, palladium, and iron present after the reaction.



Negishi Coupling

Multiple Removal Scavenging Results

Silia*MetS*	Palladium	Iron	Zinc
Silia*MetS* Cysteine	95%	> 99%	98%
Silia*MetS* DMT	83%	93%	99%
Silia*MetS* Imidazole	84%	91%	97%
Silia*MetS* TAAcONa	97%	> 99%	> 99%
Initial Concentration:	188 ppm in THF	110 ppm in THF	6 ppm in THF

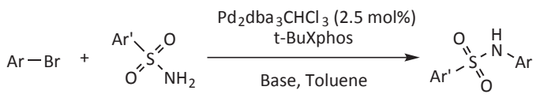
Conditions: 4 eq. of Silia*MetS* (*relative to palladium*), 4 h, 22°C.

Silia*MetS* Success Stories Published by Customers

Silia*MetS* Metal Scavengers are being used by many pharmaceutical companies, several of which are now using them in pilot plants. In the literature, you can find a number of success stories published by customers highlighting the ease of use and reliable performance of **Silia*MetS***. Some examples are presented in the following pages.

An Amgen Case Study[1]

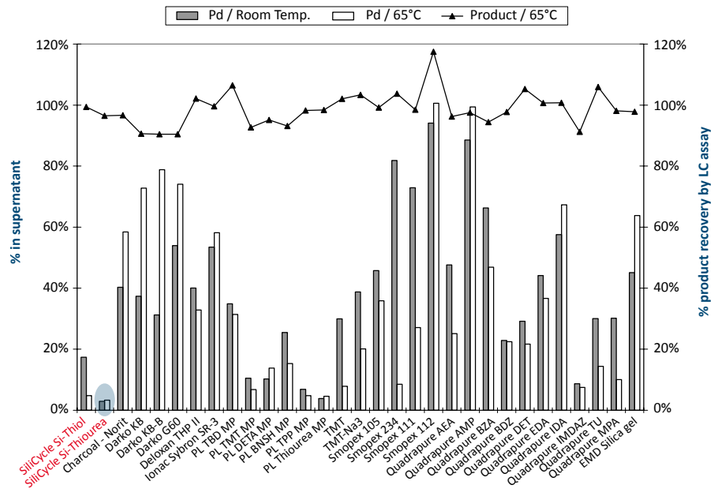
In 2009, Amgen published a chapter in "Catalysis of Organic Reactions" related to the use of scavengers for the removal of palladium in small to multi-kilogram production scale. In their study, they evaluated various parameters such as the scavenging efficiency, the influence of the scavenger loading and the loss of product to adsorption (*recovery*). The study was based on a palladium-catalyzed sulfonamide coupling and scavenger screening was performed at both room temperature and 65°C using 31 different scavengers.



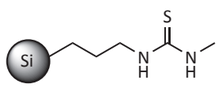
Amgen Scavenger Screening Results

Condition: 20 mg of each scavenger (*20% w/w*) in 2 mL HPLC vial that contains 1 mL of crude reaction mixture containing 100 mg of product. Each vial was sealed and agitated overnight. Initial palladium concentration was 423 ppm.

The **BEST** scavenger identified during their study was the Silia*MetS* Thiourea providing the lowest Pd content (*residual palladium concentration: 3% or < 14 ppm*) without product sequestration. They mentioned that Silia*MetS* Thiourea was used extensively in early process development work.



SILIA*METS* THIOUREA WAS THE BEST!



Silia*MetS* Thiourea

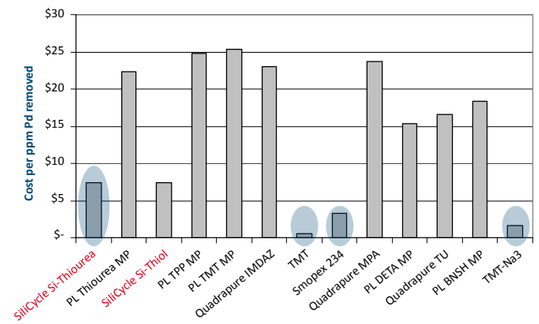
[1]Catalysis of Organic Reactions, Chapter 5. Application of Scavengers for the Removal of Palladium in Small Lot Manufacturing

Allgeier & al., Amgen Inc., Thousand Oaks (*California*)

Cost Comparison for Most Efficient Scavengers (≥ *80%*)

At pilot-plant scale, the optimal compromise between the cost per ppm removed and the scavenging efficiency is crucial. The histogram at right shows a cost comparison on best scavengers identified.

Results highlighted by the graph reduced the number of options to only 4 candidates for further evaluation: in pole position the Silia*MetS* Thiourea , and then the TMT, TMT-Na3, and the Smopex 234.



Top 4 Scavengers Overview

A screening validation was conducted on 1 g scale purification (*10 mL of solution*) with 20% w/w of each top 4 scavengers at 65°C overnight. After filtration, residual metal concentration was analyzed by ICP-MS and product recovery was determined by HPLC (*see below*). Silia*MetS* Thiourea was chosen for the large scale purification. See Amgen's paper for further details.

Screening Validation Results on Top 4 Scavengers

Scavengers	Residual Metal Concentration (*ppm*)			Product Recovery	Commentary
	Screening Exp. in Solution	Validation Exp. in Solution	Validation Exp. in Solid Product[1]		
SiliCycle Thiourea	14	11	158	102%	Best performance but also most expensive.
TMT	33	15	264	104%	Fine in suspension, filterability concerns on scale.
Smopex 234	36	38	496	84%	Favorable cost but product recovery inadequate
TMT-Na3	85	81	1 555	78%	Very basic compounds (*not effective with base-sensitive groups*). Low recovery.
Purification Scale:	100 mg	1 g	1 g	1 g	
Initial Concentration:	423 ppm	381 ppm	3,577 ppm		

Note: [1]Solid product is obtained by dividing the metal concentration in ppm by the amount of product in the test (*1g*).

Amgen's Conclusion

"Scavengers offer a practical and expedient option for removal of palladium from process streams to ensure quality of organic products... The screening protocol involves treatment of a candidate process stream with 20% w/w scavenger on product at both room temperature and 65°C followed by analysis of Pd and product adsorption. High-temperature treatment increased the efficiency of Pd removal... Evaluation of process costs is key to identifying Pd removal solutions. While scavengers add cost to a process, their use is often justified by the speed to production in early phase development."

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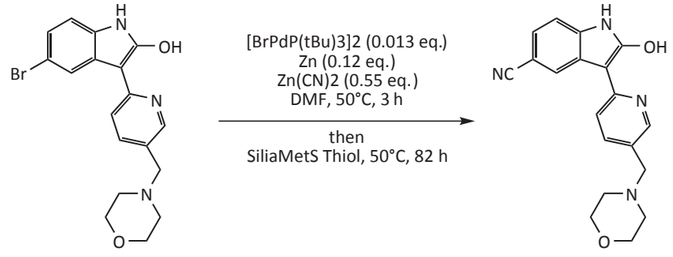
An AstraZeneca Case Study

Publication: Ryberg, P., *Organic Process Research & Development, 12,* **2008**, *540*
Process Chemistry, AstraZeneca PR&D, Sweden.

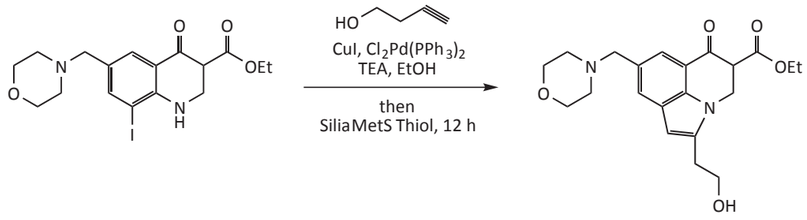
In 2008, AstraZeneca published a paper in which they removed palladium impurities in a large-scale process. The workup method found to work the best was a treatment with Silia*MetS* Thiol (*25% w/w or ~1.4 kg*) at 50°C to purify more than 6.7 kg of material. Final residual palladium concentration was as low as 1-2 ppm.



A Pfizer Global R&D Case Study

Publication: Dorow, R.L. & all, *Organic Process Research & Development, 10,* **2006**, *493*
Pfizer Global Research and Development, Kalamazoo, Michigan (*USA*)

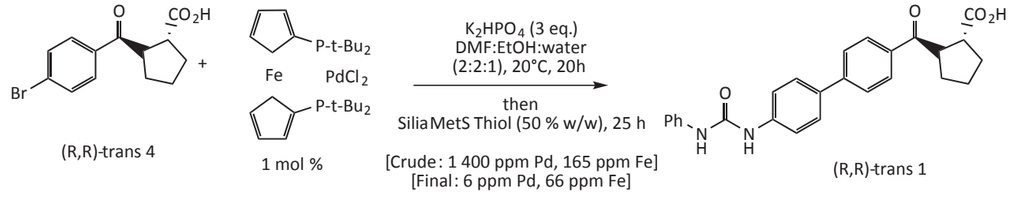
In 2006, Pfizer published a paper in which they removed palladium & copper impurities in a 20 kg pilot plant scale. They made two subsequent treatments using Silia*MetS* Thiol (*20% + 7% w/w*) at room temperature for 12 hours. After scavenging with Silia*MetS* Thiol, the desired product was obtained with a yield of 76% containing only 17 ppm Pd and 1 ppm Cu. An alternative method was also tried using 80% w/w of Deloxan THP (*Degussa AG*) overnight followed by basification with Na_2CO_3. Residual metal concentration with this method was higher compared to that of Silia*MetS* and the yield was lower (*about 60%-70%*). Silia*MetS* allows lower residual metal concentration & higher yield with fewer manipulations!



An Abbott Laboratories Case Study

Publication: Ravn, M.M. & all, P., *Organic Process Research & Development, 14,* **2010**, *417*
Global Pharmaceutical R&D, Process Research & Development and Discovery, Abbott Laboratories, Chicago, Illinois (*USA*)

In 2010, Abbott Laboratories published a paper in which they removed palladium and iron impurities using Silia*MetS* Thiol (*50% w/w relative to* ***1***). Thus, palladium and iron levels were respectively 6 ppm and 66 ppm. Refer to Abbott's publication for more details.

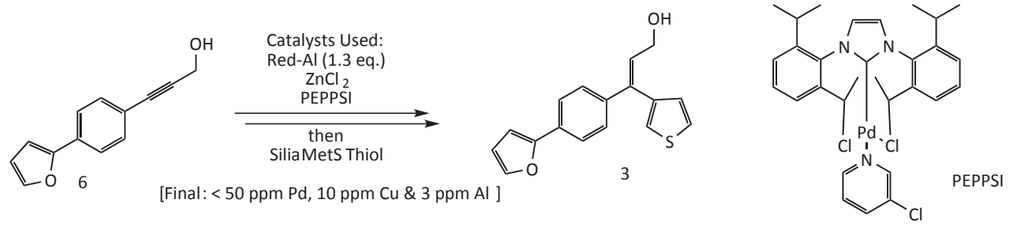


A Johnson & Johnson Case Study

Publication: Houpis I.N. & all, *Organic Process Research & Development, 13,* **2009**, *598*
Johnson & Johnson PRD, API Development, Belgium, and Solvias A.G., Synthesis and Catalysis, Switzerland

In 2009, Johnson & Johnson (*J&J*) in collaboration with Solvias published a paper in which they developed a mild Sonogashira reaction using various metal catalysts. Treatment with Silia*MetS* Thiol simultaneously removed Pd, Cu & Al. Residual concentrations were below 50, 10, and 3 ppm, respectively, in the isolated product **3**. Refer to J&J's publication for more details.

Note: copper comes from a previous synthesis step.



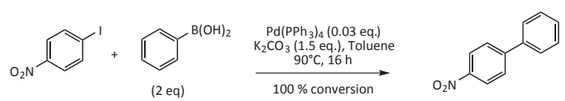
Silia*MetS* Leaching & Stability Studies

Silia*MetS* Metal Scavengers are being used by many pharmaceutical and biotechnological companies. Each **Silia*MetS*** manufactured by SiliCycle is submitted to an extensive washing procedure to ensure the product exhibits extremely low levels of extractables and leachables.

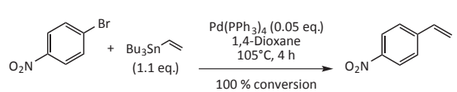
SiliCycle has implemented a quality control procedure to prevent leaching that includes loading and reactivity determination, as well as leachables and extractables analysis (*silica gel purity ≥ 99.995%*). The solution must be free of contaminants for the product to successfully pass the rigorous quality control tests.

To address the end users concerns for potential leaching of impurities into reaction mixtures using Silia*MetS*, we have performed three typical metal containing reactions. We then investigated the detection, identification, and quantification of possible impurities resulting from the scavengers used.

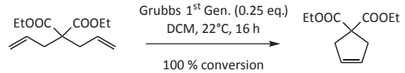
The following three transition metal catalyzed reactions were performed:



Suzuki Coupling



Stille Coupling



Ring-Closing Metathesis

Experimental Procedure

Crude reaction mixtures (*8 mL*) were placed in a standard polypropylene tube equipped with a 20 µm frit, filled with 1 g of the appropriate Silia*MetS* Metal Scavenger, and mixed for 4 h at either room temperature or 80°C. Solutions were then filtered through a 0.02 µm filter prior to analysis.

Leaching Analysis

For each Silia*MetS*, silane leaching was analyzed by ICP-OES, which has proven to be very sensitive for silicon quantification (*detection limit in solution is 0.125 ppm*). Traces of non-silicon containing impurities were also analyzed by GC-MS and ^{1}H NMR Analysis. Only results for Silia*MetS* Thiol and DMT are shown. However, no evidence of impurities was found for all Silia*MetS*. Contact us for the complete study results.

Gel Purity Calculation Example

Impurity % : $\frac{\text{2 mg of silicon}}{\text{1,000,000 mg of Silia}\textit{MetS}} \times 100 \Rightarrow 0.0002\%$ impurity

Gel purity = 100 - (*Impurity %*) => 99.9998% purity

Silane Leaching Analysis by ICP-OES

Results shown in the table below for Silia*MetS* Thiol & DMT confirm that minimal leaching occurs with SiliCycle Silia*MetS*.

*Note: concentration given are in ppm and represent mg of silicon leached per kg of Silia*MetS*.*

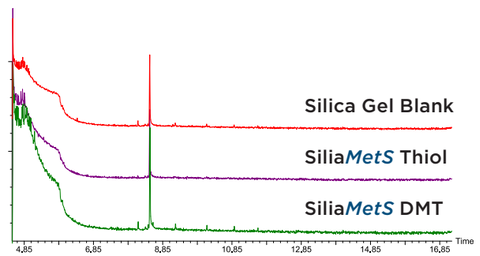
Stability of Silia*MetS* in Suzuki, Stille and Ring-Closing Metathesis reactions

Reaction (*solvent*)	Temperature	Silia*MetS* Thiol		Silia*MetS* DMT	
		[Silicon]	Gel Purity	[Silicon]	Gel Purity
Suzuki (*Toluene*)	22°C	2 ppm	99.9998%	1 ppm	99.9999%
	80°C	2 ppm	99.9998%	2 ppm	99.9998%
Stille (*1,4-Dioxane*)	22°C	2 ppm	99.9998%	1 ppm	99.9999%
	80°C	1 ppm	99.9999%	3 ppm	99.9997%
Ring-Closing Met. (*DCM*)	22°C	2 ppm	99.9998%	2 ppm	99.9998%

Note: Very low levels of silicon were detected in most experiments, giving product purities higher than 99.995%.

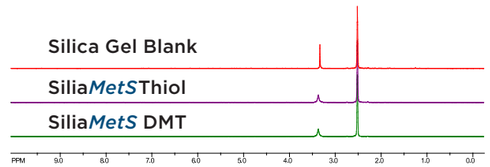
Non-Silicon Leaching Analysis

Gas chromatography-mass spectrometry (*GC-MS*)



Compared to the silica blank spectrum (*bare silica in solvent*), neither experiment showed evidence of any impurities for either Silia*MetS* Thiol or DMT.

1H NMR Analysis (*d6-dmso*)



Note: each experiment was run on a 1 g aliquote of Silia*MetS* and was shaken for one hour at room temperature. In GC-MS spectrum, peak at 8.5 minutes is the internal standard (*1-fluoronaphthalene, 100 ppm*). In NMR spectrum, peaks at 2.4 and 3.4 ppm are, respectively, d6-dmso and water contained in deuterated solvent.

Stability Study (*Shelf Life*)

SiliCycle certifies that Silia*MetS* Metal Scavengers stored under recommended conditions in an undamaged container are guaranteed to perform for over two years from the manufacturing date without loss of performance (*results at right*).

Silia*MetS* Thiol after Two Years

Lot #	QC Date	Scavenging
11577	January 2008	> 99.9%
	October 2010	99.6%
12218	February 2008	99.9%
	October 2010	99.1%

Scavenging: 1 000 ppm of $Pd(OAc)_2$ in DMF.
Conditions: 2 eq. of Silia*MetS* Thiol, 1 h, 22°C.

Silia*MetS* Metal Scavengers Ordering Information

Silia*MetS* Bulk Ordering Information

Metal Scavenger	Part Number	Metal Scavenger	Part Number
Silia*MetS* Thiol	R51030B	**Silia*MetS*** Diamine	R49030B
Silia*MetS* Thiourea	R69530B	**Silia*MetS*** Triamine	R48030B
Silia*MetS* Cysteine	R80530B	**Silia*MetS*** Imidazole	R79230B
Silia*MetS* DMT	R79030B	**Silia*MetS*** TAAcOH	R69030B
Silia*Bond* Amine	R52030B	**Silia*MetS*** TAAcONa	R69230B

Formats: 5g, 10g, 25g, 50g, 100g, 250g, 500g, 1kg, 5kg, 10kg, 25kg, ... up to multi-ton scale. Contact us for details.

Silia*Sep* Metal Scavenger Cartridges Ordering Information (*see SiliaSep's section at page 157*)

Silia*Sep* Type Quantity per box	Silia*Sep* 4 g 2/box	Silia*Sep* 12 g 1/box	Silia*Sep* 25 g 1/box	Silia*Sep* 40 g 1/box	Silia*Sep* 80 g 1/box
Silia*Sep* Thiol	FLH-R51030B-ISO04	FLH-R51030B-ISO12	FLH-R51030B-ISO25	FLH-R51030B-ISO40	FLH-R51030B-ISO80
Silia*Sep* Thiourea	FLH-R69530B-ISO04	FLH-R69530B-ISO12	FLH-R69530B-ISO25	FLH-R69530B-ISO40	FLH-R69530B-ISO80
Silia*Sep* Cysteine	FLH-R80530B-ISO04	FLH-R80530B-ISO12	FLH-R80530B-ISO25	FLH-R80530B-ISO40	FLH-R80530B-ISO80
Silia*Sep* DMT	FLH-R79030B-ISO04	FLH-R79030B-ISO12	FLH-R79030B-ISO25	FLH-R79030B-ISO40	FLH-R79030B-ISO80
Silia*Sep* Amine	FLH-R52030B-ISO04	FLH-R52030B-ISO12	FLH-R52030B-ISO25	FLH-R52030B-ISO40	FLH-R52030B-ISO80
Silia*Sep* Diamine	FLH-R49030B-ISO04	FLH-R49030B-ISO12	FLH-R49030B-ISO25	FLH-R49030B-ISO40	FLH-R49030B-ISO80
Silia*Sep* Triamine	FLH-R48030B-ISO04	FLH-R48030B-ISO12	FLH-R48030B-ISO25	FLH-R48030B-ISO40	FLH-R48030B-ISO80
Silia*Sep* Imidazole	FLH-R79230B-ISO04	FLH-R79230B-ISO12	FLH-R79230B-ISO25	FLH-R79230B-ISO40	FLH-R79230B-ISO80
Silia*Sep* TAAcOH	FLH-R69030B-ISO04	FLH-R69030B-ISO12	FLH-R69030B-ISO25	FLH-R69030B-ISO25	FLH-R69030B-ISO80
Silia*Sep* TAAcONa	FLH-R69230B-ISO04	FLH-R69230B-ISO12	FLH-R69230B-ISO25	FLH-R69230B-ISO25	FLH-R69230B-ISO80

Silia*Sep* Metal Scavenger Cartridges Ordering Information

Silia*Sep* Type Quantity per box	Silia*Sep* 120 g 2/box	Silia*Sep* 220 g 1/box	Silia*Sep* 330 g 1/box	Silia*Sep* XL 800 g 1/box	Silia*Sep* XL 1600 g 1/box
Silia*Sep* Thiol	FLH-R51030B-IS120	FLH-R51030B-IS220	FLH-R51030B-IS330	FLH-R51030B-IS750	FLH-R51030B-I1500
Silia*Sep* Thiourea	FLH-R69530B-IS120	FLH-R69530B-IS220	FLH-R69530B-IS330	FLH-R69530B-IS750	FLH-R69530B-I1500
Silia*Sep* Cysteine	FLH-R80530B-IS120	FLH-R80530B-IS220	FLH-R80530B-IS330	FLH-R80530B-IS750	FLH-R80530B-I1500
Silia*Sep* DMT	FLH-R79030B-IS120	FLH-R79030B-IS220	FLH-R79030B-IS330	FLH-R79030B-IS750	FLH-R79030B-I1500
Silia*Sep* Amine	FLH-R52030B-IS120	FLH-R52030B-IS220	FLH-R52030B-IS330	FLH-R52030B-IS750	FLH-R52030B-I1500
Silia*Sep* Diamine	FLH-R49030B-IS120	FLH-R49030B-IS220	FLH-R49030B-IS330	FLH-R49030B-IS750	FLH-R49030B-I1500
Silia*Sep* Triamine	FLH-R48030B-IS120	FLH-R48030B-IS220	FLH-R48030B-IS330	FLH-R48030B-IS750	FLH-R48030B-I1500
Silia*Sep* Imidazole	FLH-R79230B-IS120	FLH-R79230B-IS220	FLH-R79230B-IS330	FLH-R79230B-IS750	FLH-R79230B-I1500
Silia*Sep* TAAcOH	FLH-R69030B-IS120	FLH-R69030B-IS220	FLH-R69030B-IS330	FLH-R69030B-IS750	FLH-R69030B-I1500
Silia*Sep* TAAcONa	FLH-R69230B-IS120	FLH-R69230B-IS220	FLH-R69230B-IS330	FLH-R69230B-IS750	FLH-R69230B-I1500

Silia*MetS* Metal Scavengers Ordering Information (*con't*)

Silia*Sep* OT Metal Scavenger Cartridges (*rated 60 psi*)

Silica Weight Quantity per box	2 g 20/box	5 g 20/box	10 g 16/box	15 g 16/box	20 g 16/box
Silia*Sep* OT Thiol	SPE-R51030B-12U	SPE-R51030B-20X	FLH-R51030B-70Y	FLH-R51030B-70i	FLH-R51030B-70Z
Silia*Sep* OT Thiourea	SPE-R69530B-12U	SPE-R69530B-20X	FLH-R69530B-70Y	FLH-R69530B-70i	FLH-R69530B-70Z
Silia*Sep* OT Cysteine	SPE-R80530B-12U	SPE-R80530B-20X	FLH-R80530B-70Y	FLH-R80530B-70i	FLH-R80530B-70Z
Silia*Sep* OT DMT	SPE-R79030B-12U	SPE-R79030B-20X	FLH-R79030B-70Y	FLH-R79030B-70i	FLH-R79030B-70Z
Silia*Sep* OT Amine	SPE-R52030B-12U	SPE-R52030B-20X	FLH-R52030B-70Y	FLH-R52030B-70i	FLH-R52030B-70Z
Silia*Sep* OT Diamine	SPE-R49030B-12U	SPE-R49030B-20X	FLH-R49030B-70Y	FLH-R49030B-70i	FLH-R49030B-70Z
Silia*Sep* OT Triamine	SPE-R48030B-12U	SPE-R48030B-20X	FLH-R48030B-70Y	FLH-R48030B-70i	FLH-R48030B-70Z
Silia*Sep* OT Imidazole	SPE-R79230B-12U	SPE-R79230B-20X	FLH-R79230B-70Y	FLH-R79230B-70i	FLH-R79230B-70Z
Silia*Sep* OT TAAcOH	SPE-R69030B-12U	SPE-R69030B-20X	FLH-R69030B-70Y	FLH-R69030B-70i	FLH-R69030B-70Z
Silia*Sep* OT TAAcONa	SPE-R69230B-12U	SPE-R69230B-20X	FLH-R69230B-70Y	FLH-R69230B-70i	FLH-R69230B-70Z

Silia*Sep* OT Metal Scavenger Cartridges (*rated 60 psi*)

Silica Weight Quantity per box	25 g 10/box	50 g 10/box	70 g 10/box	100 g 12/box
Silia*Sep* OT Thiol	FLH-R51030B-95K	FLH-R51030B-95M	FLH-R51030B-95N	FLH-R51030B-276F
Silia*Sep* OT Thiourea	FLH-R69530B-95K	FLH-R69530B-95M	FLH-R69530B-95N	FLH-R69530B-276F
Silia*Sep* OT Cysteine	FLH-R80530B-95K	FLH-R80530B-95M	FLH-R80530B-95N	FLH-R80530B-276F
Silia*Sep* OT DMT	FLH-R79030B-95K	FLH-R79030B-95M	FLH-R79030B-95N	FLH-R79030B-276F
Silia*Sep* OT Amine	FLH-R52030B-95K	FLH-R52030B-95M	FLH-R52030B-95N	FLH-R52030B-276F
Silia*Sep* OT Diamine	FLH-R49030B-95K	FLH-R49030B-95M	FLH-R49030B-95N	FLH-R49030B-276F
Silia*Sep* OT Triamine	FLH-R48030B-95K	FLH-R48030B-95M	FLH-R48030B-95N	FLH-R48030B-276F
Silia*Sep* OT Imidazole	FLH-R79230B-95K	FLH-R79230B-95M	FLH-R79230B-95N	FLH-R79230B-276F
Silia*Sep* OT TAAcOH	FLH-R69030B-95K	FLH-R69030B-95M	FLH-R69030B-95N	FLH-R69030B-276F
Silia*Sep* OT TAAcONa	FLH-R69230B-95K	FLH-R69230B-95M	FLH-R69230B-95N	FLH-R69230B-276F

Silia*Prep* Metal Scavenger Cartridges (*see SiliaPrep's section at page 173*

Formats Quantity per box	200 mg / 3 mL 50 / box	500 mg / 3 mL 50 / box	500 mg / 6 mL 50 / box	1 g / 6 mL 50 / box	2 g / 6 mL 50 / box
Silia*Prep* OT Thiol	SPE-R51030B-03G	SPE-R51030B-03P	SPE-R51030B-06P	SPE-R51030B-06S	SPE-R51030B-06U
Silia*Prep* OT Thiourea	SPE-R69530B-03G	SPE-R69530B-03P	SPE-R69530B-06P	SPE-R69530B-06S	SPE-R69530B-06U
Silia*Prep* OT Cysteine	SPE-R80530B-03G	SPE-R80530B-03P	SPE-R80530B-06P	SPE-R80530B-06S	SPE-R80530B-06U
Silia*Prep* OT DMT	SPE-R79030B-03G	SPE-R79030B-03P	SPE-R79030B-06P	SPE-R79030B-06S	SPE-R79030B-06U
Silia*Prep* OT Amine	SPE-R52030B-03G	SPE-R52030B-03P	SPE-R52030B-06P	SPE-R52030B-06S	SPE-R52030B-06U
Silia*Prep* OT Diamine	SPE-R49030B-03G	SPE-R49030B-03P	SPE-R49030B-06P	SPE-R49030B-06S	SPE-R49030B-06U
Silia*Prep* OT Triamine	SPE-R48030B-03G	SPE-R48030B-03P	SPE-R48030B-06P	SPE-R48030B-06S	SPE-R48030B-06U
Silia*Prep* OT Imidazole	SPE-R79230B-03G	SPE-R79230B-03P	SPE-R79230B-06P	SPE-R79230B-06S	SPE-R79230B-06U
Silia*Prep* OT TAAcOH	SPE-R69030B-03G	SPE-R69030B-03P	SPE-R69030B-06P	SPE-R69030B-06S	SPE-R69030B-06U
Silia*Prep* OT TAAcONa	SPE-R69230B-03G	SPE-R69230B-03P	SPE-R69230B-06P	SPE-R69230B-06S	SPE-R69230B-06U

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SiliaBond®
Organic Scavengers

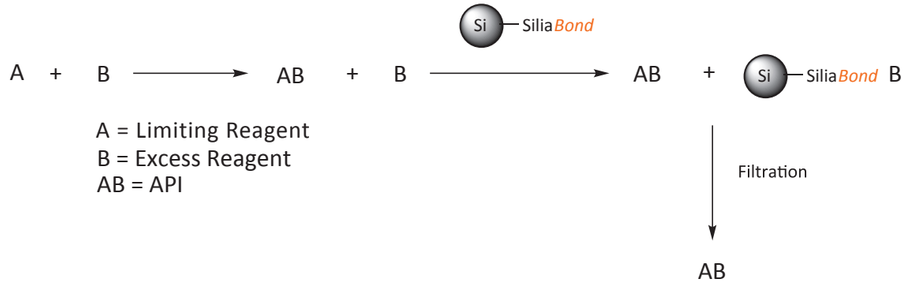
SILICYCLE Inc.
UltraPure SILICA GELS

SiliaBond Organic Scavengers

SiliaBond Organic Scavengers can be Used for the Purification of API's in 2 Different Ways:

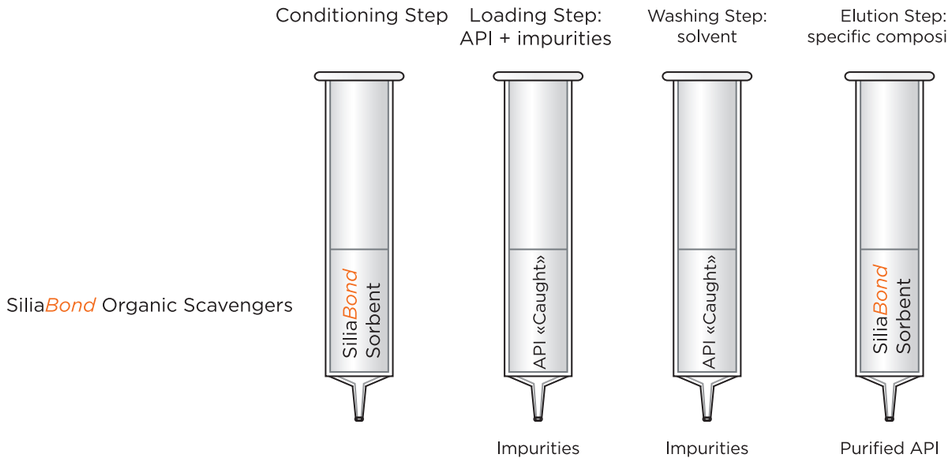
Scavenge Undesired Compounds to Isolate the API

This technique is used to trap the excess of reagent and/or the impurities on the silica matrix. The API is recovered by simple filtration as demonstrated on the following scheme.



Catch and Release of the API

This method is used in an SPE cartridge format where the API is caught on the silica matrix, then filtered to eliminate all other undesired components and finally released back in solution. The catch & release method is shown below.



Scavenging Undesired Compounds: Electrophile Scavengers

Electrophile Scavenger				
Function to be scavenged	Recommended Scavenger	Loading (mmol/g)	Typical conditions	Solvent compatibility
Acid chlorides or sulfonyl chlorides	SiliaBond Amine	1.6	- Add 2 - 4 eq. of SiliaBond SiliaMetS to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to attain acid chloride-free solution	All solvents
	SiliaMetS Diamine	1.4		All solvents
	SiliaMetS Triamine	1.2		All solvents
	SiliaBond DMAP	0.8		Organic solvents
	SiliaBond Piperazine	0.8		All solvents

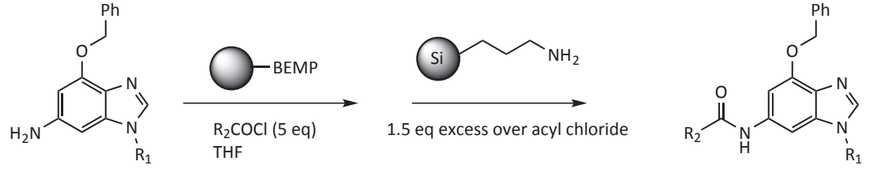
Scavenging Acid Chlorides with SiliaBond Amine

Sample Procedure

Add 1.5 eq of SiliaBond Amine to the reaction mixture, and stir for 1 h at room temperature.

Filter off the scavenger and rinse with solvent to yield acyl chloride free solution.

Related Publication: *J. Catal.*, *195,* **2000**, 412.



Electrophile Scavenger				
Function to be scavenged	Recommended Scavenger	Loading (mmol/g)	Typical conditions	Solvent compatibility
Aldehydes or carbonyls	SiliaBond Amine	1.6	- Add 2 - 4 eq. of SiliaBond to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to yield aldehyde free solution (*ketones and hindered aldehydes add 0.05 eq. of acetic acid*)	All solvents
	SiliaBond Tosyl Hydrazine	1.5		Aprotic and non carbonyl solvents

Scavenging Undesired Compounds: Electrophile Scavengers (*con't*)

Electrophile Scavenger

Function to be scavenged	Recommended Silia*Bond* scavenger	Loading (mmol/g)	Typical conditions	Solvent compatibility
Isocyanates	Silia*Bond* Amine	1.6	- Add 2 - 4 eq. of Silia*Bond* to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to afford isocyanate free solution	All solvents
	Silia*MetS* Diamine	1.4		All solvents
	Silia*MetS* Triamine	1.2		All solvents
Anhydrides	Silia*Bond* Amine	1.6	- Add 2 - 4 eq. of Silia*Bond* to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to afford anhydride free solution	All solvents
Chloroformates	Silia*Bond* Amine	1.6	- Add 2 - 4 eq. of Silia*Bond* to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to afford chloroformate free solution	All solvents
	Silia*MetS* Diamine	1.4		All solvents
	Silia*MetS* Triamine	1.2		All solvents

Scavenging Undesired Compounds: Nucleophile Scavengers

Nucleophile Scavenger

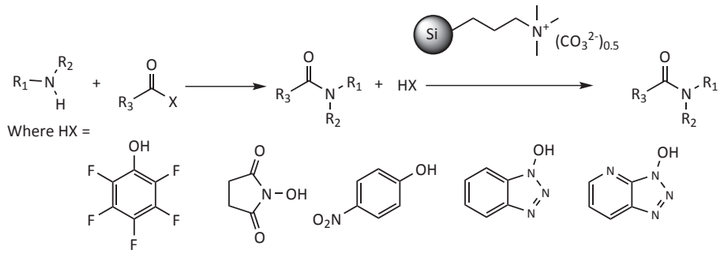
Function to be scavenged	Recommended Silia*Bond* scavenger	Loading (mmol/g)	Typical conditions	Solvent compatibility
Acids or acidic phenols	Silia*Bond* Amine	1.6	- Add 2 - 4 eq. of Silia*Bond* to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to afford acid free solution	All solvents
	Silia*MetS* Diamine	1.4		All solvents
	Silia*MetS* Triamine	1.2		All solvents
	Silia*Bond* Carbonate	0.7		Organic solvents
	Silia*Bond* TBD	0.9		All solvents

Amine free basing using Silia*Bond* Carbonate

Trifluoroacetic acid (*TFA*) is certainly the most commonly used ion-pairing agent for the separation of peptides in reversed-phase chromatography. The role of TFA is to act as a buffer, keeping the charge on the analyte and avoiding precipitation, to impart some hydrophobicity to the amino groups and to neutralize cationic charges. Silia*Bond* Carbonate is an efficient and convenient solution to this problem. See page 180 of this catalog for more details.

Scavenging phenols and acids with Silia*Bond* Carbonate

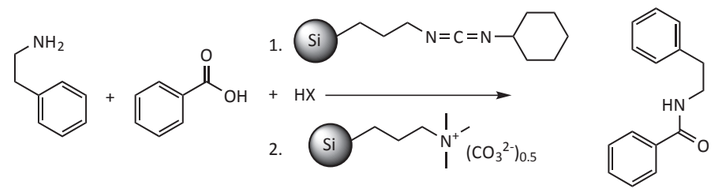
The efficiency of Silia*Bond* Carbonate as a scavenger of various coupling reagents (HX), including pentafluorophenol, N-hydroxysuccinimide (HOSu *or* NHS), 4-nitrophenol, 1-hydroxybenzotriazole (HOBt), and 1-hydroxy-7-azabenzotriazole (HOAt) is shown below, as well as a comparison with 2 suppliers of polymer-supported carbonate.



Scavenging Phenols Results

HX	Silia*Bond* Carbonate		Polymer 1		Polymer 2	
	5 min	60 min	5 min	60 min	5 min	60 min
Pentafluorophenol[1]	2	2	8	5	15	6
N-Hydroxysuccinimide	7	< 5	59	36	60	58
4-Nitrophenol	6	4	11	5	23	12
1-Hydroxybenzotriazole[2]	12	4	32	8	74	4
1-Hydroxy-7-azabenzotriazole[2]	3	3	28	4	70	8

Initial concentration: 5,000 ppm - 3 eq. of Silia*Bond* Carbonate. Analyzed by UV. [1] Analyzed by GC-MS, [2] in THF



Amide Coupling Results

HX	Yield (%)	Purity (%)
No Catalyst	35.4	95.1
Hydroxysuccinimide[1]	67.2	98.0
1-Hydroxybenzotriazole[2]	98.9	97.7
1-Hydroxy-7-azabenzotriazole[2]	100	99.2

1.0 eq. of amine, 1.5 eq. acid, 1.7 eq. catalyst (*HX*), 2.0 eq. Silia*Bond* Carbodiimide, 7.0 eq. Silia*Bond* Carbonate. Yield refers to the mass of isolated product. Purity was determined by GC-FID. [1] in DCM, [2] in THF

Related publication

P. Wipf at al., *Tetrahedron, 61,* **2005**, 11488.
B. Desai at al., *Tetrahedron, 62,* **2006**, 4651.
S. Mao et al., *J. Comb. Chem.*, *10*, **2008**, 235.
T. Emmerich et al., *Bioorg. Med. Chem. lett.*, *20*, **2010**, 232.
D. R. Saueur at al., *Org. Lett.*, *5*, **2003**, 4721.
S. Werner at al., *J. Comb. Chem.*, *9*, **2007**, 677.

Scavenging Undesired Compounds: Nucleophile Scavengers (*con't*)

Nucleophile Scavenger

Function to be scavenged	Recommended SiliaBond scavenger	Loading (mmol/g)	Typical conditions	Solvent compatibility
Alcohols	SiliaBond Tosyl Chloride	1.0	- Add 2 - 4 eq of SiliaBond to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to remove alcohol from solution	Anhydrous aprotic solvents and unstable in DMF
Alkoxides	SiliaBond Tosyl Chloride	1.0	- Add 2 - 4 eq of SiliaBond to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to obtain alkoxide-free solution	Anhydrous aprotic solvents and unstable in DMF Anhydrous aprotic organic solvents
	SiliaBond Isocyanate	1.2		
Amines (*primary, secondary or anilines*)	SiliaBond Carboxylic Acid	1.4	- Add 2 - 4 eq of SiliaBond to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to remove amine from solution	All solvents
	SiliaBond Tosic Acid	0.8		All solvents
	SiliaBond Propylsulfonic Acid	1.0		Allsolvents
	SiliaBond Isocyanate	1.2		Anhydrous aprotic organic solvents
	SiliaBond Tosyl Chloride	1.0		

Scavenging of amine with SiliaBond Isocyanate

Scavenging Amines Results

Scavenger	Benzylamine	Aniline
SiliaBond Isocyanate	98.7	94.4
Polymer 1	100	98.9
Polymer 2	100	99.2

Conditions: 3 eq. relative to amine, 1 h at room temperature in DCE
% scavenged determined by GC-MS

Scavenging of benzylamines with SiliaBond Isocyanate in different solvents

Scavenging Benzylamine Results

Scavenger	THF	DCM	ACN
SiliaBond Isocyanate	> 98%	> 98%	95%
Polymer 1	> 98%	> 98%	79%
Polymer 2	> 98%	> 98%	88%

Conditions: 3 eq. relative to amine, 1 h at room temperature
% scavenged determined by GC-MS

Nucleophile Scavenger

Function to be scavenged	Recommended SiliaBond scavenger	Loading (mmol/g)	Typical conditions	Solvent compatibility
Boronic acids	SiliaBond Carbonate	0.7	- Add 2-4 eq of SiliaBond to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to yield boronic acid-free solution	Organic solvents
	SiliaBond Diol	1.0		All solvents
	SiliaBond TBD	0.9		All solvents

Scavenging boronic acids with SiliaBond Diol

Scavenging Boronic acids Results

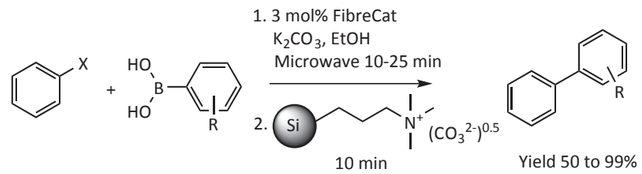
Equivalent	Time	Efficiency
2	1 h	75%
4	1 h	100%

Conditions: 2-4 eq. relative to boronic acid, 1 h at room temperature
% scavenged determined by GC-MS

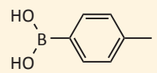
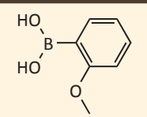
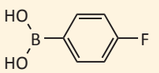
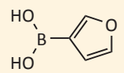
Scavenging boronic acids with SiliaBond Carbonate

Related publication

Y. Wang and D. R. Sauer, *Org. Lett.*, *6*, **2004**, 2793.



Scavenging Boronic acids Results

Equivalent				
10	100%	100%	100%	100%

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Scavenging Undesired Compounds: Nucleophile Scavengers (*con't*)

Nucleophile Scavenger

Function to be scavenged	Recommended Silia*Bond* scavenger	Loading (mmol/g)	Typical conditions	Solvent compatibility
Hydrazines	Silia*Bond* Tosyl Chloride	1.0	- Add 2 - 4 eq. of Silia*Bond* to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to remove hydrazine from solution	Anhydrous aprotic solvents Unstable in DMF
Organometallics	Silia*Bond* Tosyl Chloride	1.0	- Add 2 - 4 eq. of Silia*Bond* to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to obtain organometallic-free solution	Anhydrous aprotic solvents Unstable in DMF
Thiol or thiolates	Silia*Bond* Isocyanate	1.2	- Add 2 - 4 eq. of Silia*Bond* to the reaction mixture - Stir for 1 h at room temperature - Filter off the scavenger and wash with solvent to yield thiol-free solution	Anhydrous aprotic organic solvents
	Silia*Bond* Maleimide (*thiol*)	0.7		Polar solvents (*DMF, MeOH and H_2O*)

Catch and Release of the API

Catch and Release the API

Function to be isolated	Recommended Silia*Bond* scavenger	Loading (mmol/g)	Typical conditions
Amines	Silia*Bond* Tosic Acid (*SCX*)	0.8	- Catch the amine on the Silia*Bond* - Wash with methanol - Release with a solution of 2 M NH_3 in methanol
	Silia*Bond* Propylsulfonic acid (*SCX-2*)	1.0	
Carboxylic acids	Silia*Bond* TMA Acetate (*SAX-2*)	1.0	- Catch the carboxylic acid on the Silia*Bond* - Wash with methanol - Release with 2% AcOH in MeOH or 1% HCl in ACN

Scavenging 2-Iodobenzoic Acid using Silia*Bond* TMA Acetate and Carbonate

Dess Martin Periodinane (*DMP*) is a mild and chemoselective oxidant. It is readily accessible, environmentally benign and has a good shelf-life. Further, the ease of handling, simple reaction work-up, product purification and good yields obtained with DMP make it a valuable reagent in organic synthesis.

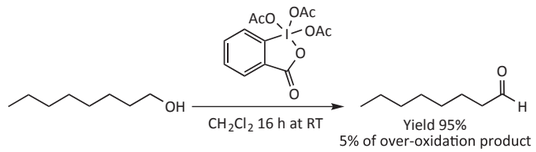
2-Iodobenzoic acid is the degradation product from DMP formed during the work-up. Most of it can be removed with a basic work-up, but sometimes, it can be difficult to get rid of all this side product.

General Procedure

A solution of 1-octanol (*1 mmol*) in CH_2Cl_2 (*6 mL*) at room temperature, was added to DMP (*1.1 mmol*). The reaction mixture was stirred for 16 h, then diluted with 35 mL of MTBE and poured in 20 mL of an aqueous solution of $Na_2S_2O_3$ (*25%*). The mixture was stirred for 10 min. Another portion of 35 mL of MTBE was added for the liquid-liquid extraction. The MTBE phase was then washed with water[1] and a saturated aqueous solution of NaCl (*10 mL*) and dried on $MgSO_4$.

Scavenging was done using Silia*Bond* TMA Acetate or Carbonate, both in bulk (*1 g*) and SPE cartridge (*6 mL/1 g*) for comparison purposes. Each sample was washed or eluted with a fresh portion of MTBE (*8 mL*) and then the 2-iodobenzoic acid was monitored by GC-MS against an internal standard. Over-oxidation product (*carboxylic acid*) was scavenged with Silia*Bond* scavengers.

[1]The usual $NaHCO_3$ wash was intentionally omitted in order to get significant amount of residual 2-iodobenzoic acid in the final solution.

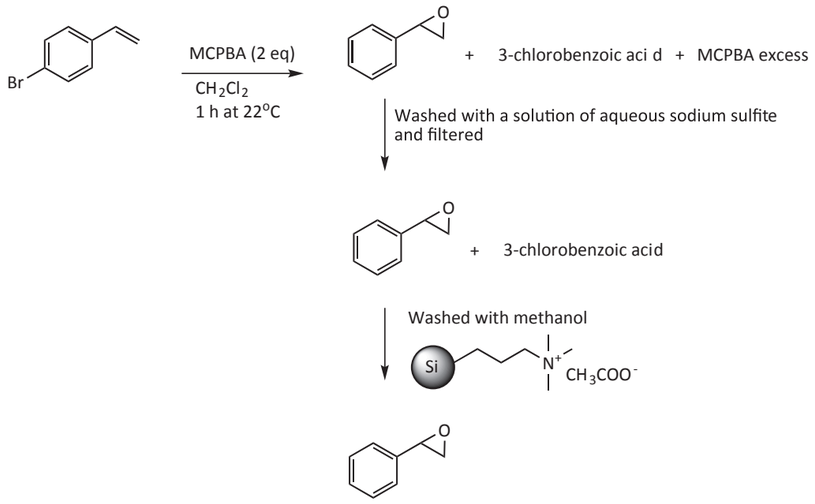


Scavenging 2-Iodobenzoic acid Results (%)

Sorbent	Bulk	Silia*Prep*
Silia*Bond* TMA Acetate	100	100
Silia*Bond* Carbonate	100	100

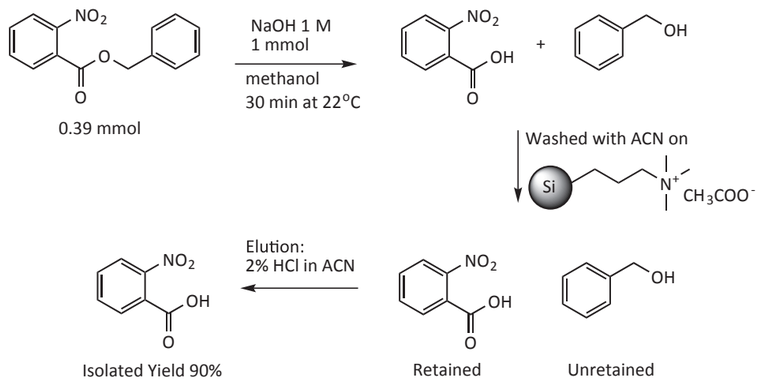
Catch and Release of the API (*con't*)

Ester hydrolysis purification using Silia*Bond* TMA Acetate



Yield of isolated product 88%

Ester hydrolysis purification using Silia*Bond* TMA Acetate



0.39 mmol

Isolated Yield 90%

Retained

Unretained

Silia*Bond* Ordering Information

Silia*Bond* Organic Scavenger Part Numbers

Scavenger	Part Number	Available Quantity
Silia*Bond* Amine	R52030B	5 g 10 g 25 g 50 g 100 g 250 g 500 g 1 kg 5 kg 10 kg 25 kg ... Multi-Ton Call us for details
Silia*Bond* Carbonate	R66030B	
Silia*Bond* Carboxylic Acid	R70030B	
Silia*MetS* Diamine	R49030B	
Silia*Bond* Diol	R35030B	
Silia*Bond* DMAP	R75530B	
Silia*Bond* Isocyanate	R50030B	
Silia*Bond* Maleimide	R71030B	
Silia*Bond* Piperazine	R60030B	
Silia*Bond* Propylsulfonic Acid	R51230B	
Silia*Bond* TBD	R68530B	
Silia*Bond* TMA Acetate	R66430B	
Silia*Bond* Tosic Acid	R60530B	
Silia*Bond* Tosyl Chloride	R44030B	
Silia*Bond* Tosyl Hydrazine	R61030B	
Silia*MetS* Triamine	R48030B	

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SiliaFlash®

Irregular Silica Gels

SILICYCLE inc.
UltraPure SILICA GELS

Chromatography at SiliCycle

SiliCycle is your partner of choice for your purification and chromatography needs.

Recognized worldwide as a leader with an outstanding quality silica gel, SiliCycle offers one of the largest selections of silica, available in different shapes, on the market.

- **Silia*Flash*®** Irregular silica
- IMPAQ® Angular silica
- **Silia*Sphere*™** Spherical silica

Ensure Unbeatable Performance with SiliCycle.

UltraPure Silica Gel from SiliCycle

SiliCycle: Silica expert.

With pore diameters ranging from 40 to 150 Å and particle sizes from 5 to 1,000 microns, SiliCycle offers products to meet all your application requirements. This is one of the most reliable portfolios for flash and gravity grades for medium to high pressure.Our silica gels are ideal for both analytical and preparative chromatography, from laboratory to pilot-plant processes and production scales.

Features & Benefits of Silia*Flash*, IMPAQ & Silia*Sphere*

Features	Benefits
High purity silica gels	Consistency, reliability, reproducibility
Exempt of fine particles or very low level of fines	No contamination, lower backpressure, superior separation
Exceptional narrow particle and pore-size distribution	Optimal separation and resolution
Batch-to-batch, year-to-year consistency	Reliable chromatography
Neutral pH	Wide range of products can be purified, even acid sensitive ones
Low metal content & controlled water content	Symmetrical peaks with no tailing
High mechanical stability	Can be used under high pressures without surface abrasion
High surface area and density	Greater loading capacity, enabling more silica for the same volume Solvent economy (*smaller dead volume*)
Availability in bulk quantities at affordable pricing	Always in stock with on-time delivery

Together, all these benefits mean optimal and reproducible separation power, saving you time and money.

Silia*Flash* Irregular Silica Gels

- **Consistency, Reliability, & Reproducibility***
- **Tight Particle and Pore Size Distributions**

The quality of a silica gel is extremely important when you are using it for chromatography purposes, particularly when dealing with difficult separations of valuable compounds. You need to be extremely confident about your recoveries.

SiliCycle is recognized worldwide as a leader in chromatography and purification with our outstanding quality products. SiliCycle's expertise and strong knowledge has been acquired over the years and this distinguishes us from the competition.

Note: characteristics listed on following pages can also be applied to IMPAQ & Silia*Sphere* brands.

High Purity Silica Gel

You can be sure of the outstanding quality of SiliCycle's silica gels because of the closely controlled manufacturing conditions at our ISO 9001:2008 certified state-of-the-art facilities. Our tight control of every manufacturing process step, affords identical and reproducible properties (*chemical, physical and structural*) as well as ensuring the same chromatographic selectivities. Hence, Silia*Flash* is suitable for validated chromatographic processes.

Furthermore, our stringent Quality Control and Quality Assurance ensures high performance with no scale-up limitations. Every product meets our quality specifications and is shipped with a Certificate of Analysis (*CofA*). Individual data sheets are also available directly from our website.

Every day, SiliCycle's Silia*Flash* products are being used by thousands of satisfied scientists for their purifications. They know that Silia*Flash* is synonymous of quality and that they know they will have reproducible results every time.

Silia*Flash* - Now Exempt of Fines*

Over the years, in our quest to improve and provide the best quality products, SiliCycle has continuously reviewed how it can make a difference for you. At SiliCycle, a major improvement on our most popular silica gel (*Silia*Flash* 40-63 microns, 60 Å*) has been the absence of fines (*small particles under 10 microns*).

In chromatography, fine particles increase backpressure and can result in clogging which is particularly dangerous when using glass columns. Fines can also pass through filters and contaminate final products. The lack of fines gives a more regular, stable, and reproducible chromatography bed and a faster and more even flow rate for better separation.

- **This improvement comes with NO EXTRA COST to you.**

*Other Silia*Flash* products have the lowest level of fines on the market.

Silia*Flash*'s Exceptional Characteristics

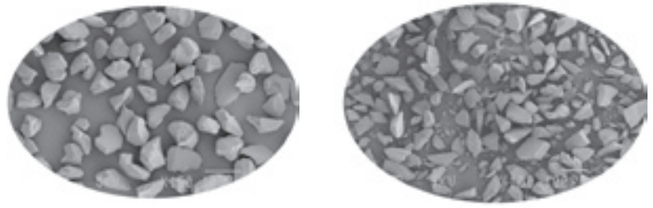
Tight Particle and Pore Size Distributions

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

Importance of tight distributions in chromatography

Tight particle size distribution	Tight pore size distribution
Greater column performance and separation	Optimal peak shape - Presence of smaller pore size leads to peak tailing
Tighter peaks and better peak shape	surface area - Presence of bigger pore size leads to lower surface availability
Better column packing, easier to pack	No molecule sequestration due to fluid diffusion inside pores
No preferential pathways (*channeling*)	
Faster flow rate with lower back-pressure	
Time and solvent savings	

Scanning Electron Microscopy (*SEM*) Comparison of Two Silica Gels 40 - 63 µm, 60 Å



SiliCycle

Competitor

Particle Size Analysis Methods

Laser Diffraction (*Malvern Analysis*)

Usually used for particle sizes below 40 microns. Particle size distributions are reported in term of D10, D50 (*average, mean*) and D90. Some manufacturers also mention the ratio of D90/D10.

Sieving

Usually for particle sizes over 40 microns. Particle size distribution is reported in percentage of undersized and oversized.

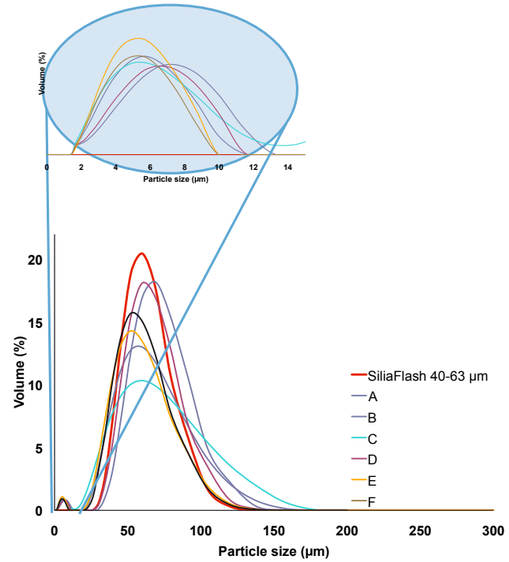
Tighter Particle Size Distribution

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 take into account 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 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 microns and a very low level of particles below 40 microns (*or "fines"*). Fines can cause several problems such as higher backpressure, clogging, contamination (*see previous section for more details*). SiliCycle has the lowest level of fines on the market.

The absence of fines gives a more regular, stable, and reproducible chromatography bed, which results in a faster and more even flow rate for better separation.

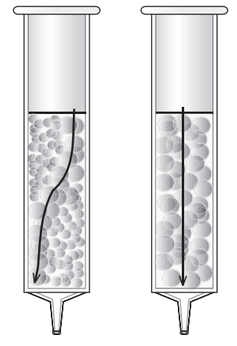


Effects of Homogeneous vs Uneven Packing

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 compared with 80% for most of the competitors.

The connection between particle size distribution and column performance is very simple. When the distribution is broad, the 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. As they exit the column, the compounds will give broad and poorly separated peaks.

The figure to the right illustrates the effect of a wide particle size distribution versus a narrow one. Narrower particle size distribution gives a more homogenous packing and thus more concentrated fractions. And, by reducing solvent consumption, the process will be more cost-efficient.



Low Trace Metal Content

Irregular silica, depending on its method of manufacturing, normally contains trace quantities of a variety of metals. This can, in turn, affect the quality of the separation. Aluminum, iron and lead are particularly problematic because they cause peak tailing. SiliCycle's proprietary technology generates a silica gel with the lowest trace metal content on the market today.

As shown in the table below, trace metal concentration in SiliCycle's silica gel is significantly lower than flash silica gels from other manufacturers. Our low trace metal content ensures you will get optimal performance from your chromatography. Tight control of trace metals in every batch also improves your reproducibility and reduces risks of interaction between metals and desired compounds.

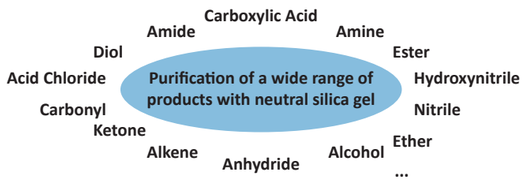
Typical Trace Metal Concentration

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

Stable Water Level Content

Water level of silica gel affects the selectivity of the silica. Silia*Flash* has a water content between 4 to 6%. This is advantageous for you since the other products have a water variation from 2 to 9% depending on the manufacturer. SiliCycle can also adjust the water level upon request.

Neutral pH & High Surface Area



Neutral pH

Our Silia*Flash* are pH-adjusted between 6.5 and 7.5 to be safely used in the separation of a wide range of products (*a neutral pH is needed to separate pH-sensitive compounds*). Once again, this is advantageous when compared to the pH range of 6 to 7 often seen in the market.

High Surface Area

Higher surface area provides greater separation power.

SiliCycle, the Silica Supplier for Every Need

With SiliCycle, No Scale-up Limitations

Each year, SiliCycle manufactures hundreds of tons of Silia*Flash*, a broad range of silica gels for chromatography applications. All our products are manufactured under tightly controlled manufacturing processes, and stringent quality control insured the highest quality.

Be confident in scaling-up your processes with our Silia*Flash*. Performance will remain the same with every particle size.

Scaling-up from laboratory to production scale

SiliCycle Has One of the Largest Selections Available

SiliCycle offers one of the largest selections of silica-based products, from bare to various functionalized silicas, required for chromatography.

These products are available in different pore diameters (*from 40 to 1,000 Å*), particle sizes (*from 5 to 1,000 µm*) and particle shape (*irregular, angular or spherical*) to provide a solution for a wide range of applications, performance and economic requirements.

All of these products are available from laboratory scale to multi-ton quantities.

Silia*Flash* is also available in fixed bed format: Silia*Sep* Flash Cartridges (*see page 158*) & Silia*Prep* SPE cartridges (*see page 173*).

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Silia*Flash* Ordering Information

Silia*Flash* Ordering Information

Product Number	Name	Particle Size (µm)	Pore Diameter (Å)
R10030A	F40	40 - 63	40
R10040A	G40	60 - 200	40
R10070A	B40	200 - 500	40
R10010B	C60	0 - 20	60
R10013B	I60	15 - 25	60
R10014B	A60	5 - 20	60
R10015B	S60	15 - 35	60
R10017B	E60	15 - 40	60
R10019B	D60	10 - 30	60
R10023B	R60	20 - 45	60
R10030B	F60	40 - 63	60
R12030B	P60	40 - 63	60
R10040B	G60	60 - 200	60
R10050B	M60	60 - 120	60
R10060B	L60	120 - 200	60
R10070B	B60	200 - 500	60
R10080B	N60	500 - 1,000	60
R10015D	S90	15 - 35	90
R10030D	F90	40 - 63	90
R10040D	G90	60 - 200	90
R10070D	B90	200 - 500	90
R10040H	G150	60 - 200	150
R10050H	M150	60 - 120	150
R10060H	L150	120 - 200	150
R10072H	B150	250 - 500	150

pH (5% w/w): 6.5 - 7.5, Volatile content: ≤ 7
Formats : 1kg, 5kg, 10kg, 25kg, ... up to multi-ton scale

Tip: Silica gel standardization is possible by eliminating the residual moisture. Place the silica inside a vacuum oven and heat at 130 °C during 30 minutes. Cool to room temperature and pack column.

Particle Size Conversion Table

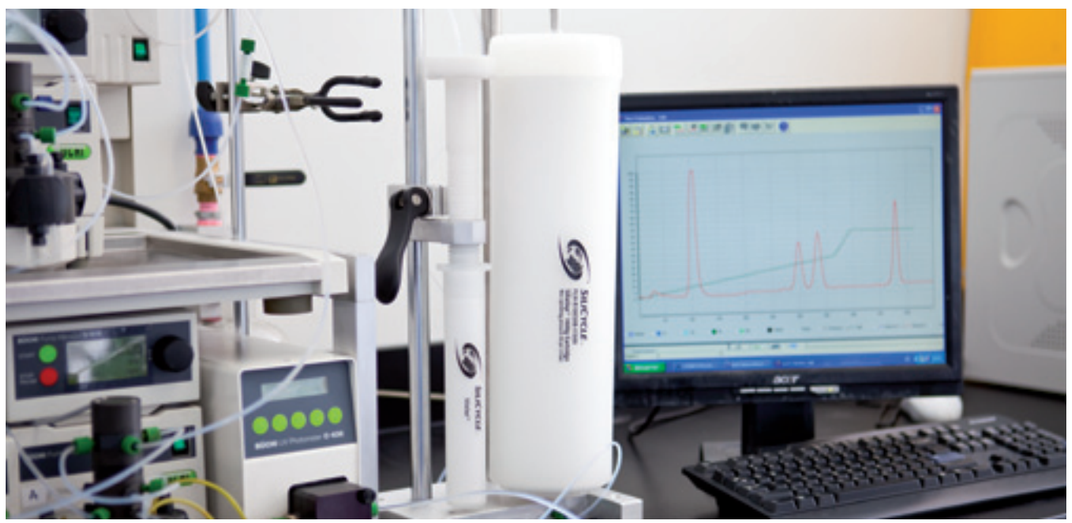
Conversion Table Microns vs Mesh

Microns	Mesh	Microns	Mesh
5 - 20	625 - 2500	60 - 120	120 - 230
15 - 25	~ 325 - 625	60 - 200	70 - 230
15 - 40	~ 400 - 1,250	120 - 200	70 - 120
20 - 45	325 - 625	200 - 500	35 - 70
40 - 63	230 - 400	500 - 1,000	18 - 35

A Particle Size for Each Application

Most Popular Particle Size Applications

Particle Size Distribution	Application
Particles for Preparative TLC Plates	
0 - 20 µm 5 - 15 µm 5 - 20 µm	• Contain neither binder (*organic or inorganic*) nor UV indicator (*F254*) • Can also be used in flash chromatography if higher resolution is required (*higher back-pressure*)
Specialized Particles for Difficult Separations	
15 - 35 µm 15 - 40 µm	• High-resolution silica for difficult separations (*similar polarities*)
Particles for Flash Chromatography	
40 - 63 µm	• Chromatography types: high-resolution flash chromatography & low to medium-pressure preparative chromatography • Narrow particle size over other flash chromatography silica • Easier to pack • More uniform packing • Superior resolution • Suitable for uses with complex matrices MOST POPULAR PARTICLE SIZE DISTRIBUTION!
60 - 120 µm	• Alternative to 40-63 µm silica for faster flow rate without pressure
Particles for Column (*or Gravity*) Chromatography	
60 - 200 µm	• Most economical silica for open column chromatography (*gravity*) • Suitable for rough purification and large-scale preparative chromatography • Easier to handle • Purification cost reduction
120 - 200 µm	• Silica for standard open column chromatography • Narrow particle size enables uniform packing • Suitable for mass overload purification



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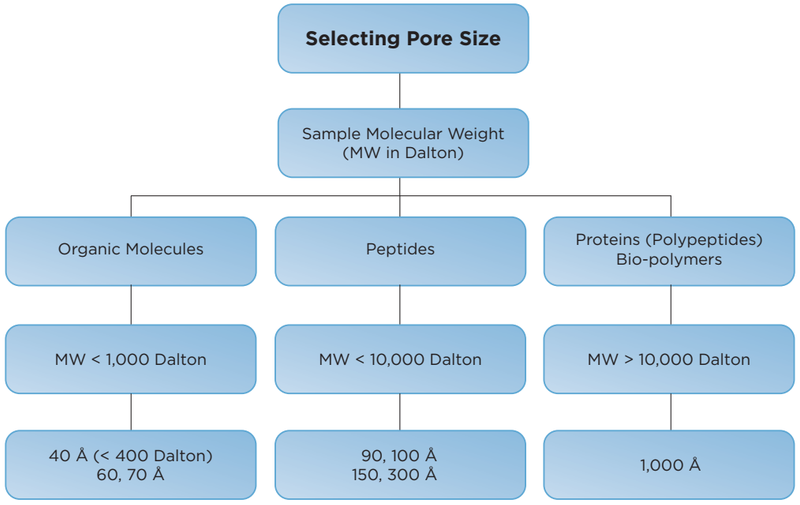
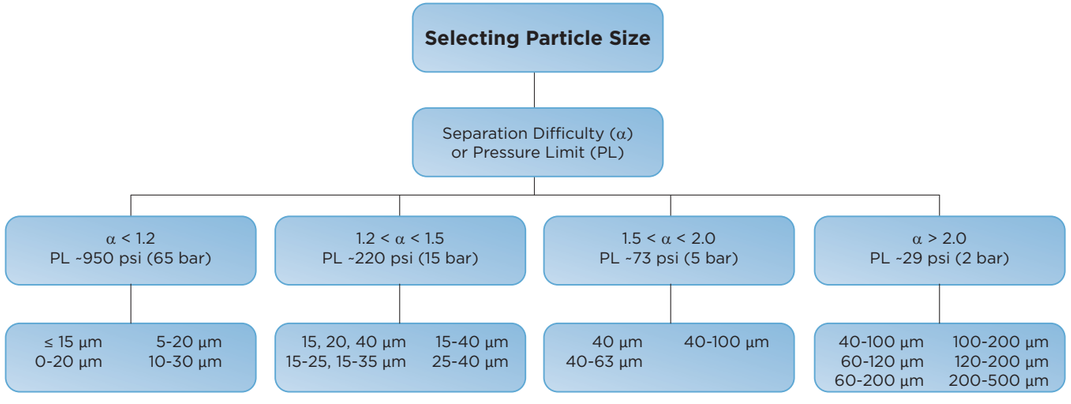
Silica Selection Guide

SiliCycle offers a wide range of Silia*Flash*, Silia*Sphere* and IMPAQ products to cover many types of applications. Selecting the most appropriate sorbent for any given application can be difficult. To help you choose the right media (*bonded or not*), our experts recommend using the diagram below as a guide. Simply follow the three pathways to select the most suitable sorbent.

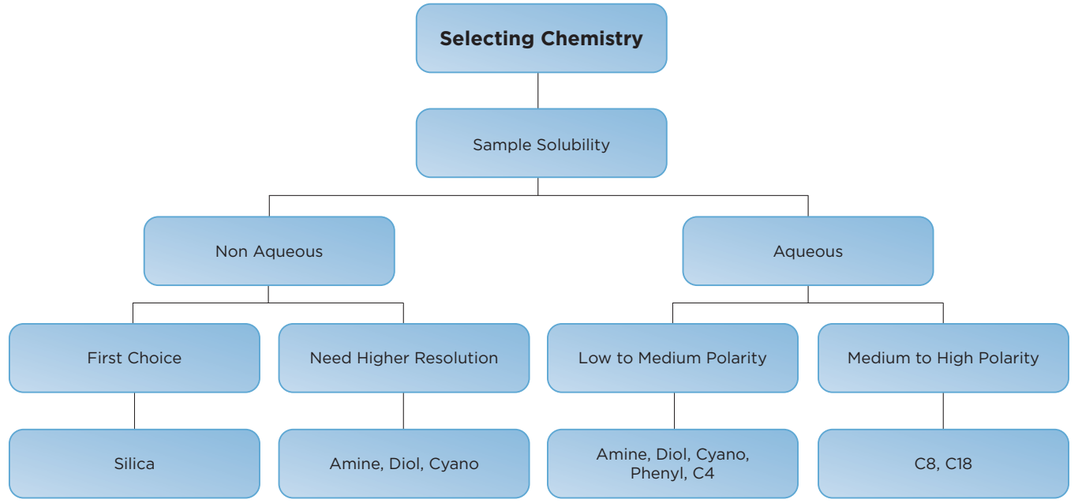
Selecting Pore Size



Selecting Particle Size



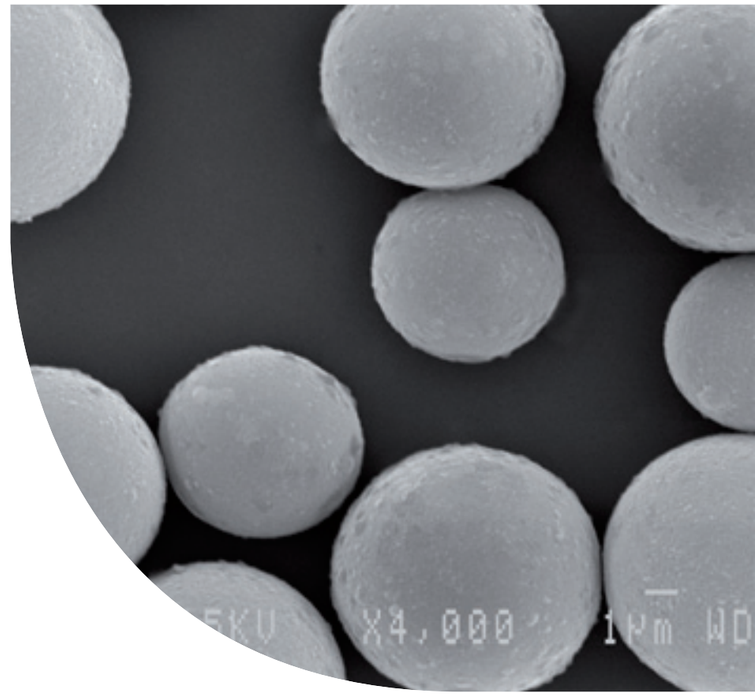
Selecting Chemistry



Note: Standard functionalized sorbents are 40-63 µm, 60 Å

SiliaSphere™

Spherical Silica Gels

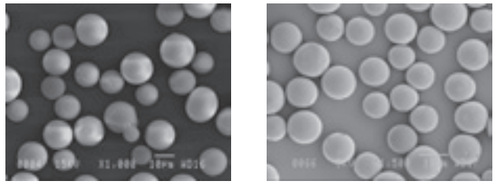


SILICYCLE Inc.
UltraPure SILICA GELS

Silia*Sphere* Spherical Silica Gel

SiliCycle is your partner of choice for your purification and chromatography needs.

- SiliCycle has improved large-scale production of its Silia*Sphere* spherical silica support. You will be happy to see that the quality is superior due to a narrower particle size distribution.
- Silia*Sphere* spherical silica gels present great advantages for your preparative chromatography applications.



Old version
S10007G

New version
S10007G-A

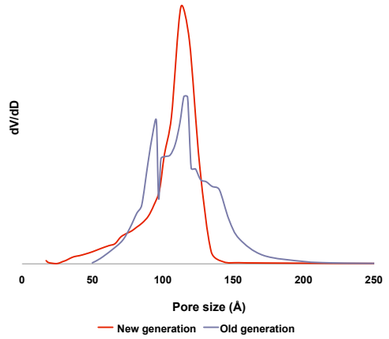
SEM picture of 10 µm

Silia*Sphere* Monodispersed Spherical Silica Gels

Our Silia*Sphere* family of silicas are monodispersed spherical silica gels with particle sizes from 1.8 to 15 µm. The 1.8, 2.2, 3, and 5 µm gels are used in analytical scale chromatography. The 10 and 15 µm gels are used in preparative chromatography. Available in 60, 80, 100, 120 and 300 Å pore sizes.

Our Silia*Sphere* are characterized by low metal content to avoid specific interaction between acid sites and analytes as well as high mechanical stability and very high purity.

The Silia*Sphere* 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 preciitation afford spherical silica gels with the desired characteristics.



The Silia*Sphere* family is characterized by a very low metal content and exceptionally stable at the low or high pH. The Silia*Sphere* manufacturing process ensures quality and reproducibility in pore size, surface area and particles sizes and morphology. The high specific surface area enables a high loading capacity with a uniform and reproducible coverage.

Please note that we are able to provide all our functionalized products (C18, C8, amine, cyano, diol, etc.) on any spherical silica gel presented in this catalog. Contact us for details!

Silia*Sphere* Product Overview

Silia*Sphere* Monodispersed Spherical Silica Gels

Product Number	Particle Size (µm) D50	Pore Diameter (Å)	Pore Volume (mL/g) / Spec. Surf. Area (m^2/g)	BONDED C18 mono Product Number
BARE Silia*Sphere* Monodispersed Spherical Silica				**C18 mono**
S10007B-A	10	60	0.85 - 1.15 / ≥ 600	S03207B-A
S10008B-A	15	60	0.85 - 1.15 / ≥ 600	S03208B-A
S10003F-A	3	80	0.85 - 1.15 / ≥ 450	S03203F-A
S10005F-A	5	80	0.85 - 1.15 / ≥ 450	S03205F-A
S10007F-A	10	80	0.85 - 1.15 / ≥ 450	S03207F-A
S10008F-A	15	80	0.85 - 1.15 / ≥ 450	S03208F-A
S10003E-A	3	100	0.85 - 1.15 / ≥ 400	S03203E-A
S10005E-A	5	100	0.85 - 1.15 / ≥ 400	S03205E-A
S10007E-A	10	100	0.85 - 1.15 / ≥ 400	S03207E-A
S10008E-A	15	100	0.85 - 1.15 / ≥ 400	S03208E-A
S10001G-A	1.8	120	0.85 - 1.15 / ≥ 300	S03201G-A
S10002G-A	2.2	120	0.85 - 1.15 / ≥ 300	S03202G-A
S10003G-A	3	120	0.85 - 1.15 / ≥ 300	S03203G-A
S10005G-A	5	120	0.85 - 1.15 / ≥ 300	S03205G-A
S10007G-A	10	120	0.85 - 1.15 / ≥ 300	S03207G-A
S10008G-A	15	120	0.85 - 1.15 / ≥ 300	S03208G-A
S10003M	3	300	0.75 - 1.05 / ≥ 80	S03203M
S10005M	5	300	0.75 - 1.05 / ≥ 80	S03205M
S10007M	10	300	0.75 - 1.05 / ≥ 80	S03207M
S10008M	15	300	0.75 - 1.05 / ≥ 80	S03208M
S10007T	10	1,000	0.75 - 1.05 / ≥ 20	S03207T
S10008T	15	1,000	0.75 - 1.05 / ≥ 20	S03208T

pH (5% w/w): 4 - 7, Volatile content: ≤ 10
Formats : 100g, 500g, 1kg, 5kg, 10kg, 25kg, ... up to multi-ton scale

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Silia*Sphere* PC (*Preparative Chromatography*)

Cost is very important in preparative and process chromatography, 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 may not provide the desired results. For these situations, SiliCycle has developed a second class of spherical silica particles for preparative chromatography. The advantage of using Silia*Sphere* PC materials over standard silica gels includes the following:

- Increased efficiency of the eluent's flow characteristics
- Improvement of the resolution between compounds of a sample
- Ease of packing

Silia*Sphere* for Preparative Chromatography

Product Number	Particle Size (µm) D50	Pore Diameter (Å)	Surface Area (m^2/g)	Product Number	Particle Size (µm) D50	Pore Diameter (Å)	Surface Area (m^2/g)
S10030B-A	50	60	≥ 650	S10020M	30	300	≥ 100
S10034B-A	75	60	≥ 650	S10030M	60	300	≥ 100
S10040B-A	100	60	≥ 650	S10040M	100	300	≥ 100
S10063B-A	150	60	≥ 650	S10020T	30	1,000	≥ 50
S10030G-A	50	150	≥ 290	S10030T	60	1,000	≥ 50
S10040G-A	100	150	≥ 290	S10040T	100	1,000	≥ 50

Formats : 250g, 1kg, 5kg, 10kg, 25kg, ... up to multi-ton scale

IMPAQ Angular Silica Gels

The IMPAQ angular silica gels are a good alternative to spherical material for preparative applications as they provide very efficient separations at a much lower price. IMPAQ is premium-grade angular silica designed for preparative chromatography where consistent high purity and narrow particle distribution and pore dimension are required. IMPAQ is a porous silica gel in which the surface area, porosity and rigidity have been optimized for loading capacity and mechanical stability.

IMPAQ for Preparative Chromatography

Product Number	Particle Size Distribution (µm)		Pore Diameter (Å)	Pore Volume (mL/g)	Spec. Surface Area (m^2/g)	pH (5% w/w)
	D50	D10/D90				
B10007B	10 µm	≤ 1.8	60	0.70 - 0.85	≥ 450	≥ 4
B10009B	20 µm	≤ 1.8	60	0.70 - 0.85	≥ 450	≥ 4
B10025B	40 µm	≤ 2.1	60	0.70 - 0.85	≥ 450	≥ 4
B10007E	10 µm	≤ 1.8	100	1.0 - 1.4	≥ 400	≥ 6
B10009E	20 µm	≤ 1.8	100	1.0 - 1.4	≥ 400	≥ 6
B10025E	40 µm	≤ 2.1	100	1.0 - 1.4	≥ 400	≥ 6

Formats : 100g, 1kg, 5kg, 10kg, 25kg, ... up to multi-ton scale

Silia*Bond*®
Chromatographic and Ion Exchange Phases

SILICYCLE inc. UltraPure SILICA GELS

Silia*Bond* Chromatographic and Ion Exchange Phases

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

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

Silia*Bond* Chromatographic Phases

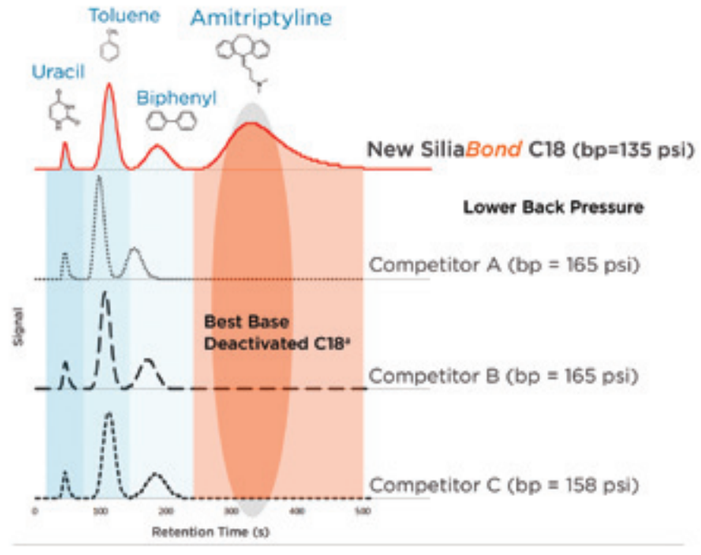
Silica is the most widely used matrix in chromatography. These bare and grafted supports possess great properties for use as stationary phases and are particularly appreciated for their high mechanical resistance. In chromatography, there are two phases: the stationary phase that is packed in a column and the mobile phase that will be eluted through the stationary phase. If the analyte is strongly soluble in the mobile phase, there will be no retention. If the analyte interacts strongly with the stationary phase, there will be no or low migration. In a mixture, the interactions between the two phases will generate the separation. So, depending on the analyte's polarity, the apropriate stationary phase has to be chosen, and the mobile phase's polarity has to be tuned.

Silia*Bond* Reversed-Phases

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

Last year, SiliCycle developed a new and innovative C18 chromatographic phase characterized by a homogeneous coverage of the alkyl chains on the surface. Consequently, the endcapping step is more controlled, which leads to much improved separations and also to inhibition of the non-specific interactions with silanol groups (*highly deactivated silanol phase*). This chromatographic phase is available on irregular (*R332-*) and spherical (*S032-*) high quality supports. This grafting process will be available soon for all other reversed phases.

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



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

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Silia*Bond* Reversed-Phases Portfolio

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

Silia*Bond* Reversed-Phases

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

Also available on all irregular Silia*Flash* Silica. Example: the 300 Å, 40-63 µm (*Rxxx30M*) [a]Based on our Standard Silia*Flash* Silica matrix R10030B, 40-63 µm, 60 Å

Typical applications using Silia*Bond* Reversed-Phases

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

Silia*Bond* Normal Phases

Normal-phase chromatography is used to separate polar compounds through polar interactions with the support. The interactions take place on the highly polar silanols of the silica gel surface, but there are also moderately polar interactions with the hydrogen bonds on amino or diol functions. The non-endcapped cyano phase can be used in applications in normal-phase chromatography as a less polar alternative to silica. The $AgNO_3$ phase is particularly useful to separate isomers that present unsaturated groups.

Silia*Bond* Normal Phases

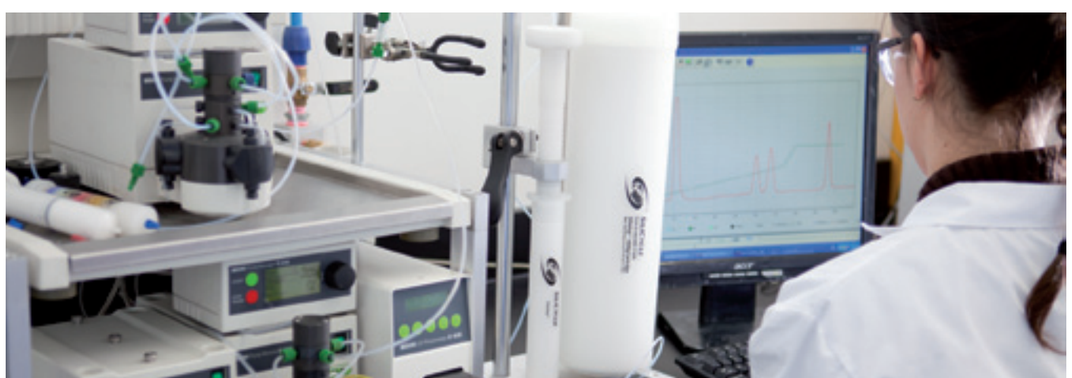
Sorbent Phase	Functional Group	Endcapping	Loading[a]	Density (g/mL)	SiliCycle P/N
SiO_2	Bare silica gel	No	N/A		R10030B
NH_2 *nec*	Amine	No	1.6	0.687	R52130B
CN *nec*	Cyano	No	1.0		R38130B
Diol *nec*	Diol	No	1.0	0.687	R35030B
$AgNO_3$	Silver Nitrate	No	10% w/w	0.604	R23530B

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

[a] Based on our Standard Silia*Flash* Silica matrix R10030B, 40-63 µm, 60 Å

Typical applications using Silia*Bond* Normal Phases

Sorbent Phase	Typical Applications
NH_2 *nec*	Sugars, nucleotides and water-soluble vitamins
CN *nec*	Polar organic compounds such as basic drugs and molecules containing π electron systems
Diol *nec*	Peptides, proteins and malto-oligosaccharides
$AgNO_3$	Cis/trans isomers of unsaturated compounds such as alkenes, lipids, steroids and terpenes



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Silia*Bond* Ion Exchange Phases

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

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

- Strong cation exchanger such as our Silia*Bond* Tosic Acid (*SCX*) and Silia*Bond* Propylsulfonic Acid (*SCX-2*)
- Weak cation exchanger such as our Silia*Bond* Carboxylic Acid (*WCX*)

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

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

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

Silia*Bond* Ion Exchange Phases

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

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

[a] Based on our Standard Silia*Flash* Silica matrix R10030B, 40-63 µm, 60 Å

Silia*Bond* Ion Exchange Phases (*con't*)

Typical applications for using Silia*Bond* Ion Exchange Phases

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

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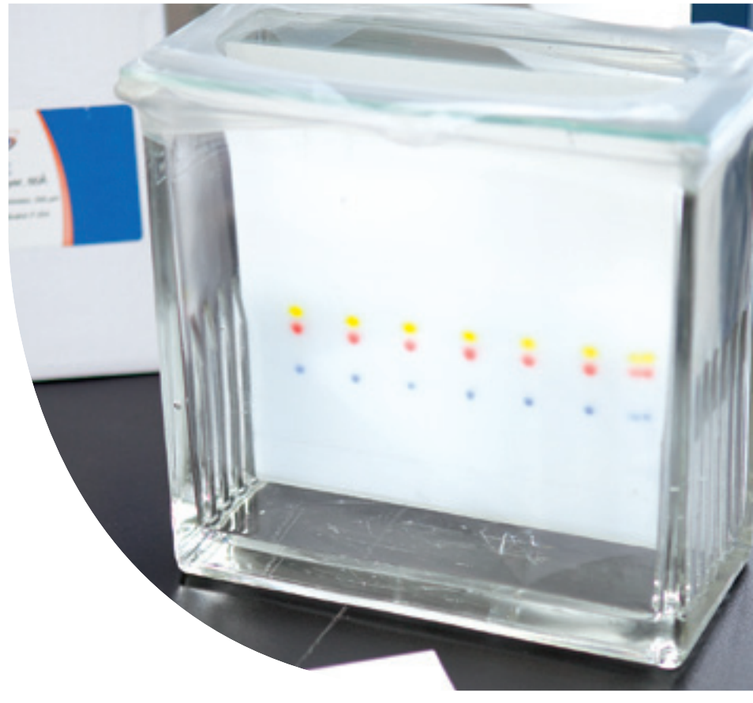
SiliaPlate™
TLC Plates



SILICYCLE Inc.
UltraPure SILICA GELS

UltraPure SiliaPlate™ for TLC

SiliCycle offers the possibility to analyse reactions on thin layer chromatography (*TLC*) support and tranfers the experiment on flash columns on the same SiliaFlash silica support. Maximixe the benefits by using our UltraPure SiliaPlate TLC plates with an extra hard layer of silica. For your convinience SiliCycle offers different sizes, choice of backing and reversed & speciality plates. All our SiliaPlate TLC have an indicator (*F254*).

UltraPure SiliaPlate TLC with different backing

SiliCycle P/N	Product Name	Plate Size (cm)	Thickness (µm)	#/box
SiliaPlate Al (*Aluminum*)				
TLA-R10011B-323	SiliaPlate Al	20 x 20	200	25
SiliaPlate Al C18				
TLA-R30411B-303	SiliaPlate Al C18	20 x 20	200	25
SiliaPlate G (*Glass*)				
TLG-R10011B-423	SiliaPlate G	2.5 x 5	250	25
TLG-R10011B-124	SiliaPlate G	2.5 x 7.5	250	100
TLG-R10011B-624	SiliaPlate G	2.5 x 10	250	100
TLG-R10011B-527	SiliaPlate G	5 x 10	250	200
TLG-R10011B-424	SiliaPlate G	5 x 20	250	100
TLG-R10011B-723	SiliaPlate G	10 x 20	250	25
TLG-R10011B-323	SiliaPlate G	20 x 20	250	25
TLG-R10011B-333	SiliaPlate G	20 x 20	500	25
Scored SiliaPlate G (*Glass*)				
TLGSR10011B-423	SiliaPlate G (*scored*)	20 x 20	250	25
TLGSR10011B-350	SiliaPlate G (*scored*)	20 x 20	250	100
TLGSR10011B-353	SiliaPlate G (*scored*)	20 x 20	250	25
SiliaPlate Prep (*Glass Preparative*)				
TLG-R10011B-341	SiliaPlate Prep	20 x 20	1000	25
TLG-R10011B-350	SiliaPlate Prep	20 x 20	2000	12
TLG-R10011B-353	SiliaPlate Prep	20 x 20	2000	25

UltraPure SiliaPlate Functionalized TLC

SiliCycle P/N	Product Name	Plate Size (cm)	Thickness (µm)	#/box
SiliaPlate C18				
TLG-R30411B-213	SiliaPlate C18	10 x 10	150	25
TLG-R30411B-303	SiliaPlate C18	20 x 20	150	25
SiliaPlate C18 Prep				
TLG-R30411B-341	SiliaPlate C18 Prep	20 x 20	1000	25
SiliaPlate C8				
TLG-R31030B-203	SiliaPlate C8	10 x 10	150	25
TLG-R31030B-303	SiliaPlate C8	20 x 20	150	25
SiliaPlate C2				
TLG-R32611B-203	SiliaPlate C2	10 x 10	150	25
TLG-R32611B-303	SiliaPlate C2	20 x 20	150	25
SiliaPlate NH_2 (*Amine*)				
TLG-R52011B-203	SiliaPlate NH_2	10 x 10	150	25
TLG-R52011B-303	SiliaPlate NH_2	20 x 20	150	25
SiliaPlate CN (*Cyano*)				
TLG-R38011B-203	SiliaPlate CN	10 x 10	150	25
TLG-R38011B-303	SiliaPlate CN	20 x 20	150	25
SiliaPlate Diol				
TLG-R35011B-203	SiliaPlate Diol	10 x 10	150	25
TLG-R35011B-303	SiliaPlate Diol	20 x 20	150	25
SiliaPlate Ag (*Silver Nitrate 10% impregnated*)				
TLG-R23511B-423	SiliaPlate Ag	10 x 10	150	25
TLG-R23511B-303	SiliaPlate Ag	20 x 20	150	25

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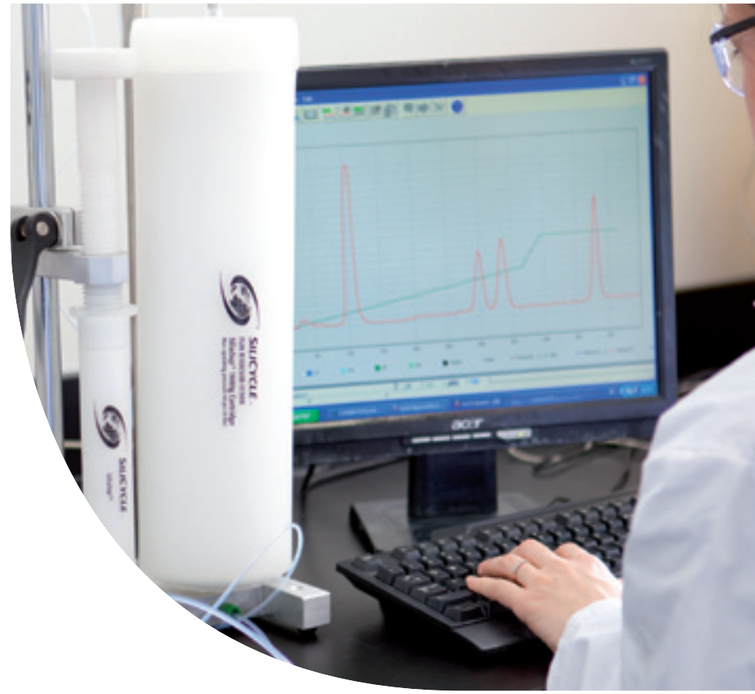
SiliaSep™
Flash Cartridges



SILICYCLE inc.
UltraPure SILICA GELS

SiliCycle Silia*Sep*™ Flash Cartridges

SiliCycle is the partner of choice for your flash cartridge needs.

- The use of pre-packed flash cartridges improves purification efficiency by offering superior reproducibility and productivity compared to conventional manual flash chromatography.
- Pre-packed cartridges offer to chromatographers:
  - More tightly packed silica bed
  - Homogeneous packing
  - Better separation

Silia*Sep* Flash Cartridges

Today, various manufacturers offer pre-packed flash cartridges, but performance and quality varies. SiliCycle supplies pre-packed columns offered under the brand name of Silia*Sep* Flash Cartridges.

SiliaSep offers superior performances over competitive cartridges. They are available in two silica gel grades (*40-63 & 15-40 microns*) and with various functionalities (*reversed & normal phases, ion exchangers and metal scavengers*) to meet any purification demand.

Important Separation Parameters

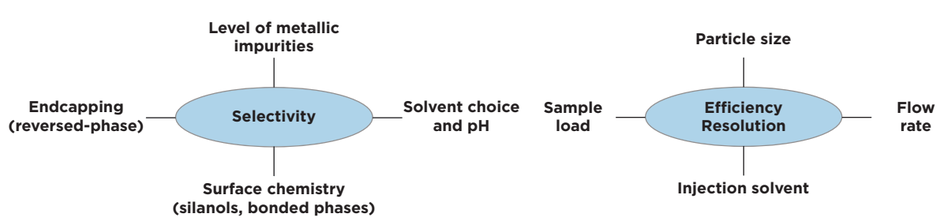
Selectivity

Selectivity refers to the ability to retain or release certain types of compounds.

Efficiency & Resolution

The performance of flash cartridges can be measured by different parameters including plate count (N) and symmetry (SI). The higher (N), the better the separation.

Influencing Factors



Silia*Sep* Features & Benefits

*Speed, Reliability, & Selectivity, with SiliCycle's Silia*Sep* flash cartridges you will benefit from the same quality that all our products are known for. We have the best silica gel available on the market with no fines. Silia*Sep* offers:

*ALWAYS ASSURED WITH SILIASEP FLASH CARTRIDGES

Features & Benefits of Silia*Sep*	
Features	**Benefits**
Highest silica gel quality with no fines	No product contamination Homogeneous packing, no channelling (*no peak tailing*) High loading capacity (*high surface area*) Direct transfer from TLC to flash chromatography (same silica)
Innovative packing technology	Consistent packing for reproducible high plate count (*N*) Superior performance & separation Higher resolution with improved band definition (*no tailing*) Greater compound purity & higher recovery
Versatility	Wide choice of cartridge sizes from 4 grams up to 1.5 kg Coming soon: 2.5 kg cartridge Purification scale-up from milligram to hundreds of grams! Variety of sorbents to address any separation need
Reproducibility, reliability & safety	Leak-free guaranteed by unique one-piece cartridge design Reproducible performance from lot-to-lot (*stringent quality control*) Excellent durability to withstand high pressures Universal luer fittings for compatibility with any flash systems
Cost effective	Excellent ratio performance vs price Readily available from stock inventory for many volumes

Silia*Sep* Flash Cartridge Design

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SiliaSep Portfolio Overview

SiliaSep Sorbents for Non-Ionic Compounds

NEED ANOTHER SORBENT? CONTACT US!

SiliaSep Sorbents for Non-Ionic Compounds Characteristics

Sorbent	Structure	Characteristics	Typical Applications	Storage Conditions* (*Never dry out*)
Silica R10030B	Si–OH	Part. size: 40 - 63 µm	Most popular sorbent for day-to-day use for the purification of non-ionic polar organic compounds	Single use recommended.
Silica HP R10017B	Si–OH	Part. size: 15 - 40 µm	High performance sorbent for difficult separations (*isomers*). Higher loading capacity. Faster flow rate. Less solvent used.	Single use recommended.
Amine R52030B	Si–NH_2	Part. size: 40 - 63 µm Endcapping: Yes %N: ≥ 1.68%	Good alternative for normal phase purification of compounds with basic properties, which would normally have to be purified by reversed phase. Note: imine formation can be seen with the purification of aldehydes and ketones.	Flush the cartridge 3x with: - 80% acetonitrile in water
Diol R35030B	Si–O–OH, OH	Part. size: 40 - 63 µm Endcapping: Yes %C: ≥ 6.98%	Good alternative for difficult separation of low to medium polarity samples. Offers a better retention time compared to normal phase. Note: nucleophilic addition reactions can be seen with the purification of ketones and amines (*1° and 2°*).	Flush the cartridge 3x with: - 80% acetonitrile in water
Cyano R38030B	Si–N≡	Part. size: 40 - 63 µm Endcapping: Yes %N: ≥ 1.93%	Versatile sorbent that can be used either as normal or reversed phase. Indicated for products with intermediate to extreme polarity. The slightly hydrophobic nature of the cyano group offers alternative selectivities.	Flush the cartridge 3x with: - 80% methanol in water or - 80% acetonitrile in water
C18 R33230B	Si–C_{18}	Part. size: 40 - 63 µm Endcapping: Yes %C: 17%	Indicated for the purification of medium to high polarity compounds, SiliaSep C18 are packed with the new generation of SiliaBond C18 monomeric reversed-phase. They provide reproducible purification without the complexity and cost of preparative HPLC.	Flush the cartridge 3x with: - 80% methanol in water or - 80% acetonitrile in water

Decreasing polarity

SiliaSep Sorbents for Ionic Compounds

SiliaSep Sorbents for Ionic Compounds Characteristics

Sorbent	Structure	Characteristics	Typical Applications (*single use recommanded*)
SCX-2 *nec* (*propylsulfonic acid*) R51230B	Si–S(=O)(=O)–OH	Part. size: 40 - 63 µm Endcapping: No %S: ≥ 2.35% meq ≥ 0.63 mmol/g	Packed with the strong cation exchange silica SCX-2, they can be used to fully retain basic compounds for clean-up or to isolate them by a catch and release process.
SAX *nec* (*TMA Chloride*) R66530B	Si–N^+–, Cl^-	Part. size: 40 - 63 µm Endcapping: No %N: ≥ 1.42% meq ≥ 0.8 mmol/g	Packed with the strong anionic exchange silica SAX, they can be used to fully retain acidic compounds for clean-up or to isolate them by a catch and release process.
SAX-2 *nec* R66430B	Si–N^+–, CH_3COO^-	Part. size: 40 - 63 µm Endcapping: No %N: ≥ 1.00% meq ≥ 0.5 mmol/g	Strong anion exchange sorbent with a low selectivity acetate counter ion retains more favorably acidic compounds with pKa's < 5, such as carboxylic acid.

SiliaSep Cartridge Types Overview

SiliaSep Cartridges Characteristics / **SiliaSep Cartridges Characteristics**

Characteristics	Units	SiliaSep 4 g	SiliaSep 12 g	SiliaSep 25 g	SiliaSep 40 g	SiliaSep 80 g	SiliaSep 120 g	SiliaSep 220 g	SiliaSep 330 g	SiliaSep XL 800 g	SiliaSep XL 1600 g	Units	Characteristics
Cartridge Code	-	ISO04	ISO12	ISO25	ISO40	ISO80	IS120	IS220	IS330	IS750	I1500	-	Cartridge Code
Silica weight	g	Bare: 4 g Bonded: ≥ 5 g	Bare: 12 g Bonded: ≥ 15 g	Bare: 25 g Bonded: ≥ 30 g	Bare: 40 g Bonded: ≥ 40 g	Bare: 80 g Bonded: ≥ 90 g	Bare: 120 g Bonded: ≥ 130 g	Bare: 220 g Bonded: ≥ 230 g	Bare: 330 g Bonded: ≥ 360 g	Bare: 800 g Bonded: ≥ 870 g	Bare: 1,600 g Bonded: ≥ 1,700 g	g	Silica weight
#/box	unit	Bare: 20 Bonded: 2	Bare: 20 Bonded: 1	Bare: 15 Bonded: 1	Bare: 15 Bonded: 1	Bare: 12 Bonded: 1	Bare: 10 Bonded: 1	Bare: 4 Bonded: 1	Bare: 4 Bonded: 1	Bare: 2 Bonded: 1	Bare: 2 Bonded: 1	unit	#/box
Dimension (OD x Length)	mm	16 x 98	25 x 117	25 x 165	32 x 169	36 x 237	42 x 256	66 x 195	66 x 268	89 x 382	120 x 429	mm	Dimension (OD x Length)
Column volume	mL	4.9	17	31	47	123	190	306	441	1,500	2,900	mL	Column volume
Recom. flow rate	mL/min	15 - 25	20 - 40	20 - 45	25 - 50	40 - 80	60 - 120	60 - 180	80 - 180	200 - 300	300 - 450	mL/min	Recom. flow rate
Loading capacity	g	0.005 - 0.4	0.015 - 1.2	0.025 - 2.5	0.05 - 4.0	0.10 - 8.0	0.15 - 12.0	0.25 - 22.0	0.50 - 33.0	0.80 - 80.0	1.60 - 160.0	g	Loading capacity

How to create product number:
Product Number => FLH - [Chemistry Code] - [Cartridge Code] Ex. cartridge 4 g with silica gel => FLH-R10030B-ISO04

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Silia*Sep* Ordering Information

Silia*Sep* Cartridges Ordering Information					
Silia*Sep* Type	Silia*Sep* 4 g	Silia*Sep* 12 g	Silia*Sep* 25 g	Silia*Sep* 40 g	Silia*Sep* 80 g
Quantity per box	20 / box	20 / box	15 / box	15 / box	12 / box
Silia*Sep* Silica	FLH-R10030B-ISO04	FLH-R10030B-ISO12	FLH-R10030B-ISO25	FLH-R10030B-ISO40	FLH-R10030B-ISO80
Silia*Sep* Silica HP	FLH-R10017B-ISO04	FLH-R10017B-ISO12	FLH-R10017B-ISO25	FLH-R10017B-ISO40	FLH-R10017B-ISO80
Other Silia*Sep* Phases					
Quantity per box	2 / box	1 / box	1 / box	1 / box	1 / box
Silia*Sep* Amine	FLH-R52030B-ISO04	FLH-R52030B-ISO12	FLH-R52030B-ISO25	FLH-R52030B-ISO40	FLH-R52030B-ISO80
Silia*Sep* Diol	FLH-R35030B-ISO04	FLH-R35030B-ISO12	FLH-R35030B-ISO25	FLH-R35030B-ISO40	FLH-R35030B-ISO80
Silia*Sep* Cyano	FLH-R38030B-ISO04	FLH-R38030B-ISO12	FLH-R38030B-ISO25	FLH-R38030B-ISO40	FLH-R38030B-ISO80
Silia*Sep* C18 (17%)	FLH-R33230B-ISO04	FLH-R33230B-ISO12	FLH-R33230B-ISO25	FLH-R33230B-ISO40	FLH-R33230B-ISO80
Silia*Sep* SCX-2	FLH-R51230B-ISO04	FLH-R51230B-ISO12	FLH-R51230B-ISO25	FLH-R51230B-ISO40	FLH-R51230B-ISO80
Silia*Sep* SAX *nec*	FLH-R66530B-ISO04	FLH-R66530B-ISO12	FLH-R66530B-ISO25	FLH-R66530B-ISO25	FLH-R66530B-ISO80
Silia*Sep* SAX-2 *nec*	FLH-R66430B-ISO04	FLH-R66430B-ISO12	FLH-R66430B-ISO25	FLH-R66430B-ISO25	FLH-R66430B-ISO80

Note: for Metal Scavengers Cartridges, see page 113 for more information.

Silia*Sep* Cartridges Ordering Information					
Silia*Sep* Type	Silia*Sep* 120 g	Silia*Sep* 220 g	Silia*Sep* 330 g	Silia*Sep* XL 800 g	Silia*Sep* XL 1600 g
Quantity per box	10 / box	4 / box	4 / box	2 / box	2 / box
Silia*Sep* Silica	FLH-R10030B-IS120	FLH-R10030B-IS220	FLH-R10030B-IS330	FLH-R10030B-IS750	FLH-R10030B-I1500
Silia*Sep* Silica HP	FLH-R10017B-IS120	FLH-R10017B-IS220 NEW PRODUCT	FLH-R10017B-IS330 NEW PRODUCT	-	-
Other Silia*Sep* Phases					
Quantity per box	2 / box	1 / box	1 / box	1 / box	1 / box
Silia*Sep* Amine	FLH-R52030B-IS120	FLH-R52030B-IS220	FLH-R52030B-IS330	FLH-R52030B-IS750	FLH-R52030B-I1500
Silia*Sep* Diol	FLH-R35030B-IS120	FLH-R35030B-IS220	FLH-R35030B-IS330	FLH-R35030B-IS750	FLH-R35030B-I1500
Silia*Sep* Cyano	FLH-R38030B-IS120	FLH-R38030B-IS220	FLH-R38030B-IS330	FLH-R38030B-IS750	FLH-R38030B-I1500
Silia*Sep* C18 (17%)	FLH-R33230B-IS120	FLH-R33230B-IS220	FLH-R33230B-IS330	FLH-R33230B-IS750	FLH-R33230B-I1500
Silia*Sep* SCX-2	FLH-R51230B-IS120	FLH-R51230B-IS220	FLH-R51230B-IS330	FLH-R51230B-IS750	FLH-R51230B-I1500
Silia*Sep* SAX *nec*	FLH-R66530B-IS120	FLH-R66530B-IS220	FLH-R66530B-IS330	FLH-R66530B-IS750	FLH-R66530B-I1500
Silia*Sep* SAX-2 *nec*	FLH-R66430B-IS120	FLH-R66430B-IS220	FLH-R66430B-IS330	FLH-R66430B-IS750	FLH-R66430B-I1500

Silia*Sep* Solid-Load Cartridges

Usually, the use of solid-load technique (*also called dry-load*) will improve chromatography resolution. Silia*Sep* Solid-Load luer-lok cartridges are designed to be used with Silia*Sep* flash cartridges for sample loading.

Either Silia*Sep* pre-packed (*for liquid injection, various choices of media available*) or empty solid-load (*for silica-sample slurry*) are available to better suit your needs.

XL Solid-Load Flanges

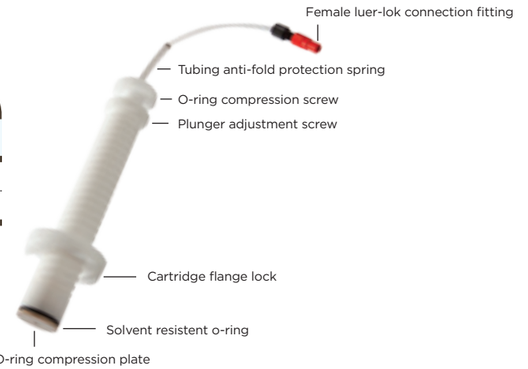
SiliaSep Solid-Load Cartridges (*Luer-Lok*)				
Product Number	Sorbent	Weight / Volume	Description	Qty per box
SPL-R10030B-10U	Silica (40 - 63 µm)	2 g / 10 mL	Silia*Sep* Silica Pre-packed Solid-Load Cartridge, 2 g, 10 mL	20
SPL-R10030B-10X	Silica (40 - 63 µm)	5 g / 10 mL	Silia*Sep* Silica Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R10030B-60Y	Silica (40 - 63 µm)	10 g / 60 mL	Silia*Sep* Silica Pre-packed Solid-Load Cartridge, 10 g, 60 mL	16
SPL-R10030B-60K	Silica (40 - 63 µm)	25 g / 60 mL	Silia*Sep* Silica Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-R10030B-065	Silica (40 - 63 µm)	65 g / 150 mL	Silia*Sep* Silica Pre-packed XL Solid-Load Cartridge, 65 g, 150 mL	12
SPL-R10030B-270	Silica (40 - 63 µm)	270 g / 700 mL	Silia*Sep* Silica Pre-packed XL Solid-Load Cartridge, 270 g, 700 mL	6
SPL-R52030B-10X	Amine	5 g / 10 mL	Silia*Sep* Amine Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R52030B-60K	Amine	25 g / 60 mL	Silia*Sep* Amine Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-R35030B-10X	Diol	5 g / 10 mL	Silia*Sep* Diol Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R35030B-60K	Diol	25 g / 60 mL	Silia*Sep* Diol Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-R38030B-10X	Cyano	5 g / 10 mL	Silia*Sep* Cyano Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R38030B-60K	Cyano	25 g / 60 mL	Silia*Sep* Cyano Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-R33230B-10X	C18 (17%)	5 g / 10 mL	Silia*Sep* C18 (17%) Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R33230B-60K	C18 (17%)	25 g / 60 mL	Silia*Sep* C18 (17%) Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-0009-010	Empty	- / 10 mL	Silia*Sep* Empty Solid-Load Cartridge, 10 mL	100
SPL-0012-060	Empty	- / 60 mL	Silia*Sep* Empty Solid-Load Cartridge, 60 mL	100
AUT-0090-150	Empty	- / 150 mL	Silia*Sep* Empty Solid-Load Cartridge, 150 mL	12
AUT-0090-700	Empty	- / 700 mL	Silia*Sep* Empty Solid-Load Cartridge, 700 mL	6

Note: for optimal purification performance, solvent removal under vacuum is highly recommended.

Silia*Sep* Plungers

Silia*Sep* Plungers*	
Product Number	Description
AUT-0060-010	Plunger for 10 mL solid-load cartridge (16 mm)
AUT-0060-060	Plunger for 60 mL solid-load cartridge (27 mm)

*Ask for Silia*Sep* Plungers Operating Instructions Guide



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Silia*Sep* Accessories for System Conversion

SiliCycle's Silia*Sep* Flash Cartridges are designed to be a universal closed-top flash columns (*luer-lok connection*). Take advantage of Silia*Sep*'s benefits and enhanced performance by converting your system with an inexpensive adaptor today.

SiliCycle manufactures Silia*Sep* cartridges that will fit perfectly (*no adaptor required*) on the following systems:

- Teledyne Isco™ CombiFlash® (XL) systems
- Varian® (Analogix®) IntelliFlash® & SimpliFlash®
- Interchim PuriFlash™ 430 evo & Spot II (Armen®)
- Büchi Sepacore™
- Grace Revelaris™

NO ADAPTOR REQUIRED

Silia*Sep* on Biotage™ Instruments

With Biotage Isolera™ Systems

With Isolera systems, you can directly use Silia*Sep* without adapters.

Even if Silia*Sep* does not have luer-loks at both ends like the SNAP cartridge, you can connect it to your current solvent line (*if you prefer to have both ends with luer fittings, contact us for optional adapters*).

With Biotage Horizon™, SP1 & SP4 Systems

Note: if you are already using SNAP with your system, no modification is required. See previous point.

With these Biotage's systems, you have two different options:

Option A: toggle between Biotage and SiliCycle columns
Option B: use only Silia*Sep* cartridges. No compression module will be necessary from now on.

Option A: Using Adaptors

If you are using the Biotage compression modules (*metal cylinders*), simply link your existing solvent line connection to the Silia*Sep* adaptors.

To do so, attach the Biotage Adaptor Kit (PN: *KAD-1006*) to the existing Swagelock™ stainless steel connectors, which will allow them to connect to SiliCycle Silia*Sep* columns. This is only if you want to toggle between Biotage and SiliCycle columns.

Biotage Adapter Kit (*2 pieces*)
PN: KAD-1006

Option B: Changing Solvent Lines

Changing the solvent lines from your current system to a Luer-Lok one will allow direct connection of Silia*Sep* Flash Cartridges. No compression module will be needed from now on.

To switch completely to the Silia*Sep* columns, simply unscrew the fittings that are currently installed on the Biotage systems and screw in the Silia*Sep* Solvent Line Replacement (PN: *KAD-1014*) with the luer connections we supply.

The female luer (*A*), which connects to the top of the column, goes onto the flow outlet of the instrument (*top hole on the instrument*).

The male luer (*B*) fits on top of the slip tip on the bottom of the column and connects to the flow inlet of the instrument (*coming out of the column*).

Note: Silia*Sep* Solid-Load Cartridges and Plungers are SiliCycle's alternative to Biotage's samplet (*see page 163*).

Biotage Solvent Line Replacement
PN: KAD-1014

Silia*Sep* Support Rings

Support rings will allow Silia*Sep* cartridges to sit on the Biotage SP1 & SP4 instruments.

Silia*Sep* Support Rings

Product Number	Description	Qty per box
AUT-0068-004	Support ring-4 (16 mm)	5
AUT-0068-010	Support ring-12, 25 (25 mm)	5
AUT-0068-040	Support ring-40 (32 mm)	5
AUT-0068-080	Support ring-80 (36 mm)	5
AUT-0068-120	Support ring-120 (42 mm)	5
KAD-1008	Support ring kit	5 different sizes

Silia*Sep* on FlashMaster™ Instruments

On FlashMaster systems, cartridges are running upside down (*reversed*). To use Silia*Sep* cartridges on any FlashMaster instrument, simply connect the Silia*Sep* cartridge directly on the instrument using the FlashMaster Adapter Kit (*PN: KAD-1016*) without the plunger. One piece holds the cartridge in place while the other connects to the solvent line.

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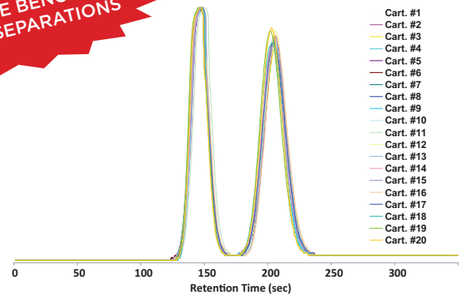
Silia*Sep* Reproducibility

SILIASEP, THE BENCHMARK FOR ALL SEPARATIONS

Silia*Sep* (*HP*) Flash Cartridges offer incredible performance over competitive products due to the higher silica gel quality and innovative packing technology. Both cartridge series allow superior results and can be considered the products of choice for all purification needs.



Silia*Sep* 40 g

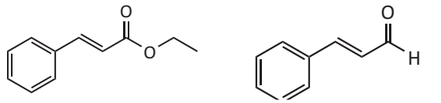
Silia*Sep* Superior Performance

SiliCycle evaluated the performance of Silia*Sep* cartridges against some established players in chromatography and purification. In this study, cartridge performances were evaluated by the determination of different parameters including plate count (N), reduced plate height (h), symmetry index ($SI_{10\%}$) and resolution factor (R). In all cases, Silia*Sep* allows excellent performance over the competition.

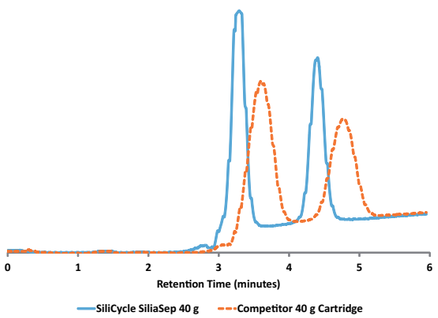
Better Separations with Silia*Sep* - Higher Plate Count (N)

Silia*Sep* 40 g versus another 40 g cartridge (*irregular silica 40-63 µm*)

As shown below, although both cartridges have a comparable symmetry index, the Silia*Sep* 40 g gives a better separation due to a higher plate count and a smaller plate height compared to the cartridge from the competition.



a) Ethyl cinnamate b) trans-cinnamaldehyde



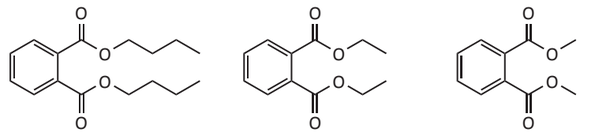
Separation test description

Mobile phase: **Solvent A**: EtOAc
Solvent B: Hexane
Gradient: 0 to 75% of solvent B in 8 minutes
Flow rate: 40 mL / min
Injection volume: 5 mL
Wavelength: 254 nm

Observed Chromatographic Parameters

Cartridge	N	h	$SI_{10\%}$	R
Silia*Sep* 40 g	2,157	1.14	0.98	3.06
Competitor 40 g	830	2.80	1.11	1.57

High Resolution with Silia*Sep* HP



a) Dibutylphthalate b) Diethylphthalate c) Dimethylphthalate

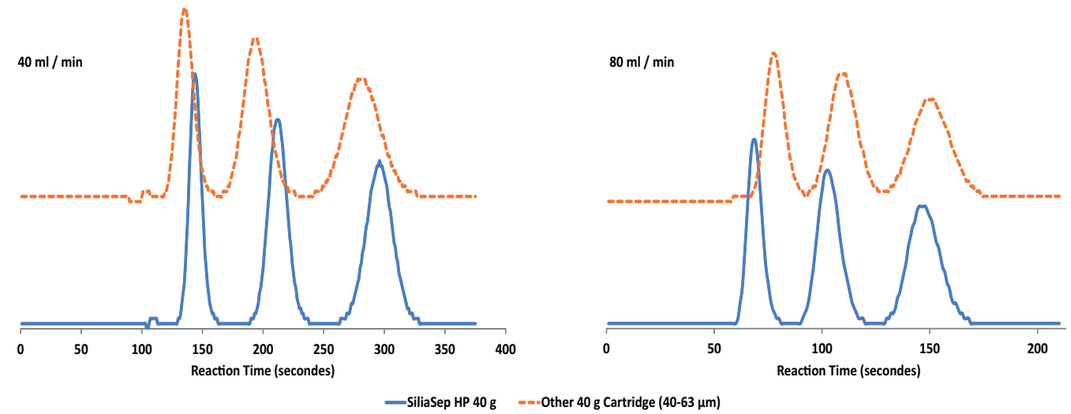
Separation test description for the two experiments shown below

Mobile phase: 20% EtOAc in Hexane
Flow rate: 40 mL / min or 80 mL / min
Injection volume: 5 mL
Wavelength: 254 nm

Silia*Sep* HP - Save Time with Faster Flow Rates

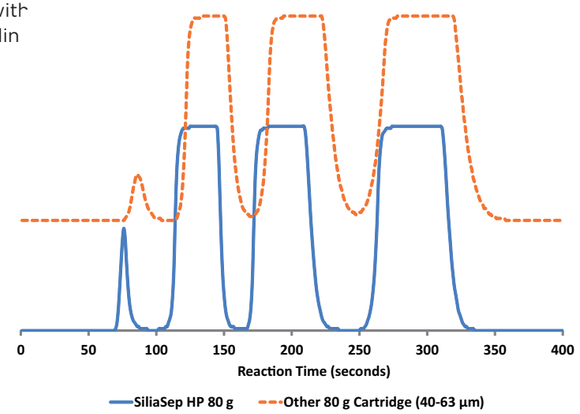
GREENER! LESS SOLVENT CONSUMED

The high resolution from Silia*Sep* HP allows the purification to run at a higher flow rate with the same high efficiency without compromising the quality of the separation.



Silia*Sep* HP - Higher Loading Capacity

The high performance of Silia*Sep* HP, associated with the higher plate count, can also yield a higher loadin capacity. As shown in the results below, Silia*Sep* HP may be loaded with over 50% more products compared to other 40-63 µm cartridges and still provide very good separation.

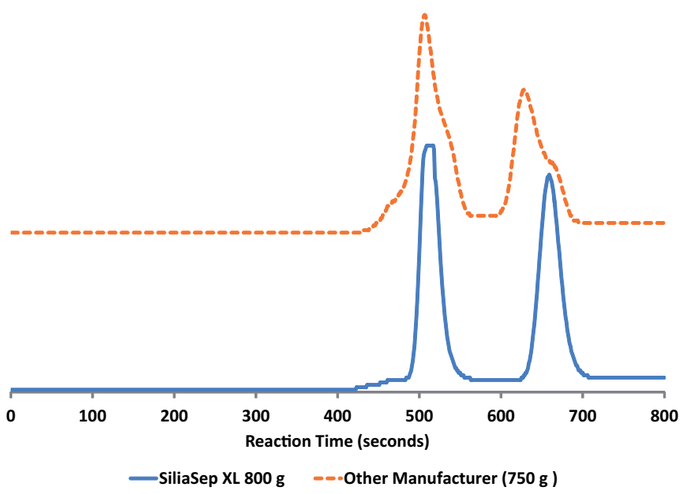


SiliaSep XL - Superior Resolution

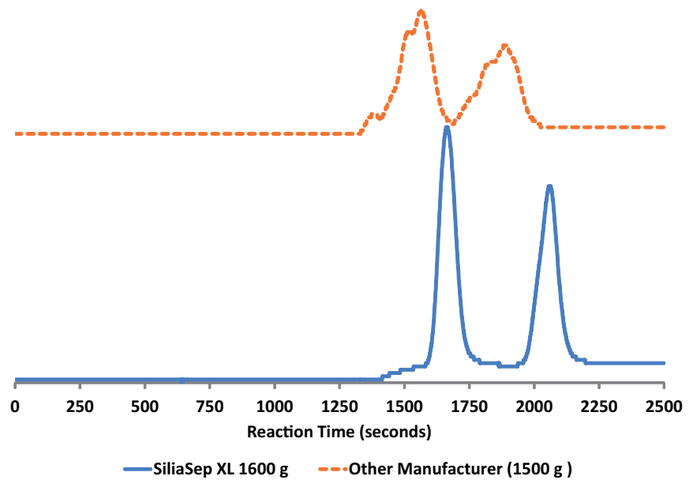
COMPARED TO A WELL-KNOWN BRAND.

SiliCycle evaluated the performance of the SiliaSep XL cartridges compared to a well-known brand. For both sizes, SiliaSep XL outperforms the competition.

SiliaSep XL 800 g



SiliaSep XL 1 600 g



SiliaSep OT (*FlashMaster™ Compatible Flash Cartridges*)

SiliaSep OT Cartridges (*rated 60 psi*)					
Silica Weight	2 g	5 g	10 g	15 g	20 g
Dimensions ID x L	15.8 x 90 mm	20.5 x 100 mm	26.8 x 154 mm	26.8 x 154 mm	26.8 x 154 mm
Volume	12 mL	25 mL	70 mL	70 mL	70 mL
Quantity per box	20 / box	20 / box	16 / box	16 / box	16 / box
SiliaSep OT Phases					
SiliaSep OT Silica	FLH-R10030B-15U	SPE-R10030B-25X	FLH-R10030B-70Y	FLH-R10030B-70i	FLH-R10030B-70Z
SiliaSep OT Amine	SPE-R52030B-12U	SPE-R52030B-20X	FLH-R52030B-70Y	FLH-R52030B-70i	FLH-R52030B-70Z
SiliaSep OT Diol	SPE-R35030B-12U	SPE-R35030B-20X	FLH-R35030B-70Y	FLH-R35030B-70i	FLH-R35030B-70Z
SiliaSep OT Cyano	SPE-R38030B-12U	SPE-R38030B-20X	FLH-R38030B-70Y	FLH-R38030B-70i	FLH-R38030B-70Z
SiliaSep OT C18 (17%)	SPE-R33230B-12U	SPE-R33230B-20X	FLH-R33230B-70Y	FLH-R33230B-70i	FLH-R33230B-70Z
SiliaSep OT SCX-2	SPE-R51230B-12U	SPE-R51230B-20X	FLH-R51230B-70Y	FLH-R51230B-70i	FLH-R51230B-70Z
SiliaSep OT SAX *nec*	SPE-R66530B-12U	SPE-R66530B-20X	FLH-R66530B-70Y	FLH-R66530B-70i	FLH-R66530B-70Z
SiliaSep OT SAX-2 *nec*	SPE-R66430B-12U	SPE-R66430B-20X	FLH-R66430B-70Y	FLH-R66430B-70i	FLH-R66430B-70Z

Note: for Metal Scavengers Cartridges, see page 113 for more information.

SiliaSep OT Cartridges (*rated 60 psi*)				
Silica Weight	25 g	50 g	70 g	100 g
Dimensions ID x L	38.2 x 170 mm	38.2 x 170 mm	38.2 x 170 mm	40.0 x 220 mm
Volume	150 mL	150 mL	150 mL	276 mL
Quantity per box	10 / box	10 / box	10 / box	12 / box
SiliaSep OT Phases				
SiliaSep OT Silica	FLH-R10030B-95K	FLH-R10030B-95M	FLH-R10030B-95N	FLH-R10030B-276F
SiliaSep OT Amine	FLH-R52030B-95K	FLH-R52030B-95M	FLH-R52030B-95N	FLH-R52030B-276F
SiliaSep OT Diol	FLH-R35030B-95K	FLH-R35030B-95M	FLH-R35030B-95N	FLH-R35030B-276F
SiliaSep OT Cyano	FLH-R38030B-95K	FLH-R38030B-95M	FLH-R38030B-95N	FLH-R38030B-276F
SiliaSep OT C18 (17%)	FLH-R33230B-95K	FLH-R33230B-95M	FLH-R33230B-95N	FLH-R33230B-276F
SiliaSep OT SCX-2	FLH-R51230B-95K	FLH-R51230B-95M	FLH-R51230B-95N	FLH-R51230B-276F
SiliaSep OT SAX *nec*	FLH-R66530B-95K	FLH-R66530B-95M	FLH-R66530B-95N	FLH-R66530B-276F
SiliaSep OT SAX-2 *nec*	FLH-R66430B-95K	FLH-R66430B-95M	FLH-R66430B-95N	FLH-R66430B-276F

SiliaSep OT are also available with bar code for automation purposes.

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SiliaSep BT (*Biotage™ "i" Compatible Flash Cartridges*)

SiliaSep BT Cartridges (*rated 100 psi*)					
Cartridge Type	12S	12M	40S	40M	40L
Dimensions ID x L	12 x 75 mm	12 x 150 mm	40 x 75 mm	40 x 150 mm	40 x 200 mm
Quantity per box	20 / box	20 / box	12 / box	12 / box	12 / box
SiliaSep BT Silica	FLH-R10030B-12iS	FLH-R10030B-12iM	FLH-R10030B-40iS	FLH-R10030B-40iM	FLH-R10030B-40iL
Other SiliaSep BT Phases					
Quantity per box	2 / box	2 / box	1 / box	1 / box	1 / box
SiliaSep BT Amine	FLH-R52030B-12iS	FLH-R52030B-12iM	FLH-R52030B-40iS	FLH-R52030B-40iM	FLH-R52030B-40iL
SiliaSep BT Diol	FLH-R35030B-12iS	FLH-R35030B-12iM	FLH-R35030B-40iS	FLH-R35030B-40iM	FLH-R35030B-40iL
SiliaSep BT Cyano	FLH-R38030B-12iS	FLH-R38030B-12iM	FLH-R38030B-40iS	FLH-R38030B-40iM	FLH-R38030B-40iL
SiliaSep BT C18 (17%)	FLH-R33230B-12iS	FLH-R33230B-12iM	FLH-R33230B-40iS	FLH-R33230B-40iM	FLH-R33230B-40iL
SiliaSep BT SCX-2	FLH-R51230B-12iS	FLH-R51230B-12iM	FLH-R51230B-40iS	FLH-R51230B-40iM	FLH-R51230B-40iL
SiliaSep BT SAX *nec*	FLH-R66530B-12iS	FLH-R66530B-12iM	FLH-R66530B-40iS	FLH-R66530B-40iM	FLH-R66530B-40iL
SiliaSep BT SAX-2 *nec*	FLH-R66430B-12iS	FLH-R66430B-12iM	FLH-R66430B-40iS	FLH-R66430B-40iM	FLH-R66430B-40iL

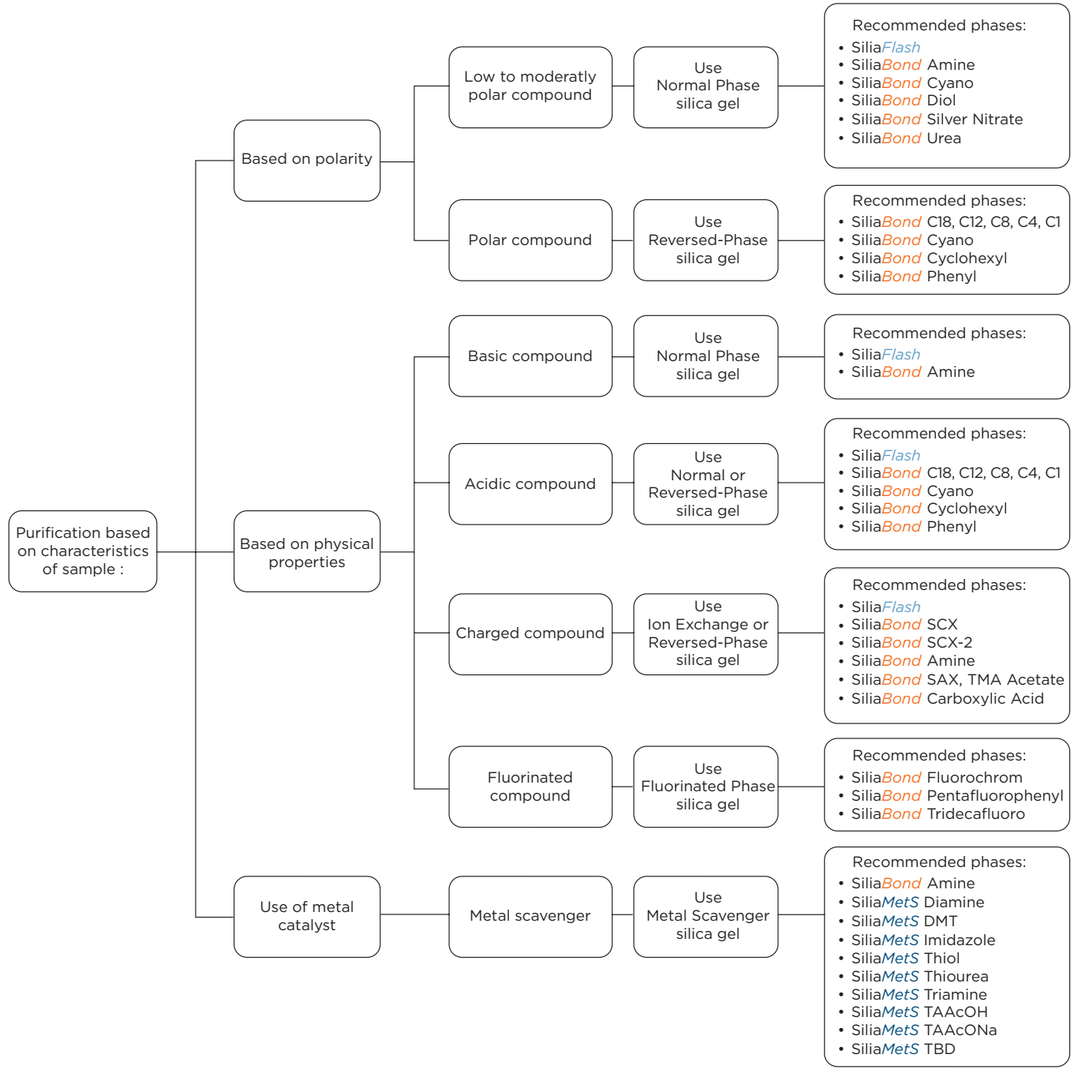
SiliaSep BT Cartridges (*rated 100 psi*)				
Cartridge Type	65	75S	75M	75L
Dimensions ID x L	65 x 200 mm	75 x 90 mm	75 x 170 mm	75 x 350 mm
Quantity per box	6 / box	2 / box*	2 / box*	2 / box*
SiliaSep BT Silica	FLH-R10030B-65i	FLH-R10030B-75iS	FLH-R10030B-75iM	FLH-R10030B-75iL
Other SiliaSep BT Phases				
Quantity per box	1 / box	1 / box	1 / box	1 / box
SiliaSep BT Amine	FLH-R52030B-65i	FLH-R52030B-75iS	FLH-R52030B-75iM	FLH-R52030B-75iL
SiliaSep BT Diol	FLH-R35030B-65i	FLH-R35030B-75iS	FLH-R35030B-75iM	FLH-R35030B-75iL
SiliaSep BT Cyano	FLH-R38030B-65i	FLH-R38030B-75iS	FLH-R38030B-75iM	FLH-R38030B-75iL
SiliaSep BT C18 (17%)	FLH-R33230B-65i	FLH-R33230B-75iS	FLH-R33230B-75iM	FLH-R33230B-75iL
SiliaSep BT SCX-2	FLH-R51230B-65i	FLH-R51230B-75iS	FLH-R51230B-75iM	FLH-R51230B-75iL
SiliaSep BT SAX *nec*	FLH-R66530B-65i	FLH-R66530B-75iS	FLH-R66530B-75iM	FLH-R66530B-75iL
SiliaSep BT SAX-2 *nec*	FLH-R66430B-65i	FLH-R66430B-75iS	FLH-R66430B-75iM	FLH-R66430B-75iL

*Box of 10 also available.

SiliaSep BT Silica Characteristics				
Product Number	Silica Weight (g)	Column Volume (mL)	Recommended Flow Rate (mL/min)	Loading Capacity (g)
FLH-R10030B-12iM		6	3 - 12	0.005 - 0.45
FLH-R10030B-12iM	9	12	3 - 12	0.01 - 0.90
FLH-R10030B-40iS	45	60	25 - 50	0.05 - 4.5
FLH-R10030B-40iM	90	120	25 - 50	0.10 - 9.0
FLH-R10030B-40iL	135	160	25 - 50	0.15 - 13.5
FLH-R10030B-65i	350	350	65 - 85	0.4 - 35
FLH-R10030B-75iS	200	300	100 - 250	0.2 - 20
FLH-R10030B-75iM	400	500	100 - 250	0.4 - 40
FLH-R10030B-75iL	800	1,000	100 - 250	0.8 - 80

SiliaSep Sorbent Selection Chart

SiliCycle offers a wide range of SiliaSep sorbents to cover many kinds of purification. The following chart is designed to serve as a guide for the selection of the appropriated sorbent based on the characteristics of the sample to be purified.



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Silia*Sep* Loading Chart

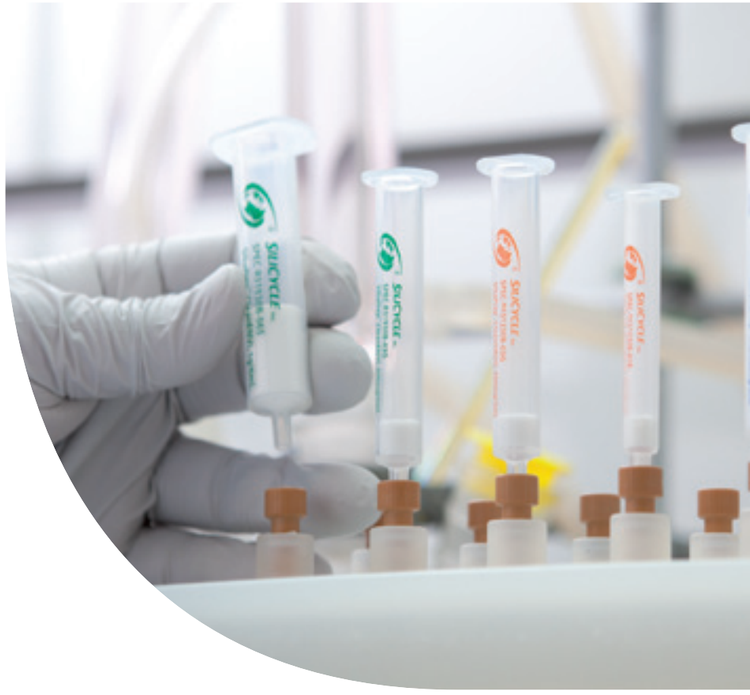
Silia*Sep* Cartridge Loading Chart

Dimensions OD x L (mm x mm)	SiliaSep Format	ΔCV = 0.1-0.6 Load (g)	ΔCV = 0.7-1.2 Load (g)	ΔCV = 1.3-1.8 Load (g)	ΔCV = 1.9-2.4 Load (g)	ΔCV = 2.5-3.1 Load (g)	ΔCV = 3.2-3.8 Load (g)	ΔCV = 3.9-4.5 Load (g)	ΔCV = 4.6-5.2 Load (g)	ΔCV = 5.3-6.0 Load (g)	ΔCV = > 6 Load (g)
16 x 98	4 g	0.040	0.080	0.120	0.160	0.200	0.240	0.280	0.320	0.360	0.400
16 x 98	4 g (HP)	0.052	0.104	0.156	0.208	0.260	0.312	0.364	0.416	0.468	0.520
25 x 117	12 g	0.120	0.240	0.360	0.480	0.600	0.720	0.840	0.960	1.080	1.200
25 x 117	12 g (HP)	0.156	0.312	0.468	0.624	0.780	0.936	1.092	1.248	1.404	1.560
25 x 165	25 g	0.250	0.500	0.750	1.000	1.250	1.500	1.750	2.000	2.250	2.500
25 x 165	25 g (HP)	0.325	0.650	0.975	1.300	1.625	1.950	2.275	2.600	2.925	3.250
32 x 169	40 g	0.400	0.800	1.200	1.600	2.000	2.400	2.800	3.200	3.600	4.000
32 x 169	40 g (HP)	0.520	1.040	1.560	2.080	2.600	3.120	3.640	4.160	4.680	5.200
36 x 237	80 g	0.800	1.600	2.400	3.200	4.000	4.800	5.600	6.400	7.200	8.000
36 x 237	80 g (HP)	1.040	2.080	3.120	4.160	5.200	6.240	7.280	8.320	9.360	10.400
42 x 256	120 g	1.200	2.400	3.600	4.800	6.000	7.200	8.400	9.600	10.800	12.000
42 x 256	120 g (HP)	1.560	3.120	4.680	6.240	7.800	9.360	10.920	12.480	14.040	15.600
66 x 195	220 g	2.200	4.400	6.600	8.800	11.000	13.200	15.400	17.600	19.800	22.000
66 x 195	220 g (HP)	2.860	5.720	8.580	11.440	14.300	17.160	20.020	22.880	25.740	28.600
66 x 268	330 g	3.300	6.600	9.900	13.200	16.500	19.800	23.100	26.400	29.700	33.000
66 x 268	330 g (HP)	4.290	8.580	12.870	17.160	21.450	25.740	30.030	34.320	38.610	42.900
89 x 382	800 g	8.000	16.000	24.000	32.000	40.000	48.000	56.000	64.000	72.000	80.000
120 x 429	1 600 g	10.000	20.000	30.000	40.000	50.000	60.000	70.000	80.000	90.000	100.000

Correlation Rf vs CV

Rf	CV
0.95	1.05
0.90	1.10
0.85	1.17
0.80	1.25
0.75	1.33
0.70	1.40
0.65	1.54
0.60	1.65
0.55	1.81
0.50	2.00
0.45	2.22
0.40	2.50
0.35	2.86
0.30	3.33
0.25	4.00
0.20	5.00
0.15	6.67
0.10	10.00
0.05	20.00

Silia*Prep*™
SPE Cartridges and Well Plates



SILICYCLE inc.
UltraPure SILICA GELS

Silia*Prep*™ SPE Cartridges and Well Plates

Using SiliaPrep SPE Cartridges and Well Plates guarantees the following benefits:

- Choice of a wide variety of Silia*Bond* high-quality sorbents
- Very good separation (*tight particle size distribution and no fines*)
- High recoveries and yields
- Less time and solvent required for conditioning the sorbent
- Reproducible flow rates from lot-to-lot
- Excellent packing and storage qualities

Silia*Prep* Solid-Phase Extraction *SPE* Cartridges and Well Plates

Solid-phase extraction (*SPE*) is designed for rapid sample preparation and purification prior to chromatographic analysis. You can optimize your SPE protocols by using SiliCycle Silia*Prep* SPE Cartridges and Well Plates.

SiliCycle offers products to meet your specific purification needs. Silia*Prep* products are available in different formats including SPE cartridges and 48-, 96-, and 384-well plates, with different sorbents (*Silia*Flash *and Silia*Bond*), and in bed weights up to 20 grams (*>20 g are also available in Silia*Sep *OT formats, see page 167*). The well plates are used in high throughput combinatorial chemistry, drug discovery and screening, metabolic pharmacokinetic applications, and for automated methods such as a multiprobe approach.

By using Silia*Prep* products you will generate higher purity samples and reduce the number of false positives in your screening, giving you higher quality data. Silia*Prep* cartridges are packed with fines-free Silia*Flash* silica gel sorbents.

Sorbent Specifications

Silia*Prep* products are packed with SiliCycle's Silia*Flash* to provide superior performance for all types of applications. This is due to the narrow particle size distribution and high purity. Although the standard products included in this brochure are made of Silia*Flash* F60 (*40-63 µm, 60 Å*), custom products are available with any type of silica offered in our catalog or website (*IMPAQ, irregular and spherical in various pore and particle sizes, etc.*) and in any format on a custom order basis. Contact us for more information.

Plastic Device Specifications

Standard Silia*Prep* cartridges are made with flanged polypropylene (*PP*) tubes and 20 µm polyethylene (*PE*) frits. Other plastic materials (*Teflon®, HDPE, etc.*), frit porosity (*10 µm*), and cartridge rim's (*flangeless*) are available on a custom order basis.

Silia*Prep* Accessories

Silia*Prep* Adapters:

Enable cartridge stacking or easy SPE cartridge connection with syringe or gas lines (*for positive pressure*).

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

Silia*Prep* Vacuum Adapters:

Fast, user friendly, and economical adapters for SPE cartridges. Only a vacuum source is needed.

AUT-0043	20/40 Silia*Prep* Vacuum Adapter
AUT-0044	19/22 Silia*Prep* Vacuum Adapter
AUT-0045	14/20 Silia*Prep* Vacuum Adapter
AUT-0046	22/400 Vial-Silia*Prep* Vacuum Adapter without Vial Connector
AUT-0047	22/400 Vial-Silia*Prep* Vacuum Adapter with Vial Connector

Silia*Prep* Vacuum Manifold:

Run 12 or 24 samples simultaneously with a controlled flow rate for higher reproducibility.

AUT-0128-12	Silia*Prep* Vacuum Manifold 12 positions
AUT-0129-24	SiliaPrep Vacuum Manifold 24 positions

Silia*Prep* Empty tubes:

Empty Tubes

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

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Standard Method Development Procedure

The solid phase methodology will vary depending on the sorbent (*normal, reversed, ion exchange*). Here, we propose generic methods for each phase based on sample and sorbent properties. However, procedures can be slightly different from one sample to another.

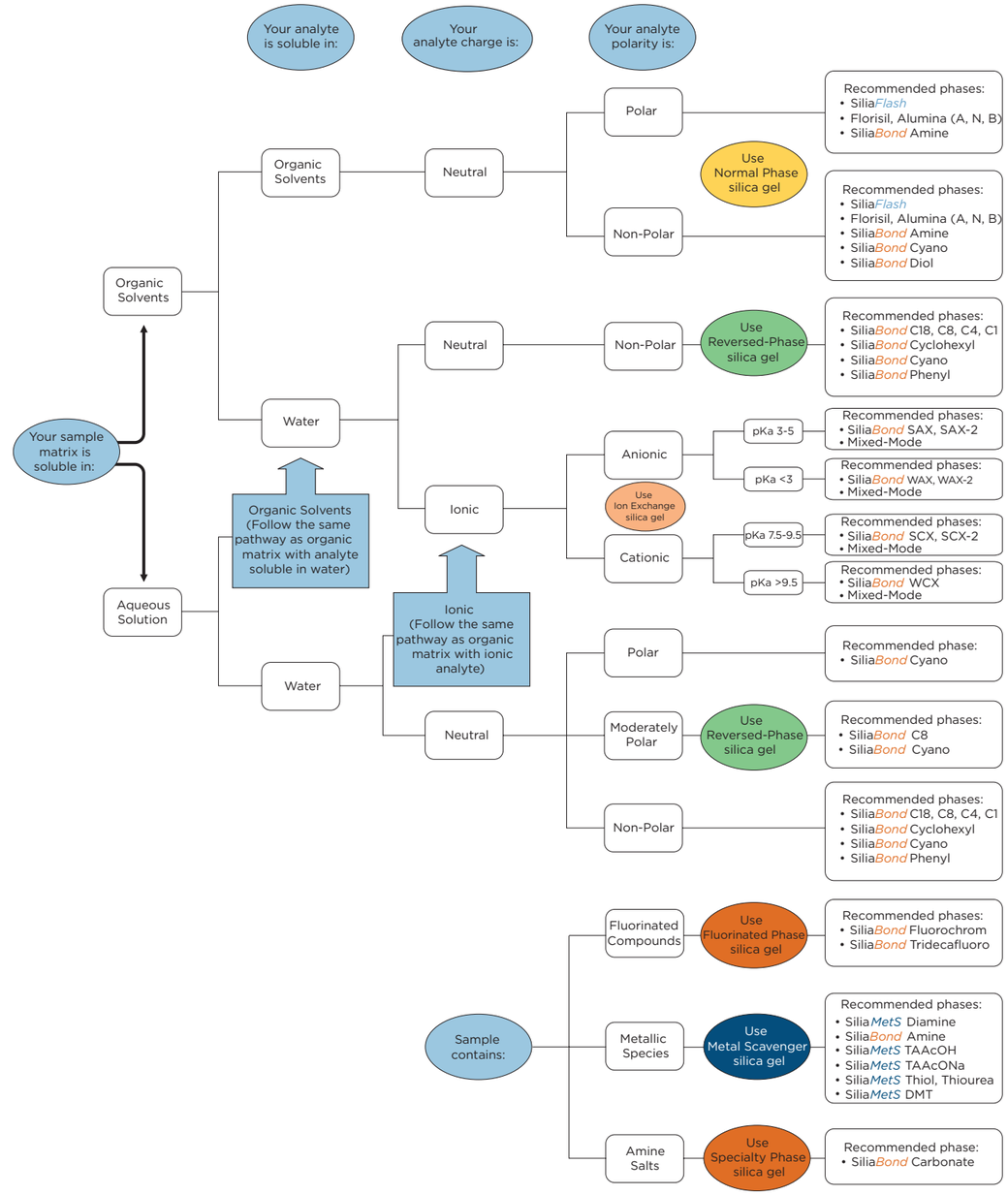
Standard Method Development Procedure

Procedure Step	Reversed-Phase	Ion Exchange Phase	Normal-Phase
Analyte properties	Non-polar, uncharged or neutralized, hydrophobic	Ionized or charged	Slightly to moderately polar, uncharged
Matrix sample properties	Solvents and aqueous (*buffer*)	Aqueous (*buffer*) and pH-ajusted solutions	Organic solvents
Conditioning step	Water-miscible organic solvents	Water-miscible organic solvents or aqueous buffered solution	Sample solvent or methanol
Sample loading	Dissolve analyte in highly polar solvents	Dissolve analyte in highly polar solvents	Dissolve analyte in low polar solvents
Washing	Aqueous or buffered solution and polar solvents	Aqueous solutions containing salts	Non-polar solvents
Elution	Polar or non-polar organic solvents	Polar solvents, may contain acids or bases	Mixture of non-polar (*5 - 50%*) an polar solvents

Suggested Elution Solvents

Reversed-Phase	Polarity	Ion Exchange Phase	Polarity	Normal Phase
THF Acetone Ethyl acetate Acetonitrile Methanol	Low ↓ High	For complete ionization, sample should be adjusted 2 pH units above or below the analyte pKa. pH can be used to neutralize analyte or sorbent. Use 2% strong acid or base in acetonitrile or methanol.	Low ↓ High	Hexane CH2Cl2 THF Acetone Acetonitrile

Product Selection Guide by Sample Properties



Product Selection Guide by Manufacturer

Product Selection Guide by Manufacturer

SiliCycle SiliaPrep	SiliCycle Part Number	Agilent Bond Elut®	Biotage Isolute®	Macherey-Nagel Chromabond®		Macron Chemicals[a] Bakerbond®	Phenomenex Strata®	Supelco Discovery® and SupelClean®	Whatman (*GE Healthcare*)	Waters Sep-Pak®
Non Polar Phases										
SiliaPrep C18 *nec* (23 %)	SPE-R30130B-xxx		C18							
SiliaPrep C18 (17 %)	SPE-R31930B-xxx	C18	C18 (EC)	C18 ec		Octadecyl (C18)	C18-E	DSC-18 and ENVI-18	ODS-5	tC18
SiliaPrep C18 *nec* (17 %)	SPE-R35530B-xxx	C18 OH		C18		Light Load Octadecyl	C18-U			
SiliaPrep C18 WPD	SPE-R33229G-xxx		MFC18	C18 ec f			C18-T			C18
SiliaPrep C8	SPE-R31030B-xxx		C8 (EC)			Octyl (C8)	C8	DSC-8 and ENVI-8	C8	C8
SiliaPrep C8 *nec*	SPE-R31130B-xxx		C8	C8						
SiliaPrep Cyclohexyl	SPE-R61530B-xxx	CH	CH (EC)	C_6H_{11}ec		Cyclohexyl (C_6H_{11})				
SiliaPrep Phenyl	SPE-R34030B-xxx	PH	PH (EC)	C_6H_5		Phenyl (C_6H_5)	Phenyl	DSC-Ph and LC-Ph		
Polar Phases										
SiliaPrep Silica	SPE-R10030B-xxx	SI	SI	SiOH		Silica gel (SiOH)	Silica (Si-1)	Silica	SIL	Silica
SiliaPrep Cyano	SPE-R38030B-xxx	Cyano	CN (EC)	CN		Cyano (CN)	Cyano (CN)[b]	DSC-CN and LC-CN		Cyanopropyl
SiliaPrep Diol *nec*	SPE-R35030B-xxx	Diol (2OH)[b]	DIOL	OH		Diol (COHCOH)		DSC-Diol and LC-Diol		Diolb
SiliaPrep Florisil	SPE-AUT-0014-xxx	Florisil	FL	Florisil		Florisil (Mg_2SiO_3)		ENVI-Florisil	FLO	Florisil
SiliaPrep Frorisil PR	SPE-AUT-0015-xxx						Florisil (FL-PR)			
SiliaPrep Alumina Acidic	SPE-AUT-0053-xxx	Alumina A (AL-A)	AL-A	Alox A				LC-Alumina-A		Alumina A
SiliaPrep Alumina Neutral	SPE-AUT-0054-xxx	Alumina N (AL-N)	AL-N	Alox N		Alumina Neutral	Alumina-N (AL-N)	LC-Alumina-N		Alumina N
SiliaPrep Alumina Basic	SPE-AUT-0055-xxx	Alumina B (AL-B)	AL-B	Alox B				LC-Alumina-B		Alumina B
Ion Exchange Phases										
SiliaPrep SAX *nec*	SPE-R66530B-xxx	SAX[b]	SAX	SB		Quaternary Amine	SAX[b]	DSC-SAX and LC-SAX	SAX	Accell Plus QMA
SiliaPrep SAX-2 *nec*	SPE-R66430B-xxx	PRS[b]	PE-AX							
SiliaPrep SCX	SPE-R60530B-xxx	SCX[b]	SCX-3[b]	SA		Aromatic Sulfonic Acid	SCX[b]	DSC-SCX and LC-SCX	SCX[b]	
SiliaPrep SCX-2	SPE-R51230B-xxx		SCX-2[b]	PSA						
SiliaPrep WAX	SPE-R52030B-xxx	NH_2[b]	NH_2	NH_2		Amino (NH_2)	NH_2/WAX[b]	DSC-NH_2 and LC-NH_2[b]	NH_2[b]	Aminopropyl
SiliaPrep Diamine (WAX-2)	SPE-R49030B-xxx	PSA[b]	Diamino	Diamino		Diamino (NH_2NH)		PSA		PSA
SiliaPrep WCX	SPE-R70030B-xxx	CBA	CBA[b]	PCA		Carboxylic Acid (COOH)	WCX[b]	DSC-WCX & LC-WCX		Accell Plus CM
Mixed-Mode and Special Phases										
SiliaPrep C8/SAX-2	SPM-R661230B-xxx	Certify II	HAX				Screen-A	DSC-MCAX		
SiliaPrep SCX-2/SAX	SPM-R802830B-xxx	AccuCAT								
SiliaPrep C8/SCX-2/SAX	SPM-R02802830B-xxx		Multimode				Screen-C[c]			
SiliaPrep CleanDRUG	SPEC-R651230B-xxx	Certify[b]	HCX[d]	Drug 1						
SiliaPrep CleanENVI	SPEC-R31930B-xxx			C18 PAH						
SiliaPrep Activated Carbon	SPE-AUT-0110-xxx	Carbon						ENVI-Carb		AC2
SiliaPrep DL AC/WAX	SP2-R11098-xxx							ENVI-Carb/NH_2		Carbon Black/Amino
SiliaPrep DL AC/Diamine	SP2-R11007-xxx							ENVI-CarbII/PSA		Carbon Black/PSA
SiliaPrep PCB	SP2-R00650030B-xxx			SA/SiOH						

[a] Mallinkrodt Baker, [b] Non-endcapped, [c] Endcapped, [d] Ion exchange phase is non-endcapped xxx = Formats

All SiliCycle products are endcapped unless noted by « nec » (*non-endcapped*)

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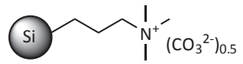
Silia*Prep* Most Popular Phases

Silia*Prep* Carbonate

Description

SiliCycle has developed innovative specialty phases in Silia*Prep* formats for specific applications, including Silia*Prep* Carbonate. It is the silica-bound equivalent of tetramethyl ammonium carbonate, and it can be used as a general base to quench a reaction, free base amines in their ammonium salt form and to scavenge acids, boronic acids and acidic phenols, including HOBt (*widely used in amide coupling reactions*).

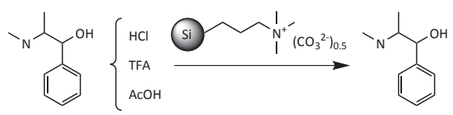
Carbonate



Amine Free Basing Purification

General Procedure

1. Silia*Prep* Carbonate (*2-4 eq. of carbonate in respect to the amine*) is conditioned with THF.
2. The amine solution in THF is loaded onto the Silia*Prep* Carbonate cartridge.
3. Free salt amine is eluted with THF under gravity.



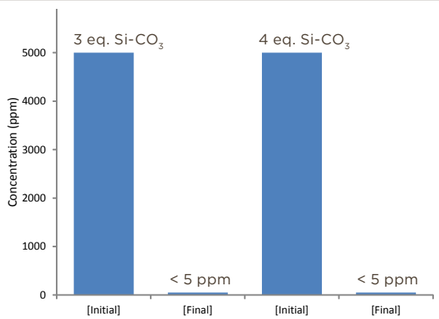
Note: other solvents can be used (methanol, ACN).
Related publication: *Org. Lett.*, *4*, **2002**, 1167

Amine Free Basing Purification Results

Amine Salts		Yield (%)[a]	Purity (%)[b]
Ephedrine•	HCl	98.7	94.4
	TFA	100	98.9
	AcOH	100	99.2

[a]Yield refers to the isolated product, [b]Purity determined by GC-FID

Scavenging HOBt with Silia*Prep* Carbonate



Silia*Prep* Carbonate SPE Formats

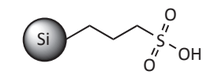
Formats	Qty/Box	Silia*Prep* Product Number
Silia*Prep* Cartridges		
1 mL/50 mg	100	SPE-R66030B-01B
1 mL/100 mg	100	SPE-R66030B-01C
3 mL/200 mg	50	SPE-R66030B-03G
3 mL/500 mg	50	SPE-R66030B-03P
6 mL/500 mg	50	SPE-R66030B-06P
6 mL/1 g	50	SPE-R66030B-06S
6 mL/2 g	50	SPE-R66030B-06U
12 mL/2 g	20	SPE-R66030B-12U
25 mL/5 g	20	SPE-R66030B-20X
Silia*Prep* Large Reservoir Volume SPE Cartridges		
10 mL/200 mg	50	SPC-R66030B-10G
10 mL/500 mg	50	SPC-R66030B-10P
Silia*Prep* 96-Well Plates		
2 mL/50 mg	1	96W-R66030B-B
2 mL/100 mg	1	96W-R66030B-C

Silia*Prep* Propylsulfonic acid and Tosic Acid

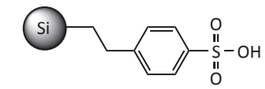
Description

SiliCycle offers Silia*Bond* Propylsulfonic Acid (*Si-SCX-2*) and Silia*Bond* Tosic Acid (*Si-SCX*). Both are considered strong cation exchangers, as 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, tests have demonstrated that they both have comparable strengths. The most common use is probably as a strong cation exchanger for amine “catch and release” purification in SPE cartridges.

Propylsulfonic Acid (*SCX-2*)



Tosic Acid (*SCX*)



Catch and Release Amine Purification

General procedure

The amine (*1 eq.*) was dissolved in methanol (*2,500 ppm*)

1. Cartridge was conditioned with methanol
2. Cartridge was loaded with the amine.
3. Cartridge was then washed with CH_3OH (*1 mL/min*)
4. Finally, the amine was released by 2 M ammonia/methanol

Catch and Release Results

Amine	# eq.	Silia*Prep* SCX-2		Silia*Prep* SCX	
		Catch (%)[a]	Release[b]	Catch (%)[a]	Release[b]
Tributylamine	2	98	90	98	97
Aniline	2	100	100	100	100
2-Aminothiazole	4	100	100	100	100
4-Nitroaniline	4	100	100	100	100

[a] Determined from the initial solution. [b] Determined by(GC-FID) analysis of isolated product

Silia*Prep* SPE Formats

Formats	Qty/Box	Silia*Prep* Propylsulfonic Acid	Silia*Prep* Tosic Acid
Silia*Prep* Cartridges			
1 mL/50 mg	100	SPE-R51230B-01B	SPE-R60530B-01B
1 mL/100 mg	100	SPE-R51230B-01C	SPE-R60530B-01C
3 mL/200 mg	50	SPE-R51230B-03G	SPE-R60530B-03G
3 mL/500 mg	50	SPE-R51230B-03P	SPE-R60530B-03P
6 mL/500 mg	50	SPE-R51230B-06P	SPE-R60530B-06P
6 mL/1 g	50	SPE-R51230B-06S	SPE-R60530B-06S
6 mL/2 g	50	SPE-R51230B-06U	SPE-R60530B-06U
12 mL/2 g	20	SPE-R51230B-12U	SPE-R60530B-12U
25 mL/5 g	20	SPE-R51230B-20X	SPE-R60530B-20X
Silia*Prep* Large Reservoir Volume SPE Cartridges			
10 mL/200 mg	50	SPC-R51230B-10G	SPC-R60530B-10G
10 mL/500 mg	50	SPC-R51230B-10P	SPC-R60530B-10P
Silia*Prep* - 96 Well Plates			
2 mL/50 mg	1	96W-R51230B-B	96W-R60530B-B
2 mL/100 mg	1	96W-R51230B-C	96W-R60530B-C

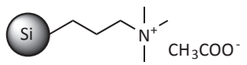
SiliaPrep TMA Acetate *nec* (*SAX-2*)

Description

Strong anion exchangers (*SAX*) have been widely used in both chromatography and ion exchange SPE to selectively bind acidic drugs and/or analytes. In particular, weakly acidic compounds can be effectively extracted as SAX sorbents retain a permanent positive charge across the pH range.

SiliCycle has developed SiliaBond TMA Acetate nec (*Si-SAX-2*), a strong anion exchange sorbent with a low-selectivity acetate counter ion already in place. Typical loading is 1.00 mmol/g, which is higher than available equivalents. This sorbent more favorably retains acidic compounds with pKas < 5, such as carboxylic acids. This property can be used in organic chemistry applications to selectively purify acidic compounds or remove acidic impurities from reaction mixtures.

TMA Acetate *nec* (*SAX-2*)



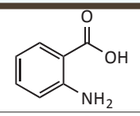
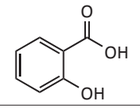
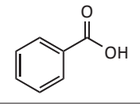
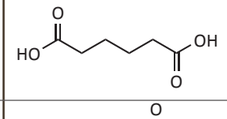
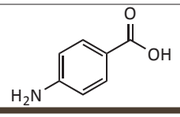
Catch and Release of Acidic Compounds

General procedure

SiliaPrep TMA Acetate nec 2 g/6 mL (*SPE-R66430B-06U*) Solutions containing 1 and 2 mmol of acidic compounds in methanol were investigated.

1. Cartridge was conditioned with methanol.
2. Cartridge was loaded with the acidic solution.
3. Cartridge was then washed with methanol to remove any impurities.
4. The acid was released using a 10 mL solution of acetic acid in methanol or acetonitrile.

Catch and Release Purification Results

pKa	Acid	Recovery (%)[a]	
		1 mmol	2 mmol
2.1		100	99
3.0		88	83
4.2		100	100
4.4		99	91
4.9		90	83

[a] Determined from the isolated product

Separation of Acids Based on pKa Results

General Procedure

A solution containing equimolar quantities of phenol, benzoic acid and salicylic acid in methanol was prepared. The solution was loaded onto a SiliaPrep TMA Acetate nec 2 g/6 mL cartridge (*SPE-R66430B-06U*). The phenol is not retained and a simple wash with methanol allows the isolation of the clean product. Elution with a 2% solution of acetic acid in methanol allowed the isolation of clean benzoic acid. Finally a 2% solution of HCl in acetonitrile was required to isolate clean salicylic acid. All yields were above 90% as indicated in table below.

Separation of Acids Based on pKa Results

Compounds	Salicylic Acid	Benzoic Acid	Phenol
pKa	3.0	4.2	10.0
Initial Amount (mg)	103	92	70
Isolated Amount (mg)	102	88	65
Recovery (%)[a]	99	96	93

[a]Recovery measured from isolated product

SiliaPrep TMA Acetate nec SPE Formats

Formats	Qty/Box	SiliaPrep Product Number
SiliaPrep Cartridges		
1 mL/50 mg	100	SPE-R66430B-01B
1 mL/100 mg	100	SPE-R66430B-01C
3 mL/200 mg	50	SPE-R66430B-03G
3 mL/500 mg	50	SPE-R66430B-03P
6 mL/500 mg	50	SPE-R66430B-06P
6 mL/1 g	50	SPE-R66430B-06S
6 mL/2 g	50	SPE-R66430B-06U
12 mL/2 g	20	SPE-R66430B-12U
25 mL/5 g	20	SPE-R66430B-20X
SiliaPrep Large Reservoir Volume SPE Cartridges		
10 mL/200 mg	50	SPC-R66430B-10G
10 mL/500 mg	50	SPC-R66430B-10P
Mini-SiliaPrep SPE Cartridges		
300 mg	50	SPS-R66430B-J
600 mg	50	SPS-R66430B-Q
900 mg	50	SPS-R66430B-R
SiliaPrep 96-Well Plates		
2 mL/50 mg	1	96W-R66430B-B
2 mL/100 mg	1	96W-R66430B-C

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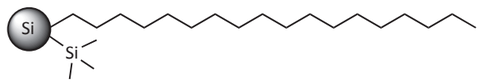
Silia*Prep* Reversed-Phases

Description

Silia*Prep* C18

SiliCycle recently developed a new and innovative C18 phase characterized by a homogeneous coverage of the silane on the surface. Consequently the endcapping step is well controlled, improving the separation and inhibiting specific interactions with silanol groups (*highly deactivated silanol phase*). This strongly hydrophobic and non-polar sorbent is used to extract acidic, neutral and basic compounds from aqueous solutions, various organic compounds from water, and drugs and metabolites from physiological fluids.

C18



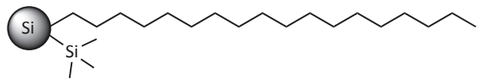
- SiliCycle Sorbent Number: R31930B
- Loading: 17 %C
- Endcapping: Yes
- Silica type: 60 Å, 500 m²/g, 40 - 63 µm

Description

Silia*Prep* C18 (*WPD*)

This strongly hydrophobic, non-polar and high-loading capacity sorbent is similar to Silia*Prep* C18 but can accommodate larger molecules and untreated matrices.

C18 (*WPD*) C18



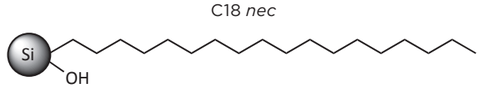
- SiliCycle Sorbent Number: R33229G
- Loading: 13 %C
- Endcapping: Yes
- Silica type: 125 Å, 300 m²/g, 37 - 55 µm

Description

Silia*Prep* C18 *nec*

This strongly hydrophobic and non-polar sorbent is similar to Silia*Prep* C18, but presents higher retention and polar selectivity for basic compounds. Unreacted surface OH's can be used for soft condition catch and release purification of glucoronides.

C18 *nec*



- SiliCycle Sorbent Number: R35530B
- Loading: 17 %C
- Endcapping: No
- Silica type: 60 Å, 500 m²/g, 40 - 63 µm

Silia*Prep* Reversed-Phases C18

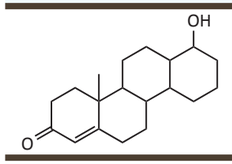
Silia*Prep* SPE Formats				
Formats	Qty/Box	Silia*Prep* C18	Silia*Prep* C18 WPD	Silia*Prep* C18 *nec*
Silia*Prep* Cartridges				
1 mL/50 mg	100	SPE-R31930B-01B	SPE-R33229G-01B	SPE-R35530B-01B
1 mL/100 mg	100	SPE-R31930B-01C	SPE-R33229G-01C	SPE-R35530B-01C
3 mL/200 mg	50	SPE-R31930B-03G	SPE-R33229G-03G	SPE-R35530B-03G
3 mL/500 mg	50	SPE-R31930B-03P	SPE-R33229G-03P	SPE-R35530B-03P
6 mL/500 mg	50	SPE-R31930B-06P	SPE-R33229G-06P	SPE-R35530B-06P
6 mL/1 g	50	SPE-R31930B-06S	SPE-R33229G-06S	SPE-R35530B-06S
6 mL/2 g	50	SPE-R31930B-06U	SPE-R33229G-06U	SPE-R35530B-06U
12 mL/2 g	20	SPE-R31930B-12U	SPE-R33229G-12U	SPE-R35530B-12U
25 mL/5 g	20	SPE-R31930B-20X	SPE-R33229G-20X	SPE-R35530B-20X
Silia*Prep* Large Reservoir Volume SPE Cartridges				
10 mL/200 mg	50	SPC-R31930B-10G	SPC-R33229G-10G	SPC-R35530B-10G
10 mL/500 mg	50	SPC-R31930B-10P	SPC-R33229G-10P	SPC-R35530B-10P
Mini-**Silia*Prep*** SPE Cartridges				
300 mg	50	SPS-R31930B-J	SPS-R33229G-J	SPS-R35530B-J
600 mg	50	SPS-R31930B-Q	SPS-R33229G-Q	SPS-R35530B-Q
900 mg	50	SPS-R31930B-R	SPS-R33229G-R	SPS-R35530B-R
Silia*Prep* 96-Well Plates				
2 mL/50 mg	1	96W-R31930B-B	96W-R33229G-B	96W-R35530B-B
2 mL/100 mg	1	96W-R31930B-C	96W-R33229G-C	96W-R35530B-C

Determination of Testosterone in Human Urine

General Procedure

1. Mini-SiliaPrep C18 (*PN: SPS-R33229G-J*) was conditioned with 5 mL of methanol and 5 mL of H_2O.
2. The urine sample (*2 mL*) was then slowly aspirated through the cartridge.
3. Cartridge was washed with 5 mL of H_2O and 5 mL of hexane.
4. Analyte was eluted with 5 mL of methanol.
5. The sample was evaporated under a nitrogen stream for 30 min at 40˚C.
6. The analyte was derivatized using 800 QL of Girard-P (*100 mM ammonium acetate buffer, pH 4.2*) and 200 QL of methanol maintained at room temperature for 12 h.
7. Quantification was done using LC-MS/MS apparatus.

Testosterone Recovery

Testosterone	Recovery (%)[a]	
	lot #1	lot #2
	94 ± 2	96 ± 1

[a]Mean Recovery N = 3, 250 ng/mL

Silia*Prep* Reversed-Phase sorbents

Description

Silia*Prep* C8

A moderately hydrophobic and non-polar sorbent used to extract extremely non-polar compounds. This phase is more selective than Silia*Prep* C18 for big compounds such as PAH, vitamin D, and oils as well as greasy compounds.

- SiliCycle Sorbent Number: R31030B
- Loading: 12 %C
- Endcapping: Yes
- Silica Type: 60 Å, 500 m^2/g, 40 - 63 µm

Description

Silia*Prep* Phenyl

A moderately hydrophobic and non-polar sorbent used to extract non-polar compounds with different selectivities through π-π interactions including aromatic compounds and other non-polar phases.

- SiliCycle Sorbent Number: R34030B
- Loading: 9 %C
- Endcapping: Yes
- Silica Type: 60 Å, 500 m^2/g, 40 - 63 µm

Description

Silia*Prep* Cyano

A moderately polar sorbent used as a normal phase (*less polar compared to silica*) to extract acidic, basic and neutral compounds from aqueous solutions. It is also used as a reversed-phase (*less hydrophobic than C8 and C18*).

- SiliCycle Sorbent Number: R38030B
- Loading: 7 %C
- Endcapping: Yes
- Silica Type: 60 Å, 500 m^2/g, 40 - 63 µm

Silia*Prep* Normal Phases

Description

Silia*Prep* Silica

The most polar sorbent, which presents a slightly acidic character and is used to extract various compounds from non-polar solvents through hydrogen bonding. This sorbent is also used for the efficient separation of analytes with similar structures and for removing the baseline noise from organic samples.

- SiliCycle Sorbent Number: R10030B
- Silica Type: 60 Å, 500 m^2/g, 40 - 63 µm

Description

Silia*Prep* Florisil

A polar sorbent (MgO_3Si) presenting a basic character used to extract non-polar to moderately polar compounds from non-polar solvents. The magnesium ion allows retention of chlorinated pesticides, polychlorinated biphenyl (*PCB's*) and polysaccharides.

- SiliCycle Sorbent Number: AUT-0014

Description

Silia*Prep* Alumina-Acidic, Neutral and Basic

Alumina can present either cationic, neutral and acidic character. It is used in a similar fashion as for the Silia*Prep* Silica. The difference is that Alumina is more stable at high pH than silica. These sorbents present favorable retention of aromatic compounds, aliphatic amines and compounds containing electronegative functions.

- SiliCycle Sorbent Number: Acidic: AUT-0053, Neutral: AUT-0054, Basic: AUT-0055
- Alumina Type: 60 Å, 0.9 g/mL, 50 - 200 µm

Silia*Prep* SPE Formats

Formats	Qty/Box	Silia*Prep* C8	Silia*Prep* Phenyl	Silia*Prep* Cyano
Silia*Prep* Cartridges				
1 mL/50 mg	100	SPE-R31030B-01B	SPE-R34030B-01B	SPE-R38030B-01B
1 mL/100 mg	100	SPE-R31030B-01C	SPE-R34030B-01C	SPE-R38030B-01C
3 mL/200 mg	50	SPE-R31030B-03G	SPE-R34030B-03G	SPE-R38030B-03G
3 mL/500 mg	50	SPE-R31030B-03P	SPE-R34030B-03P	SPE-R38030B-03P
6 mL/500 mg	50	SPE-R31030B-06P	SPE-R34030B-06P	SPE-R38030B-06P
6 mL/1 g	50	SPE-R31030B-06S	SPE-R34030B-06S	SPE-R38030B-06S
6 mL/2 g	50	SPE-R31030B-06U	SPE-R34030B-06U	SPE-R38030B-06U
12 mL/2 g	20	SPE-R31030B-12U	SPE-R34030B-12U	SPE-R38030B-12U
25 mL/5 g	20	SPE-R31030B-20X	SPE-R34030B-20X	SPE-R38030B-20X
Silia*Prep* Large Reservoir Volume SPE Cartridges				
10 mL/200 mg	50	SPC-R31030B-10G	SPC-R34030B-10G	SPC-R38030B-10G
10 mL/500 mg	50	SPC-R31030B-10P	SPC-R34030B-10P	SPC-R38030B-10P
Silia*Prep* 96-Well Plates				
2 mL/50 mg	1	96W-R31030B-B	96W-R34030B-B	96W-R38030B-B
2 mL/100 mg	1	96W-R31030B-C	96W-R34030B-C	96W-R38030B-C

Silia*Prep* SPE Formats

Formats	Qty/Box	Silia*Prep* Silica	Silia*Prep* Florisil	Silia*Prep* Acidic Alumina	Silia*Prep* Neutral Alumina	Silia*Prep* Basic Alumina
Silia*Prep* Cartridges						
1 mL/50 mg	100	SPE-R10030B-01B	SPE-AUT-0014-01B	SPE-AUT-0053-01B	SPE-AUT-0054-01B	SPE-AUT-0055-01B
1 mL/100 mg	100	SPE-R10030B-01C	SPE-AUT-0014-01C	SPE-AUT-0053-01C	SPE-AUT-0054-01C	SPE-AUT-0055-01C
3 mL/200 mg	50	SPE-R10030B-03G	SPE-AUT-0014-03G	SPE-AUT-0053-03G	SPE-AUT-0054-03G	SPE-AUT-0055-03G
3 mL/500 mg	50	SPE-R10030B-03P	SPE-AUT-0014-03P	SPE-AUT-0053-03P	SPE-AUT-0054-03P	SPE-AUT-0055-03P
6 mL/500 mg	50	SPE-R10030B-06P	SPE-AUT-0014-06P	SPE-AUT-0053-06P	SPE-AUT-0054-06P	SPE-AUT-0055-06P
6 mL/1 g	50	SPE-R10030B-06S	SPE-AUT-0014-06S	SPE-AUT-0053-06S	SPE-AUT-0054-06S	SPE-AUT-0055-06S
6 mL/2 g	50	SPE-R10030B-06U	SPE-AUT-0014-06U	SPE-AUT-0053-06U	SPE-AUT-0054-06U	SPE-AUT-0055-06U
12 mL/2 g	20	SPE-R10030B-12U	SPE-AUT-0014-12U	SPE-AUT-0053-12U	SPE-AUT-0054-12U	SPE-AUT-0055-12U
25 mL/5 g	20	SPE-R10030B-20X	SPE-AUT-0014-20X	SPE-AUT-0053-20X	SPE-AUT-0054-20X	SPE-AUT-0055-20X
Silia*Prep* Large Reservoir Volume SPE Cartridges						
10 mL/200 mg	50	SPC-R10030B-10G	SPC-AUT-0014-10G	SPC-AUT-0053-10G	SPC-AUT-0054-10G	SPC-AUT-0055-10G
10 mL/500 mg	50	SPC-R10030B-10P	SPC-AUT-0014-10P	SPC-AUT-0053-10P	SPC-AUT-0054-10P	SPC-AUT-0055-10P
Mini-Silia*Prep* SPE Cartridges						
300 mg	50	SPS-R10030B-J	SPS-AUT-0014-J	SPS-AUT-0053-J	SPS-AUT-0054-J	SPS-AUT-0055-J
600 mg	50	SPS-R10030B-Q	SPS-AUT-0014-Q	SPS-AUT-0053-Q	SPS-AUT-0054-Q	SPS-AUT-0055-Q
900 mg	50	SPS-R10030B-R	SPS-AUT-0014-R	SPS-AUT-0053-R	SPS-AUT-0054-R	SPS-AUT-0055-R
Silia*Prep* 96-Well Plates						
2 mL/50 mg	1	96W-R10030B-B	96W-AUT-0014-B	N.A.	N.A.	N.A.
2 mL/100 mg	1	96W-R10030B-C	96W-AUT-0014-C	N.A.	N.A.	N.A.

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Silia*Prep* Ion Exchange Phases

Description

Silia*Prep* TMA Chloride (*Si-SAX*)

Strong anion exchanger sorbent positively charged under all conditions. Used to extract acidic molecules (*pKa 3 - 5*).

- SiliCycle Sorbent Number: R66530B
- Loading: 1.1 mmol/g
- Endcapping: No
- Silica Type: 60 Å, 500 m²/g, 40 - 63 µm

Description

Silia*Prep* Carboxylic Acid (*Si-WCX*)

A weak cation exchanger sorbent used to extract strong basic compounds (*pKa > 9*).

- SiliCycle Sorbent Number: R70030B
- Loading: 1.4 mmol/g
- Endcapping: Yes
- Silica Type: 60 Å, 500 m²/g, 40 - 63 µm

Description

Silia*Prep* Amine (*Si-WAX*)

A weak anion exchanger used instead of a strong anion exchanger for strong anions, thus avoiding irreversible retention (*acidic molecules pKa < 3*). This sorbent is utilized in different applications such as the separation of peptides, drugs and metabolites from physiological fluids, poly- and monosaccharides and structural isomers.

- SiliCycle Sorbent Number: R52030B
- Loading: 1.6 mmol/g
- Endcapping: Yes
- Silica Type: 60 Å, 500 m²/g, 40 - 63 µm

Silia*Prep* SPE Formats

Formats	Qty/Box	Silia*Prep* TMA Chloride	Silia*Prep* Carboxylic Acid	Silia*Prep* Amine
Silia*Prep* Cartridges				
1 mL/50 mg	100	SPE-R66530B-01B	SPE-R70030B-01B	SPE-R52030B-01B
1 mL/100 mg	100	SPE-R66530B-01C	SPE-R70030B-01C	SPE-R52030B-01C
3 mL/200 mg	50	SPE-R66530B-03G	SPE-R70030B-03G	SPE-R52030B-03G
3 mL/500 mg	50	SPE-R66530B-03P	SPE-R70030B-03P	SPE-R52030B-03P
6 mL/500 mg	50	SPE-R66530B-06P	SPE-R70030B-06P	SPE-R52030B-06P
6 mL/1 g	50	SPE-R66530B-06S	SPE-R70030B-06S	SPE-R52030B-06S
6 mL/2 g	50	SPE-R66530B-06U	SPE-R70030B-06U	SPE-R52030B-06U
12 mL/2 g	20	SPE-R66530B-12U	SPE-R70030B-12U	SPE-R52030B-12U
25 mL/5 g	20	SPE-R66530B-20X	SPE-R70030B-20X	SPE-R52030B-20X
Silia*Prep* Large Reservoir Volume SPE Cartridges				
10 mL/200 mg	50	SPC-R66530B-10G	SPC-R70030B-10G	SPC-R52030B-10G
10 mL/500 mg	50	SPC-R66530B-10P	SPC-R70030B-10P	SPC-R52030B-10P
Mini-Silia*Prep* SPE Cartridges				
300 mg	50	SPS-R66530B-J	SPS-R70030B-J	SPS-R52030B-J
600 mg	50	SPS-R66530B-Q	SPS-R70030B-Q	SPS-R52030B-Q
900 mg	50	SPS-R66530B-R	SPS-R70030B-R	SPS-R52030B-R
Silia*Prep* 96-Well Plates				
2 mL/50 mg	1	96W-R66530B-B	96W-R70030B-B	96W-R52030B-B
2 mL/100 mg	1	96W-R66530B-C	96W-R70030B-C	96W-R52030B-C

Silia*Prep* Mixed Mode Phases

Description

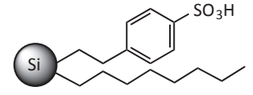
Silia*Prep* C8/Tosic Acid

Silia*Prep* C8/Propylsulfonic Acid

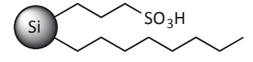
These sorbents are used to extract basic compounds from aqueous solutions, typically drugs and metabolites from physiological fluids.

- SiliCycle Sorbent Number: C8/SCX: R023830B and C8/SCX-2: R028030B
- Endcapping: Yes
- Silica Type: 60 Å, 500 m²/g, 40 - 63 µm

C8/Tosic Acid



C8/Propylsulfonic Acid



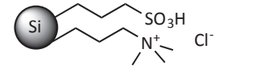
Description

Silia*Prep* Tosic Acid/TMA Chloride

This sorbent is typically used for the separation of acidic and basic molecules from non-ionizable molecules.

- SiliCycle Sorbent Number: R802830B
- Endcapping: Yes
- Silica Type: 60 Å, 500 m²/g, 40 - 63 µm

Tosic Acid/TMA Chloride



Silia*Prep* SPE Formats

Formats	Qty/Box	Silia*Prep* C8/SCX	Silia*Prep* C8/SCX-2	Silia*Prep* SCX/SAX
Silia*Prep* Cartridges				
1 mL/50 mg	100	SPE-R023830B-01B	SPE-R028030B-01B	SPE-R802830B-01B
1 mL/100 mg	100	SPE-R023830B-01C	SPE-R028030B-01C	SPE-R802830B-01C
3 mL/200 mg	50	SPE-R023830B-03G	SPE-R028030B-03G	SPE-R802830B-03G
3 mL/500 mg	50	SPE-R023830B-03P	SPE-R028030B-03P	SPE-R802830B-03P
6 mL/500 mg	50	SPE-R023830B-06P	SPE-R028030B-06P	SPE-R802830B-06P
6 mL/1 g	50	SPE-R023830B-06S	SPE-R028030B-06S	SPE-R802830B-06S
6 mL/2 g	50	SPE-R023830B-06U	SPE-R028030B-06U	SPE-R802830B-06U
12 mL/2 g	20	SPE-R023830B-12U	SPE-R028030B-12U	SPE-R802830B-12U
25 mL/5 g	20	SPE-R023830B-20X	SPE-R028030B-20X	SPE-R802830B-20X
Silia*Prep* Large Reservoir Volume SPE Cartridges				
10 mL/200 mg	50	SPC-R023830B-10G	SPC-R028030B-10G	SPC-R802830B-10G
10 mL/500 mg	50	SPC-R023830B-10P	SPC-R028030B-10P	SPC-R802830B-10P
Silia*Prep* 96-Well Plates				
2 mL/50 mg	1	96W-R023830B-B	96W-R028030B-B	96W-R802830B-B
2 mL/100 mg	1	96W-R023830B-C	96W-R028030B-C	96W-R802830B-C

Silia*Prep* CleanDRUG

Description

Silia*Prep* CleanDRUG:

Silia*Prep* CleanDRUG, a new line of solid phase extraction (*SPE*) products, is designed to extract specific analytes with more reproducibility and efficiency when using sensitive detectors. This product was developed, tested, and quality controlled for drugs of abuse applications.

- SiliCycle Sorbent Number: R651230B
- Silica Type: 60 Å, 500 m^2/g, 40 - 63 µm

Easy SPE Method for Drugs of Abuse Determination in Human Urine

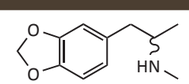
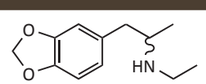
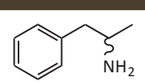
General Procedure

1. Sample (*0.5 mL*) is mixed with 2.5 mL of aqueous H_2SO_4 (*0.1 M*).
2. SiliaPrep CleanDRUG (*PN: SPE-R651230B-03G*) is conditioned with 2 column volumes of methanol, then 2 column volumes of aqueous H_2SO_4 (*0.1 M*).
3. Slowly force or aspirate the sample of urine through the cartridge.
4. Wash the cartridge with 3 mL of phosphate buffer (*KH_2PO_4/K_2HPO_4 pH = 7.0*), then with 3 mL of aqueous H_2SO_4 0.1 M, and finally with 3 mL of methanol.
5. Analyte is eluted with 2 x 3 mL of aqueous NH_4OH (*5% v/v*).
6. Sample is evaporated under a nitrogen stream and, reconstituted with distilled water and methanol (*9:1 v/v*). Finally, the quantification is done using LC-MS apparatus.

Silia*Prep* CleanDRUG SPE Formats

Formats	Qty/Box	Silia*Prep* Product Number
Silia*Prep* Cartridges		
1 mL/50 mg	100	SPEC-R651230B-01B
1 mL/100 mg	100	SPEC-R651230B-01C
3 mL/200 mg	50	SPEC-R651230B-03G
3 mL/500 mg	50	SPEC-R651230B-03P
6 mL/500 mg	50	SPEC-R651230B-06P
6 mL/1 g	50	SPEC-R651230B-06S
6 mL/2 g	50	SPEC-R651230B-06U
12 mL/2 g	20	SPEC-R651230B-12U
25 mL/5 g	20	SPEC-R651230B-20X

Drugs of Abuse Recovery

Drugs			
Recovery (%)[a]	96	98	99

[a]Mean Recovery N = 2, 10 mg/mL to 100 mg/mL

Silia*Chrom*®
HPLC Columns

SILICYCLE inc.
UltraPure SILICA GELS

SiliaChrom HPLC Columns

Using SiliaChrom HPLC Columns in chromatographic applications ensures the following:

- Excellent column efficiency
- Long lifetime and column-to-column reproducibility
- Broad pH range from 0.8 to 12
- Compatibility with 100% aqueous and organic mobile phases
- High surface coverage presenting no bleeding for LC-MS applications

Presentation of the SiliaChrom HPLC Column Series

SiliCycle manufactures a variety of HPLC columns for reversed and normal phase applications. The SiliaChrom series contain more than 40 different phases, and we continue to develop additional, unique and powerful HPLC sorbents. Most of the SiliaChrom products are based on silica. You can be assured of the quality, from raw material synthesis through to the packing process.

We pack bonded phases in a wide range of column dimensions, including standard narrowbore and analytical columns in lengths of 30 to 250 mm, internal diameters of 2.0-4.6 mm, with particle sizes of 2.5, 3.0, 5.0 or 10.0 µm. Also, preparative and semi-preparative HPLC columns are available, in 10, 20, 30 and 50 mm ID with lengths of 50, 100, 150 and 250 mm with particle sizes of 5 and 10 µm. This new product line is designed for the most popular HPLC applications. These columns exhibit superior performance for any type of compound. The SiliaChrom series, with its unique sol-gel process technology, offers the total solution for HPLC end-users: broad pH range (*0.8 - 12*), compatibility with 100% aqueous and organic mobile phases, low bleeding for LC-MS, high surface coverage, and excellent column efficiency. All columns are packed using a consistent slurry packing process to achieve an uniform and stable bed for long lifetime and column-to-column reproducibility.

SiliaChrom HPLC columns

How to build your Part Number

SiliaChrom HPLC columns are available in Narrow Bore, Analytical, Semi-Preparative, and Preparative formats.

Here is an example of a SiliaChrom product number that shows you the way they are structured;

The product numbers start with the **phase** code, followed by the **particle size**, the **pore size**, the **internal diameter**, and finally the **length** codes.

Note: For Guard Columns, add the letter "G" between the "H" and the phase code.

Example;

SiliaChrom AQ C18, **3 µm**, **100 Å**, **4.6 mm** x 150 mm = **H1518**03E-N150

Particle Size

µm	Code
2.5	02
3.0	03
5.0	05
7.0	06
10	07
20	09

Pore Size

Å	Code
100	E
120	G
150	H
300	M

Internal Diameter

Type of Columns	mm	Code
Narrow Bore	2.1	G
Narrow Bore	3.0	H
Analytical	4.6	N
Semi-Preparative	10	Q
Preparative	20	Y
Preparative	30	V
Preparative	50	W
Preparative	100	X

Column Length

mm	Code
10	010
20	020
30	030
50	050
100	100
150	150
200	200
250	250

Particle Size

Pore Size

Internal Diameter

Column Length

phase code

SiliaChrom HPLC column Characteristics

SiliaChrom	Pore size (Å)	Particle size (µm)	Specific Surface area (m^2/g)	Carbon Load (%)	pH range	UPS Code	T Limit* (°C)	Pressure Limit (psi)	Phase Code
Silia*Chrom* AQ C18	100	3, 5, 10	380	18	1.5 - 9.0	L01	60	5,000	H1518
Silia*Chrom* AQ C8	100	3, 5, 10	380	14	1.5 - 8.5	L07	60	5,000	H1508
Silia*Chrom* dt C18	100	2.5, 3, 5, 10	410 - 440	18	1.5 - 9.0	L01	60	5,000	H1418
Silia*Chrom* dt Si	100	2.5, 3, 5, 10	410 - 440	NA	1.0 - 8.0	L03	45	4,500	H1418
Silia*Chrom* XT C18	150	5, 10	200	15	1.5 - 12.0	L01	60	5,000	H1718
Silia*Chrom* XT Fidelity C18	100	3, 5, 10	380	21	1.5 - 12.0	L01	60	5,000	HF1718
Silia*Chrom* SB C18	150	3, 5, 10	200 - 220	12	0.8 - 7.5	L01	60	4,500	H1018
Silia*Chrom* SB C18-300	300	3, 5, 10	80	5	0.8 - 7.5	L01	60	4,500	H1018
Silia*Chrom* SB C8	150	3, 5, 10	200 - 220	7	1.0 - 7.5	L07	60	4,500	H1008
Silia*Chrom* SB C8-300	300	3, 5, 10	80	3	1.0 - 7.5	L07	60	4,500	H1008
Silia*Chrom* XDB C18	150	3, 5, 10	200	15	1.5 - 9.0	L01	60	5,500	H1118
Silia*Chrom* XDB C8	150	3, 5, 10	200	8	1.5 - 9.0	L07	60	5,500	H1108
Silia*Chrom* XDB Si	150	3, 5, 10	200	NA	1.0 - 8.0	L03	45	4,000	H1100
Silia*Chrom* XDB1 C18	100	3, 5, 10	380 - 400	22	1.5 - 10.0	L01	60	5,500	H1218
Silia*Chrom* XDB1 C18-300	300	3, 5, 10	80	8	1.5 - 9.0	L01	60	5,500	H1218
Silia*Chrom* XDB1 C8	100	3, 5, 10	380 - 400	14	1.5 - 8.5	L07	60	5,500	H1208
Silia*Chrom* XDB1 C8-300	300	3, 5, 10	80	4	1.5 - 8.5	L07	60	5,500	H1208
Silia*Chrom* XDB1 C4	100	3, 5, 10	380 - 400	7	1.5 - 8.5	L26	60	5,500	H1204
Silia*Chrom* XDB1 C4-300	300	3, 5, 10	80	3	2.0 - 8.0	L26	60	5,500	H1204
Silia*Chrom* XDB1 C1	100	3, 5, 10	380 - 400	3	1.5 - 8.5	L13	60	5,500	H1201
Silia*Chrom* XDB1 C1-300	300	3, 5, 10	80	1	2.0 - 8.0	L13	60	5,500	H1201
Silia*Chrom* XDB1 CN	100	3, 5, 10	380 - 400	5	2.0 - 8.5	L10	60	5,500	H1220
Silia*Chrom* XDB1 CN-300	300	3, 5, 10	80	3.5	2.0 - 8.0	L10	60	5,500	H1220
Silia*Chrom* XDB1 Amino	100	3, 5, 10	380 - 400	7	2.0 - 8.5	L08	45	5,500	H1260
Silia*Chrom* XDB1 Amino-300	300	3, 5, 10	80	3.5	2.0 - 8.0	L08	45	5,500	H1260
Silia*Chrom* XDB1 Phenyl	100	3, 5, 10	380 - 400	12	1.5 - 9.0	L11	60	4,000	H1240
Silia*Chrom* XDB1 Phenyl-300	300	3, 5, 10	80	4.5	2.0 - 8.0	L11	60	4,000	H1240
Silia*Chrom* XDB1 Diol	100	3, 5, 10	380 - 400	5	2.0 - 8.0	-	45	4,000	H1250
Silia*Chrom* XDB1 Diol-300	300	5, 10	380 - 400	1	2.0 - 8.0	-	45	4,000	H1250
Silia*Chrom* XDB1 Si	100	3, 5, 10	380 - 400	NA	1.0 - 8.0	L03	45	4,000	H1223
Silia*Chrom* XDB1 Si-300	300	3, 5, 10	80	NA	2.0 - 8.0	L03	45	4,000	H1223
Silia*Chrom* XDB2 C18	100	3, 5, 10	380	18	1.5 - 9.0	L01	60	5,000	H1318
Silia*Chrom* SCX	100	3, 5, 10	380	10	2.0 - 8.5	L09	45	5,000	H1800
Silia*Chrom* SCX-300	300	5, 10	80	3.5	2.0 - 8.0	L09	45	5,000	H1800
Silia*Chrom* SAX	100	3, 5, 10	380	6	2.0 - 8.5	L14	45	5,000	H1900
Silia*Chrom* SAX-300	300	5, 10	80	1	2.0 - 8.0	L14	45	5,000	H1900
Silia*Chrom* HILIC	100	3, 5, 10	380	8	2.0 - 8.0	-	60	5 000	H1600
Silia*Chrom* HILIC-300	300	3, 5, 10	80	2.5	2.0 - 8.0	-	60	5,000	H1600

*At pH range 5.0 - 7.5

***You may also find and buy your SiliaChrom online at www.silicycle.com/products/siliachrom-hplc-columns**

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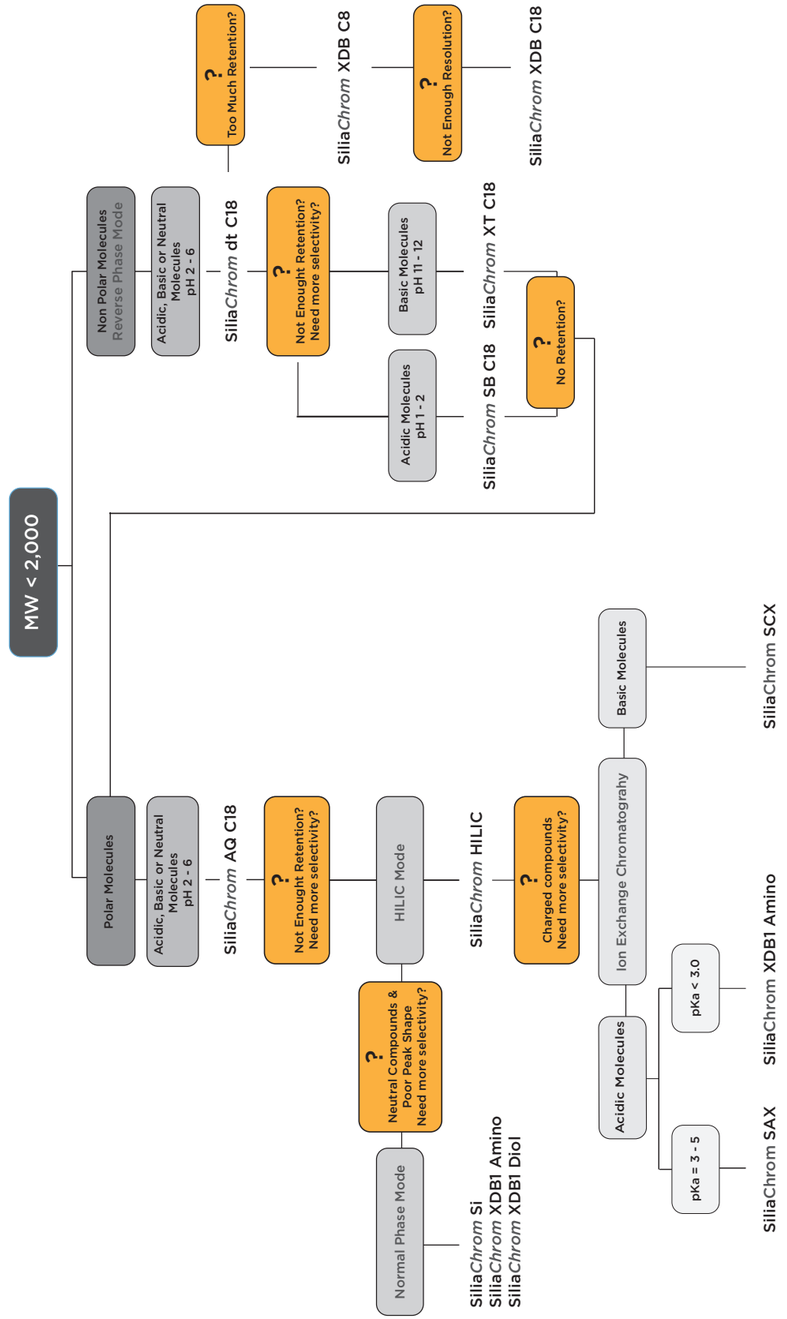
Cross-References Silia*Chrom* HPLC columns

Cross-References Silia*Chrom* HPLC Columns

SiliCycle HPLC Column	Applications	Equivalent to the commercial phase:
Silia*Chrom* AQ C18	Ideal for analytes that require more than 90% of water (*Buffer*)	Zorbax SB Aq, Atlantis dC18, YMC-PACK ODS-AQ
Silia*Chrom* dt C18	Universal C18 for most popular applications (*highest purity of silica gel*)	Inertsil ODS-3, Atlantis T3
Silia*Chrom* XT C18	Excellent durability for high pH Ideal for basic compounds	Gemini, Waters Xterra C18
Silia*Chrom* XT Fidelity C18	Excellent durability to high pH. Ideal for very polar analytes	Waters X-Bridge C18
Silia*Chrom* SB C18	Ideal for MS and ELSD of neutral to slightly polar analytes	Zorbax SB C18
Silia*Chrom* SB C8	Selectivity and peak shape similar to Zorbax SB C8	Zorbax SB C8
Silia*Chrom* XDB C18	Ideal for barbiturates, fat-soluble vitamins, fatty acids, steroids	Zorbax XDB C18, Discovery C18
Silia*Chrom* XDB C8	Selectivity and peak shape similar to Zorbax XDB C8	Zorbax XDB C8, Discovery C8
Silia*Chrom* XDB1 C18	Hydrophobic C18 phase suitable for analysis of wide range of compounds	Luna C18, Ascentis C18, Symmetry C18, Alltima HP C18 HiLoad
Silia*Chrom* XDB1 C8	Selectivity and peak shape similar to Sunfire C8, Luna C8 and Ascentis C8	Sunfire C8, Luna C8, Ascentis C8, Symmetry C8
Silia*Chrom* XDB1 CN	Excellent for basic pharmaceuticals, steroids and other basic compounds	Luna CN, Zorbax SB CN
Silia*Chrom* XDB1 Amino	Superior general purpose amino phase. Ideal for carbohydrates	Luna NH_2
Silia*Chrom* XDB1 Phenyl	Ideal for polynuclear aromatic hydrocarbons, putines and polar aromatics	Zorbax SB Phenyl
Silia*Chrom* XDB1 Diol	Excellent for normal phase applications with more hydrophobic activity	Nucleosil Diol, Luna Diol
Silia*Chrom* XDB1 Si	Ideal for normal phase applications	Luna Silica
Silia*Chrom* XDB2 C18	Perfect peak symmetry for acidic, basic and neutral compounds	Luna C18 (2), Sunfire C18
Silia*Chrom* SCX	Ideal for charged analytes	Luna SCX
Silia*Chrom* SAX	Ideal for charged analytes	Agilent SB-SAX
Silia*Chrom* HILIC	Ideal for MedChem laboratories Isolation of very polar analytes	Unique

Silia*Chrom* Selection Guide



TEL.: 1 418 874.0054 FAX: 1 418 874.0355 TOLL-FREE: 1 877.SILICYCLE (NORTH AMERICA ONLY) WWW.SILICYCLE.COM INFO@SILICYCLE.COM

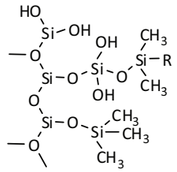
Silia*Chrom* AQ C8 and AQ C18

Description

Universal 100% aqueous-compatible HPLC columns

Silia*Chrom* AQ adsorbents present an optimum ratio of C18 (C8) short TMS chains and some free silanol groups. This new technology shows good peak shapes for any type of molecule (*acid, neutral and base*).

Structure



For C18 R = $(CH_2)_{17}CH_3$
For C8 R = $(CH_2)_7CH_3$

Silia*Chrom* AQ C18

Silia*Chrom* AQ C8

Sorbent Characteristics

- **Pore Size: 100 Å**
- **Specific Surface Area: 380 m²/g**
- **Particles Sizes Available: 3, 5 and 10 µm**
- **Typical Carbon Loading: Silia*Chrom* AQ C18 18%**
  Silia*Chrom* AQ C8 14%

Silia*Chrom* AQ Main Characteristics

- **Exceptional stability at pH 1.5 to 9.0**
- **Inertness for acidic and basic analytes**
- **Compatible from 100% aqueous mobile phase to 100% organic**
- **Rapid equilibration**
- **Reduced need for mobile phase modifiers**
- **Partially endcapped**

Dewetting Phenomena

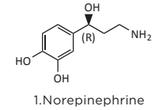
The dewetting phenomena is the formation of drops on the solid surface caused by hydrophobic repulsions of the highly hydrophobic sorbents. This phenomena is illustrated, shown by the following scheme.

General procedure

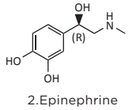
- The mixture of catecholamines is eluted on the column
- The flow is then stopped
- The column is stored in this condition during 18 h
- The mixture is then re-injected after a reconditioning step

Chromatographic conditions

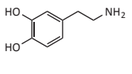
- **Column**: Silia*Chrom* AQ C18, 5 µm
- **Column size**: 4.6 x 150 mm
- **SiliCycle P/N**: H151805E-N150
- **Mobile phase**: 1% AcOH in water
- **Temperature**: 23°C
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 265 nm
- **Injection volume**: 5 µL



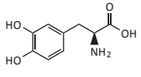
1.Norepinephrine



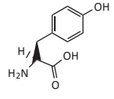
2.Epinephrine



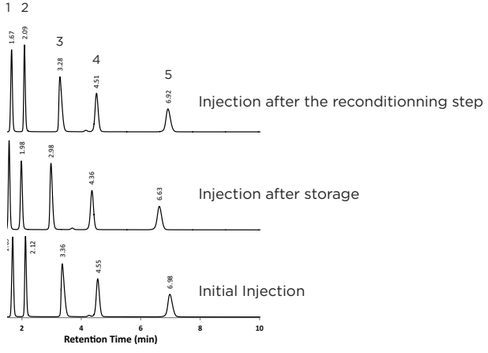
3.Dopamine



4.Levodopa



5.Tyrosine



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

Retention Capacity of DMSO on Silia*Chrom* AQ C18

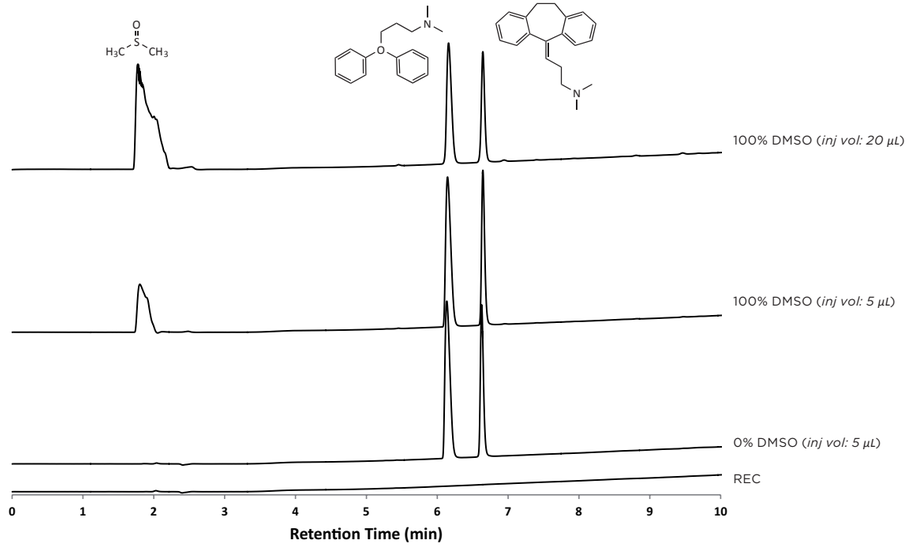
DMSO (*DimethylSulfoxide*) is an excellent solvent to solubilize most compounds. Unfortunately, this solvent is not volatile and in some C18 columns the DMSO can interact with the stationary phase and create a loss of selectivity. In this case, the only way to inhibit this effect is to use preparative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

Chromatographic conditions

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

Gradient

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



Statistic Analysis Results

Conditions	As $_{DMSO}$	Tr $_{DMSO}$ (min)	K'$_{DMSO}$	W$_{DMSO}$	Tr $_{diphenhydramine}$ (min)	Tr $_{amitriptyline}$ (min)
0% DMSO 5 µL	-	-	-	-	6.14	6.63
100% DMSO 5 µL	2.29	1.80	0.09	0.3	6.15	6.64
100% DMSO 20 µL	4.10	1.78	0.08	0.5	6.16	6.64

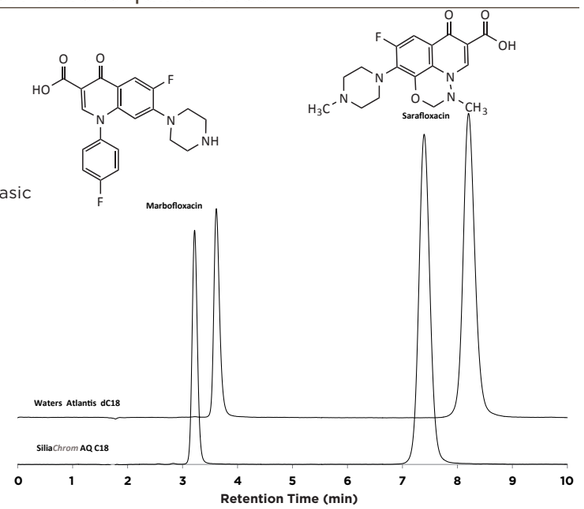
Conclusion: The study shows that DMSO does not interact with the Silia*Chrom* AQ C18. No specific retention is observed. The Silia*Chrom* AQ C18 is an excellent choice to purify components contaminated with DMSO.

Peak Shape Evaluation for Zwetterion Fluoroquinolones

High separation power for zwetterion analysis.

Chromatographic conditions

- **Column**: Silia*Chrom* AQ C18, 5 µm
- **Column size**: 4.6 x 150 mm
- **SiliCycle P/N**: H151805E-N150
- **Mobile phase**: 2.5 mM potassium phosphate monobasic (*adjust to pH 2.5 with H_3PO_4*)/ethanol (*68/32*)
- **Temperature**: 23°C
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 275 nm
- **Injection volume**: 10 µL

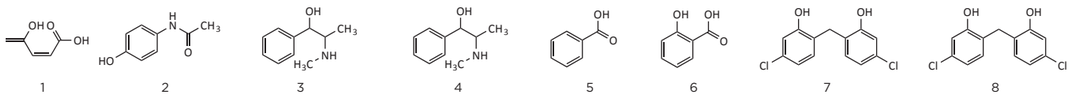


Peak Shape Results

Product	Asymmetry (USP) Silia*Chrom* AQ C18	Asymmetry (USP) Atlantis dC18
Marbofloxacin	1.12	1.29
Sarafloxacin	1.09	1.14

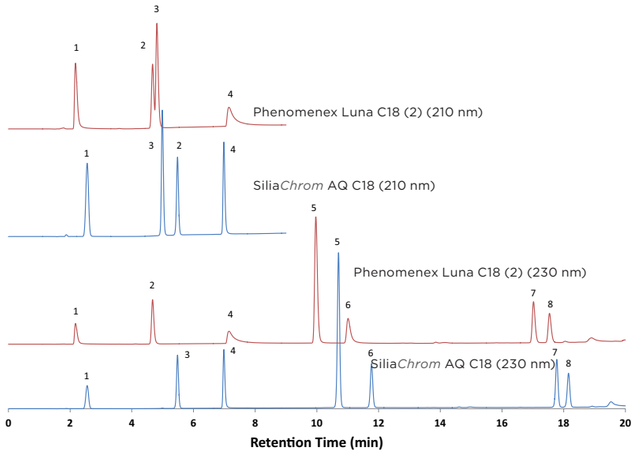
Evaluation of Resolution and Peak Shape

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



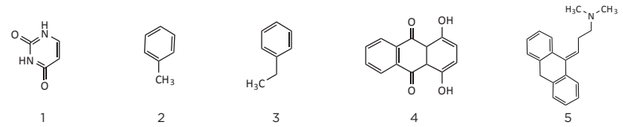
Chromatographic conditions

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



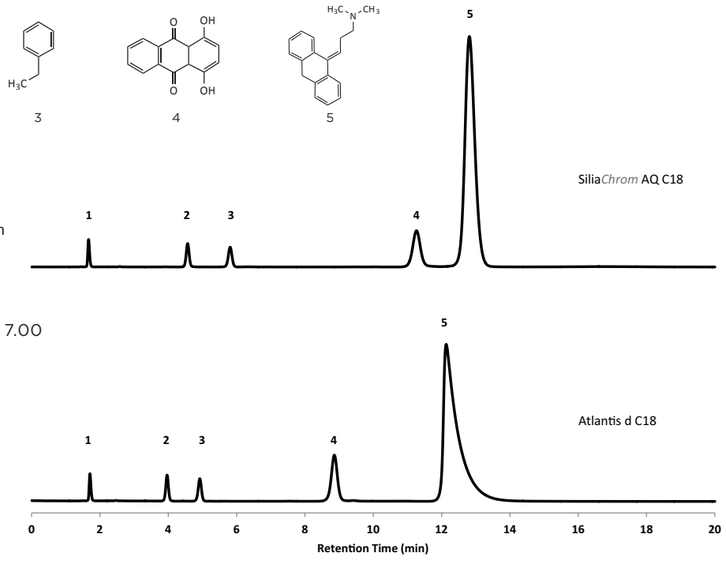
Silia*Chrom* AQ C18 for Basic Compounds

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



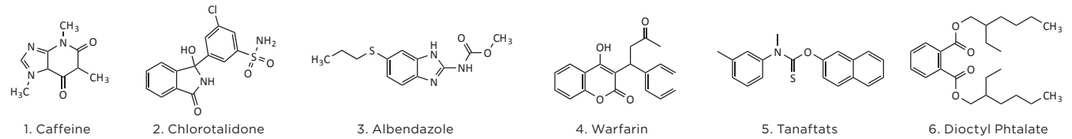
Chromatographic conditions

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



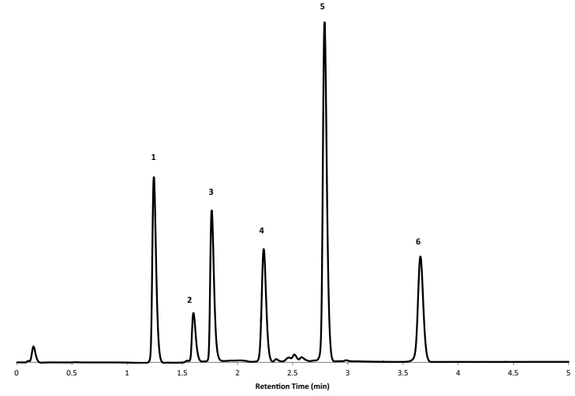
Rapid HPLC with Silia*Chrom* AQ C18 - Multi-Component Sample

Indispensable for pharmaceutical quality control, conjugate efficiency and rapidity.



Chromatographic conditions

- **Column**: Silia*Chrom* AQ C18, 5 µm
- **Column size:** 3.0 x 30 mm
  SiliCycle P/N: H151805E-H030
- **Mobile phase:**
  MPA: 0.1% TFA in ACN/water (5/95)
  MPB: 0.1% TFA in ACN/water (95/5)
  Linear gradient: MPA to MPB, 2.25 minutes
- **Temperature**: 23°C
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 254 nm
- **Injection volume**: 5 µL



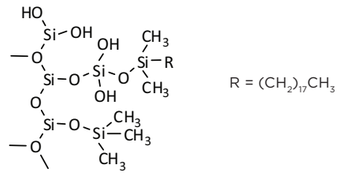
Silia*Chrom* dt C18

Description

Universal 100% aqueous compatible HPLC columns.

The modified surface chemistry of **Silia*Chrom* AQ** and **Silia*Chrom* dt** columns is identical but the silica framework does not present any metals in the dt sorbent.

Structure



R = $(CH_2)_{17}CH_3$

Silia*Chrom* AQ purity: 99.999% SiO_2

Silia*Chrom* dt Purity: 99.9999% SiO_2
(*no metal content*)

Silia*Chrom* dt C18

Sorbent Characteristics

- **Pore Size: 100 Å**
- **Specific Surface Area: 410 - 440 m^2/g**
- **Particle Sizes Available: 2.5, 3, 5 and 10 µm**
- **Typical Carbon Loading: Silia*Chrom* dt C18 18%**

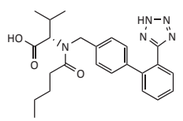
Silia*Chrom* dt Main Characteristics

- **Enhances retention of hydrophilic molecules**
- **Low bleeding and high sensitivity for LC-MS**
- **Extremely low metal content level (*< 10 ppm*)**
- **Good tolerance for direct injection of biological matrix (*dirty samples*)**
- **Higher surface area**
- **Partially endcapped**

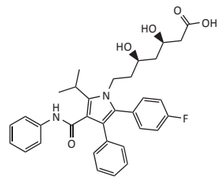
Assay for QC Testing of Blood Pressure and Cholesterol Medication

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

A: Aspirine



B: Valsartan



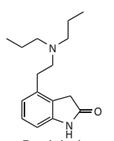
C: Atorvastatin

Chromatographic conditions

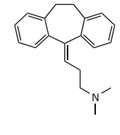
- **Column**: Silia*Chrom* dt C18, 5 µm
- **Column size**: 4.6 x 150 mm
  SiliCycle P/N: H141802E-N150
- **Mobile phase:**
  Methanol/H_2O (70/30), 0.1% (v/v) formic acid
- **Temperature**: 30°C
- **Flow rate**: 0.800 mL/min
- **Detector**: UV at 280 nm
- **Injection volume**: 10 µL

Ropinirole and Amitriptyline Detection in Human Plasma

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



Ropinirole



Amitriptyline

Chromatographic conditions

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

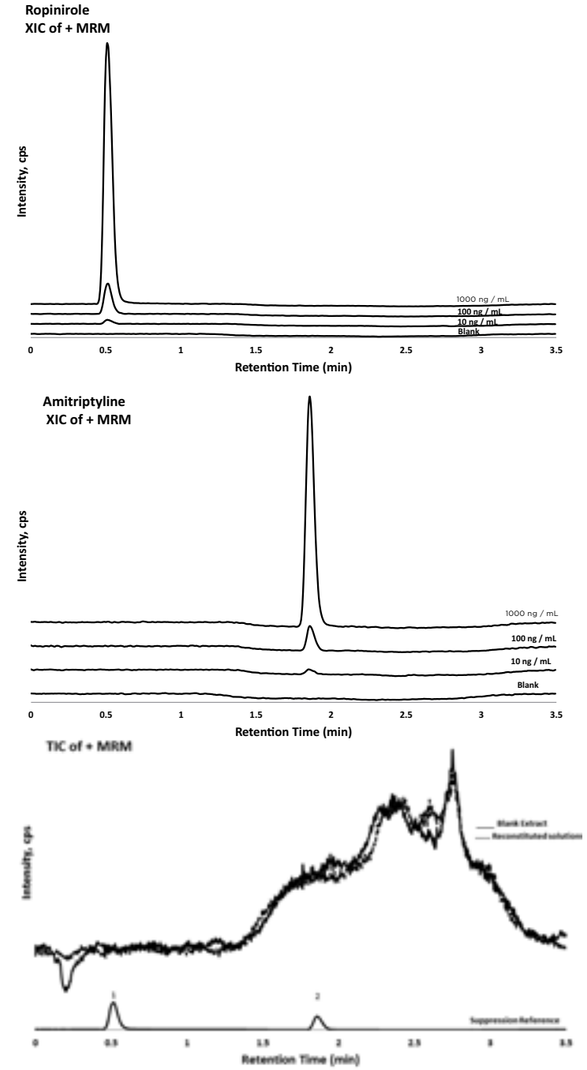
Gradient

Time (min)	MPA (%)	MPB (%)	Flow (mL/min)
0.00 - 0.20	85	15	1.000
0.21 - 1.20	50	50	1.000
1.21 - 1.60	0	100	1.000
1.61 - 3.50	85	15	1.000

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

Tandem mass spectroscopy conditions

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

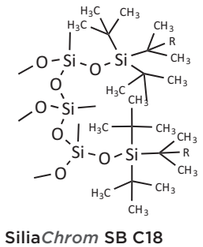


Silia*Chrom* SB C18 and C8

Description

Silia*Chrom* SB C18 and C8 surfaces are treated with an organic form of silicon to increase the number of silanol groups on the surface. After this step, the surface is bonded with a silane containing two functions. One function is a protecting group that shields the area and protects the surface from an acid attack from the mobile phase. The ion H_3O^+ does not have access to the surface to break the O-Si bond (*steric effect*). The other function is the linear hydrophobic chain with 18 or 8 carbons.

Structure



For C18 R = $(CH_2)_{17}CH_3$
For C8 R = $(CH_2)_7CH_3$

Silia*Chrom* SB C18
Silia*Chrom* SB C8

Sorbent Characteristics

- **Pore Size: 100 Å**
- **Specific Surface Area: 200 - 220 m²/g**
- **Particle Sizes Available: 3, 5 and 10 µm**
- **Typical Carbon Loading: Silia*Chrom* SB C18 12%**
  Silia*Chrom* SB C8 7%

Silia*Chrom* SB Main Characteristics

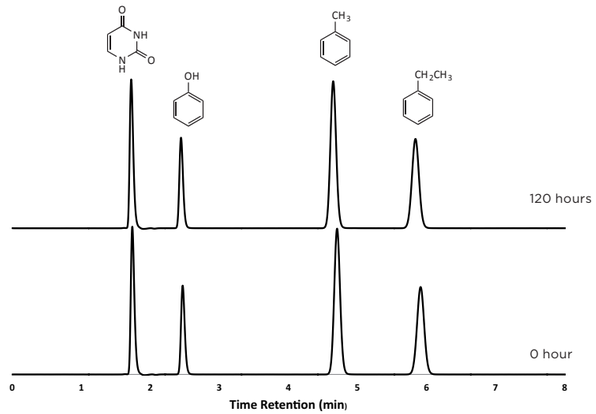
- **Extremely low pH limits (*0.5 - 7.5*)**
- **Extremely low bleeding for LC-MS applications under acidic conditions**
- **Compatible with mobile phases ranging 100% aqueous to 100% organic**
- **Non endcapped**

Stability of Silia*Chrom* SB C18 at Low pH Conditions

Acidic mobile phases have widespread applications in the reversed phase HPLC separation of many important pharmaceutical and environmental compounds. Analytes such as pharmaceuticals and biomolecules often show peak shape, retention and selectivity changes when the mobile phase pH is changed from neutral to acidic pH (*pH 1.0*). In fact, lowering the pH helps to suppress silanol interactions between basic compounds and the residual surface silanols, thus resulting in less tailing and better retention of acidic compounds (*pKa lower than 2*).

Chromatographic conditions

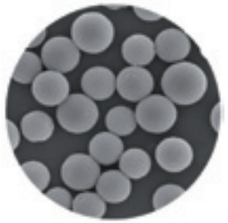
- **Column**: Silia*Chrom* SB C18, 5 µm
- **Column size**: 4.6 x 150 mm
  SiliCycle P/N: H101805H-N150
- **Mobile phase**: 2% TFA in ACN/water (*60/40*)
  Solution pH: 1.00
- **Temperature**: 23°C
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 270 nm
- **Injection volume**: 10 µL



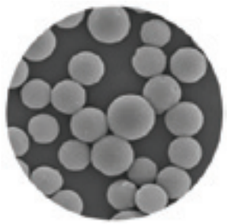
Silia*Chrom* SB C18 (*Ethylbenzene*)

Time (hour)	RT (min)	TF (USP)	N (USP)
0	5.91	1.01	14,014
24	5.89	1.02	14,085
48	5.77	1.02	14,023
72	5.83	1.02	14,076
96	5.85	1.01	14,087
120	5.84	1.02	14,050
Mean	5.85	1.02	14,056
RSD (%)	**0.84**	**0.51**	**0.23**

No column degradation under extreme pH conditions



Silia*Chrom* SB C18 before



Silia*Chrom* SB C18 after

The HPLC column was used under extreme pH conditions and, even after 5 days of continuous injections, the number of theoretical plates (*N*), the tailing factor (*TF*) and the retention times (*RT*) are comparable. The sorbent kept its chemical and structural integrity, which we have proven with similar chromatograms and scanning electron microscope pictures (*SEM*) before and after 120 hours of use.

In conclusion, our Silia*Chrom* SB C18 and SB C8 columns are stable at low pH conditions.

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Silia*Chrom* XT C18 and XT C18 Fidelity

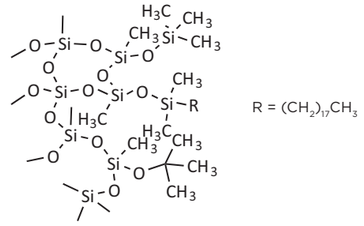
Description

Silia*Chrom* XT C18 and XT C18 Fidelity are compatible with low or high pH conditions. The key is to have a hybrid surface to reduce the solubility of silica at high pH. In fact, the Silia*Chrom* XT C18 and the XT C18 Fidelity silica are coated with a monomeric methyltriethoxysilane/tetraethoxysilane prepolymer, followed by a special thermic treatment to get a rigid surface that is less soluble than untreated silica itself at high pH.

The Silia*Chrom* XT C18 column is designed for applications to be run at very high pH (*up to 12.0*) at room temperature but it is also suitable for low pH (*down to 1.5*).

The Silia*Chrom* XT C18 Fidelity is used at high pH conditions with a higher thermal stability. The only difference between Silia*Chrom* XT C18 and the XT C18 Fidelity is the way the HPLC column is packed (*proprietary information*) which gives more robustness at high pH and temperature.

Structure



R = $(CH_2)_{17}CH_3$

Silia*Chrom* XT C18 and XT C18 Fidelity

Sorbent Characteristics

- **Pore Size: 150 Å**
- **Specific Surface Area: 380 m^2/g**
- **Particle Sizes Available: 3, 5 and 10 µm**
- **Typical Carbon Loading: Silia*Chrom* XT C18 15%**
  Silia*Chrom* XT C18 Fidelity 21%

Silia*Chrom* XT Main Characteristics

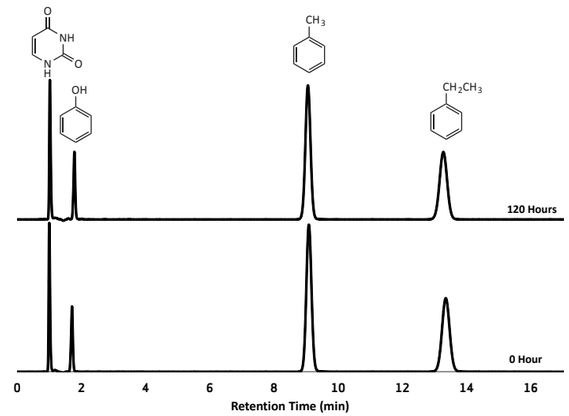
- **Excellent durability at high pH (*up to 12*)**
- **Ideal for basic compounds**
- **High thermal stability**
- **Ideal for auto-purification (*Prep. LC-MS*)**
- **Double endcapped**
- **Best HPLC columns for either metabolic or metabolite studies**

Stability of Silia*Chrom* XT C18 Fidelity at High pH Conditions

For some applications, it is necessary to work at high pH to increase the selectivity or to optimize peak shape. This is the case with basic organic compounds (*pKa > 9.0*). It is the reason why it is important to have chromatographic phases stable at alkaline pH. This study demonstrates the stability of the Silia*Chrom* XT C18 Fidelity at high pH.

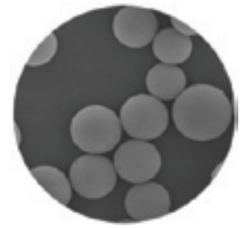
Chromatographic conditions

- **Column**: Silia*Chrom* XT C18 Fidelity, 5 µm
- **Column size**: 4.6 x 150 mm
  SiliCycle P/N: HF171805H-N150
- **Mobile phase**: 0.2% TEA in ACN/water (*55/45*) (*v/v*)
  Solution pH: 11.5
- **Temperature**: 23°C
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 270 nm

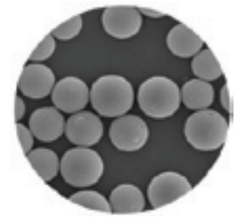


Silia*Chrom* XT C18 Fidelity (*Ethylbenzene*)

Time (hour)	RT (min)	TF (USP)	N (USP)
0	13.35	1.01	13,623
24	13.29	1.01	13,648
48	13.27	1.01	13,689
72	13.25	1.00	13,604
96	13.24	1.00	13,649
120	13.28	1.00	13,582
Mean	13.28	1.01	13,633
RSD (%)	**0.29**	**0.54**	**0.28**



Silia*Chrom* XT C18 Fidelity before



Silia*Chrom* XT C18 Fidelity after

The HPLC column was used under extreme pH conditions, and even after 5 days of continuous injections, the number of theoretical plates (*N*), the tailing factor (*TF*) and the retention times (*RT*) are comparable. The sorbent kept its chemical and structural integrity, which we have proven with similar chromatograms and scanning electron microscope (*SEM*) pictures before and after 120 hours of use.

In conclusion, our Silia*Chrom* XT C18 Fidelity columns are stable at high pH conditions.

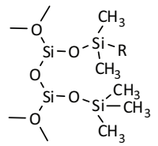
Silia*Chrom* XDB C18 & XDB C8

Description

Silia*Chrom* XDB C18 and C8 are specially designed with a bigger pore size and lower surface area for the separation of large hydrophobic molecules. The relatively low surface area allows a shorter retention time for such compounds.

Silia*Chrom* XDB phases are ideal for separation of barbiturates, fat-soluble vitamins, fatty acids and steroids.

Structure



For C18 R = $(CH_2)_{17}CH_3$
For C8 R = $(CH_2)_7CH_3$

Silia*Chrom* XDB C18

Silia*Chrom* XDB C8

Sorbent Characteristics

- **Pore Size: 150 Å**
- **Specific Surface Area: 200 m²/g**
- **Particle Sizes Available: 3, 5 and 10 µm**
- **Typical Carbon Loading: Silia*Chrom* XDB C18 15%**
  Silia*Chrom* XDB C8 8%

Silia*Chrom* XDB C18 Main Charateristics

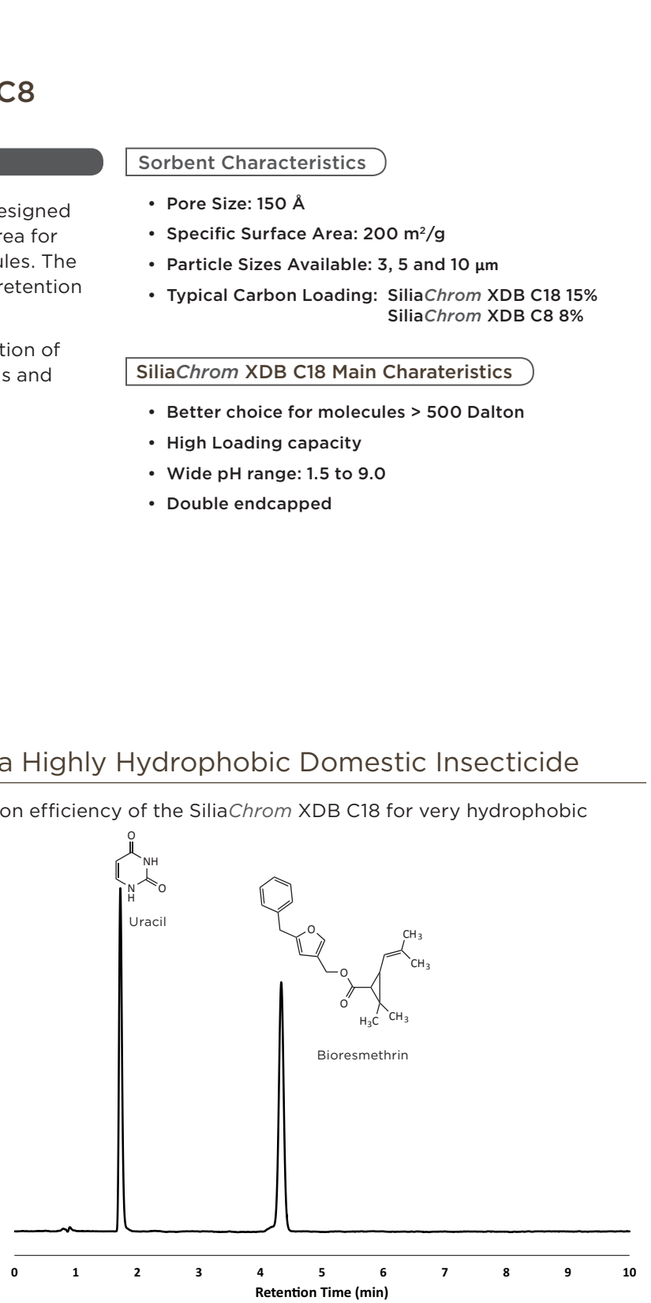
- **Better choice for molecules > 500 Dalton**
- **High Loading capacity**
- **Wide pH range: 1.5 to 9.0**
- **Double endcapped**

Resolution and Peak Shape of a Highly Hydrophobic Domestic Insecticide

This application illustrates the high separation efficiency of the Silia*Chrom* XDB C18 for very hydrophobic compounds.

Chromatographic conditions

- **Column**: Silia*Chrom* XDB C18, 5 µm
- **Column size**: 4.6 x 150 mm
  SiliCycle P/N: H111805H-N150
- **Mobile phase**: ACN/water (*90/10*)
- **Temperature**: 23°C
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 235 nm
- **Injection Volume**: 1 µL



Column Performance Results

Compounds	Retention Time (min)	Peak Asymmetry Factor (USP)	Theoretical Plates (USP)
Uracil	1.72	1.26	5,936
Bioresmethrin	4.34	1.03	14,090

Silia*Chrom* XDB1

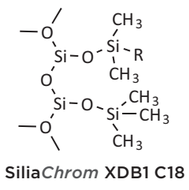
Description

Silia*Chrom* XDB1 phases have a wider range of polarity than other SiliCycle HPLC columns (*C18 to normal phase*). This phase has the maximum bonding density regardless of compound's polarity. This allows for the least amount of interaction between the analytes and the surface OH. This phase is not recommended for samples containing highly hydrophobic compounds.

All Silia*Chrom* XDB1 are available in 3, 5 and 10 µm exept the Diol-300 which is not available in 3 µm.

The Silia*Chrom* XDB1 C18: Designed for maximum hydrophobicity and efficiency for dirty samples.

Structure



R = $(CH_2)_{17}CH_3$

Silia*Chrom* XDB1 C18

Sorbent Characteristics

Silia*Chrom* XDB1 C18

- **Pore Size: 100 Å**
- **Specific Surface Area: 380 - 400 m²/g**
- **Typical Carbon Loading: 22%**
- **pH Stability: 1.5 - 10.0**

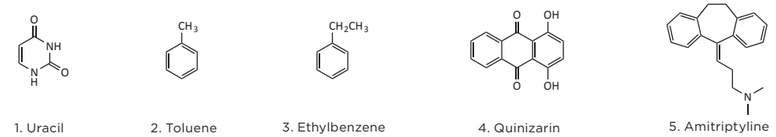
Silia*Chrom* XDB1 C18-300

- **Pore Size: 300 Å**
- **Specific Surface Area: 80 m²/g**
- **Typical Carbon Loading: 8%**
- **pH Stability: 1.5 - 9.0**

Silia*Chrom* XDB1 C8 and C18 Main Characteristics

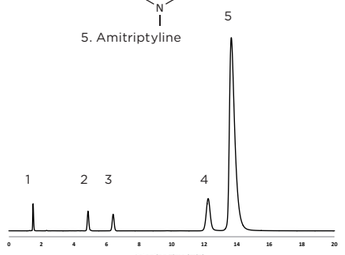
- **Better choice for molecules > 500 Dalton**
- **High Loading capacity**
- **Wide pH range: 1.5 to 10.0**
- **Double endcapped**

Highly Base Deactivated C18



Chromatographic conditions

- **Column**: Silia*Chrom* XDB1 C18, 5 µm
- **Column size**: 4.6 x 150 mm
  SiliCycle P/N: H121805H-N150
- **Mobile phase**: MeOH/20 mM potassium phosphate monobasic pH = 7.00 (*80/20*)
- **Temperature**: 23°C
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 254 nm
- **Injection Volume**: 1 µL



Column Performance Results

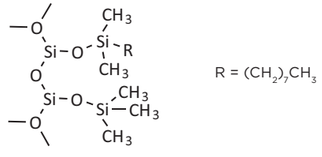
Compounds	Retention Time (min)	Peak Asymmetry Factor (USP)	Theoretical Plates (USP)
Uracil	1.49	1.27	3,778
Toluene	4.86	1.09	12,144
Ethylbenzene	6.40	1.02	13,026
Quinizarin	12.24	1.07	11,525
Amitriptyline	13.66	1.76	8,190

SiliaChrom XDB1

Description

SiliaChrom XDB1 C8: Exceptionally stable with high bonding coverage and low silanol activity.

Structure



R = $(CH_2)_7CH_3$

SiliaChrom XDB1 C8

Sorbent Characteristics

SiliaChrom XDB1 C8

- Pore Size: 100 Å
- Specific Surface Area: 380 - 400 m^2/g
- Typical Carbon Loading: 14%
- pH Stability: 1.5 - 10.0

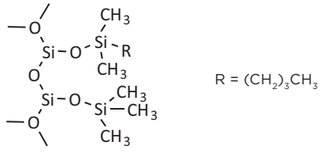
SiliaChrom XDB1 C8-300

- Pore Size: 300 Å
- Specific Surface Area: 80 m^2/g
- Typical Carbon Loading: 4%
- pH Stability: 1.5 - 8.5

Description

SiliaChrom XDB1 C4: Exceptionally stable with high bonding coverage and low silanol activity

Structure



R = $(CH_2)_3CH_3$

SiliaChrom XDB1 C4

Sorbent Characteristics

SiliaChrom XDB1 C4

- Pore Size: 100 Å
- Specific Surface Area: 380 - 400 m^2/g
- Typical Carbon Loading: 7%
- pH Stability: 1.5 - 8.5

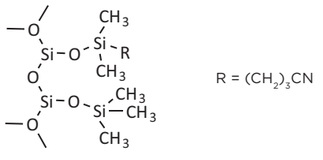
SiliaChrom XDB1 C4-300

- Pore Size: 300 Å
- Specific Surface Area: 80 m^2/g
- Typical Carbon Loading: 3%
- pH Stability: 2.0 - 8.0

Description

SiliaChrom XDB1 CN: Maximum hydrophobicity and accepts normal and reversed phase conditions.

Structure



R = $(CH_2)_3CN$

SiliaChrom XDB1 CN

Sorbent Characteristics

- SiliaChrom XDB1 CN
- Pore Size: 100 Å
- Specific Surface Area: 380 - 400 m^2/g
- Typical Carbon Loading: 5%
- pH Stability: 1.5 - 8.5

SiliaChrom XDB1 CN-300

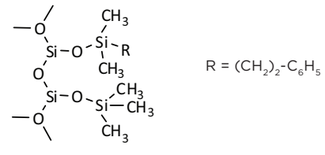
- Pore Size: 300 Å
- Specific Surface Area: 80 m^2/g
- Typical Carbon Loading: 3.5%
- pH Stability: 2.0 - 8.0

SiliaChrom XDB1

Description

SiliaChrom XDB1 Phenyl: Highly retentive phase for aromatic and unsaturated compounds.

Structure



R = $(CH_2)_2$-C_6H_5

SiliaChrom XDB1 Phenyl

Sorbent Characteristics

SiliaChrom XDB1 Phenyl

- Pore Size: 100 Å
- Specific Surface Area: 380 - 400 m^2/g
- Typical Carbon Loading: 12%
- pH Stability: 1.5 - 9.0

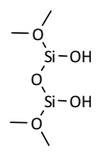
SiliaChrom XDB1 Phenyl-300

- Pore Size: 300 Å
- Specific Surface Area: 80 m^2/g
- Typical Carbon Loading: 4.5%
- pH Stability: 2.0 - 8.0

Description

SiliaChrom XDB1 Si: Designed for normal phase conditions, presents high surface area and low metal content.

Structure



SiliaChrom XDB1 Si

Sorbent Characteristics

SiliaChrom XDB1 Si

- Pore Size: 100 Å
- Specific Surface Area: 380 - 400 m^2/g
- pH Stability: 1.0 - 8.0

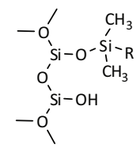
SiliaChrom XDB1 Si-300

- Pore Size: 300 Å
- Specific Surface Area: 80 m^2/g
- pH Stability: 1.0 - 8.0

Description

SiliaChrom XDB1 DIOL: Excellent for normal phase applications with more hydrophobic activity.

Structure



R = $(CH_2)_3OCH_2CH(OH)CH_2OH$

SiliaChrom XDB1 Diol

Sorbent Characteristics

SiliaChrom XDB1 DIOL

- Pore Size: 100 Å
- Specific Surface Area: 380 - 400 m^2/g
- Typical Carbon Loading: 5%
- pH Stability: 2.0 - 8.0

SiliaChrom XDB1 DIOL-300

- Pore Size: 300 Å
- Specific Surface Area: 80 m^2/g
- Typical Carbon Loading: 1%
- pH Stability: 2.0 - 8.0

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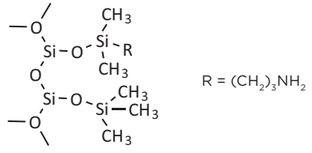
Silia*Chrom* XDB1

Description

Silia*Chrom* XDB1 AMINO: Superior general purpose amino phase. Recommended for normal phase analysis and excellent for sugar analysis.

Structure



Silia*Chrom* XDB1 AMINO

Silia*Chrom* XDB1 AMINO Main Characteristics

- **Wide pH range**
- **High carbon loading**
- **Very stable for agressive mobile phases**
- **Accepts large injection volume (*50 µL and more*)**
- **Double endcapped**

Sorbent Characteristics

Silia*Chrom* XDB1 AMINO

- **Pore Size: 100 Å**
- **Specific Surface Area: 380 - 400 m²/g**
- **Typical Carbon Loading: 6%**
- **pH Stability: 2.0 - 8.5**

Silia*Chrom* XDB1 AMINO-300

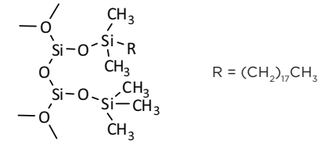
- **Pore Size: 300 Å**
- **Specific Surface Area: 80 m²/g**
- **Typical Carbon Loading: 2.5%**
- **pH Stability: 2.0 - 8.0**

Silia*Chrom* XDB2 C18

Description

Silia*Chrom* XDB2 C18: Designed to be a mid-hydrophobic C18 phase with 18% of carbon loading, like most of the popular reversed-phase HPLC columns on the market. This phase demonstrates a balanced hydrophobic adsorption in order to avoid excessive retention of hydrophobic compounds.

Structure



Silia*Chrom* XDB2 C18

Sorbent Characteristics

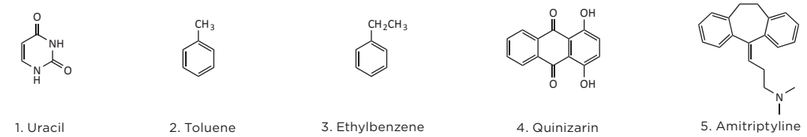
Silia*Chrom* XDB2 C18

- **Pore Size: 100 Å**
- **Specific Surface Area: 380 - 400 m²/g**
- **Particle Sizes Available: 3, 5 and 10 µm**
- **Typical Carbon Loading: 18%**
- **pH Stability: 1.5 - 9.0**

Silia*Chrom* XDB2 C18 Main Charateristics

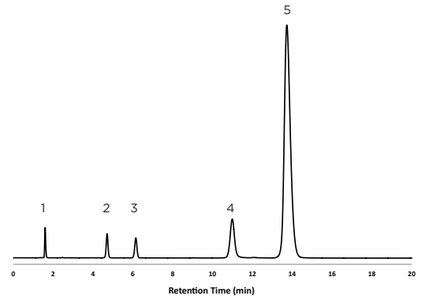
- **Great column-to-column and batch-to-batch reproducibility (*popular for QC/QA laboratory*)**
- **Typical average value for carbon loading (*18%*)**
- **Good peak shape for basic, acidic and neutral analytes**
- **Stronger separation power for isomers**
- **Double endcapped**

Highly Base Deactivated C18



Chromatographic conditions

- **Column**: Silia*Chrom* XDB2 C18, 5 µm
- **Column size**: 4.6 x 150 mm
  SiliCycle P/N: H131805H-N150
- **Mobile phase**: MeOH/20 mM potassium phosphate monobasic pH = 7.00 (*80/20*)
- **Temperature**: 23°C
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 254 nm
- **Injection Volume**: 1 µL



Column Performance Results

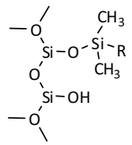
Compounds	Retention Time (min)	Peak Asymmetry Factor (USP)	Theoretical Plates (USP)
Uracil	1.61	1.24	4 618
Toluene	4.73	1.04	12 858
Ethylbenzene	6.19	1.00	13 633
Quinizarin	11.18	1.03	12 277
Amitriptyline	13.53	1.29	9 451

Silia*Chrom* HILIC

Description

Silia*Chrom* HILIC (*hydrophilic interaction chromatography*) HPLC columns are designed to retain highly polar analytes. SiliaChrom HILIC has a selectivity that is complementary to reversed-phase columns. In fact, it has a higher retention for hydrophilic compounds in HILIC mode. HILIC sorbent is more stable and offers higher reproducibility than normal phase silica or amino columns. This phase is ideal for MedChem laboratories and is approved for SFC applications.

Structure



R = $(CH_2)_3NH\text{-}CO\text{-}NH_2$

Silia*Chrom* HILIC

Sorbent Characteristics

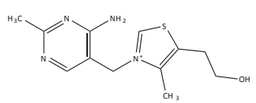
Silia*Chrom* HILIC

- **Pore Size: 100 Å**
- **Specific Surface Area: 410 - 440 m²/g**
- **Particle Sizes Available: 3, 5 and 10 µm**
- **Typical Carbon Loading: 8%**
- **pH Stability: 2.0 - 8.0**

Silia*Chrom* HILIC Main Characteristics

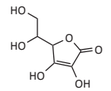
- **Unique chemistry (*urea*)**
- **Accepts normal and reversed phase applications**
- **Best replacement for amino HPLC column**
- **Provides high efficiency and rapid equilibration**
- **Enhanced sensitivity in mass spectrometry**
- **Non endcapped**

Silia*Chrom* HILIC: Separation of Vitamin B Complex and Vitamin C

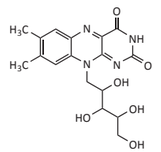


A. Thiamine (B1)

B. Pyridoxine (B6)



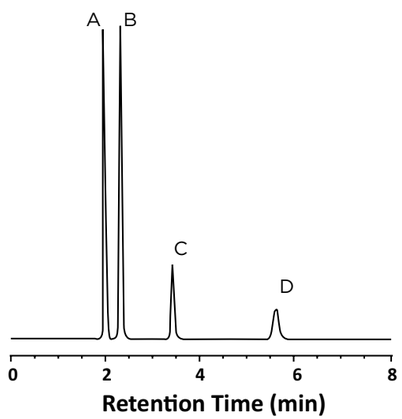
C. Ascorbic Acid (C)



D. Riboflavine (B2)

Chromatographic conditions

- **Column**: Silia*Chrom* HILIC, 5 µm
- **Column size**: 4.6 x 200 mm
  SiliCycle P/N: H131805H-N150
- **Mobile phase**: 0.1% TFA in water/0.1% in ACN (*90/10*)
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 280 nm

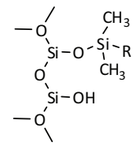


Silia*Chrom* SCX-SAX

Description

Silia*Chrom* SCX provides excellent resolution and peak shape for cationic analytes. SiliaChrom SCX contains a benzene sulfonic acid ligand that enables ion-exchange reversed phase and also π-π (*aromatic*) interactions. Silia*Chrom* SCX is used for specific applications including organic bases such as basic amino acids, anilines, drug salts, inorganic cations and nucleosides analysis.

Structure



R = $(CH_2)_2\ C_6H_4\ SO_3\text{-}H^+$

Silia*Chrom* SCX

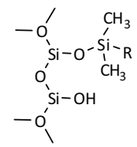
Sorbent Characteristics

- **Pore Size: 100 Å**
- **Specific Surface Area: 380 m²/g**
- **Particle Sizes Available: 3, 5 and 10 µm**
- **Typical Carbon Loading: SiliaChrom SCX 10% SiliaChrom SAX 6%**
- **pH Stability: 2.0 - 8.5**

Description

Silia*Chrom* SAX provides excellent resolution and peak shape for anionic analytes. Silia*Chrom* SAX is used for specific applications including pesticides, herbicides, pharmaceuticals, inorganic anions and biological species such as nucleotides and glucosinolates analysis.

Structure



R = $(CH_2)_3N^+(CH_3)_3Cl^-$

Silia*Chrom* SAX

Silia*Chrom* SCX and SAX Main Characteristics

- **Narrow peak shape**
- **Rapid equilibration**
- **Compatible with organic modifiers**
- **Provides high efficiency and rapid separations**
- **Endcapped**

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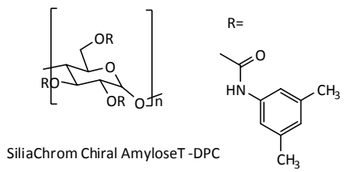
Silia*Chrom* Chiral Phases

Silia*Chrom* chiral coated polysaccharide stationary phases are made with a spherical high quality silica support physically coated with a polymeric chiral selector such as amylose or cellulose derivatives. Due to the coated nature of these supports, solvents should be carefully selected for normal phase conditions.

Description

Silia*Chrom* Chiral Amylose T-DPC:
Amylose tris-(3,5-dimethylphenylcarbamate) coated on a spherical silica support (*USP Code L51*). Silia*Chrom* Chiral Amylose T-DPC is used for chiral separation of alkaloids, tropines, amines and beta blockers.

Structure



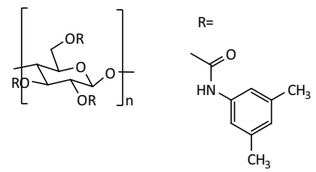
SiliaChrom Chiral AmyloseT -DPC

Silia*Chrom* Chiral Amylose T-DPC

Description

Silia*Chrom* Chiral Cellulose T-DPC:
Cellulose tris-(3,5-dimethylphenylcarbamate) coated on a spherical silica support (*USP L40*). Silia*Chrom* Chiral Cellulose T-DPC is the most popular phase for chiral separation of alkaloids, tropines, amines and beta blockers.

Structure

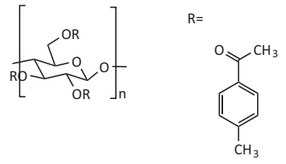


Silia*Chrom* Chiral Cellulose T-DPC

Description

Silia*Chrom* Chiral Cellulose T-MB:
Cellulose tris-(4-methylbenzoate) coated on a spherical silica support. Silia*Chrom* Chiral Cellulose T-MB is used for chiral separation of aryl methyl esters and aryl methoxy esters.

Structure

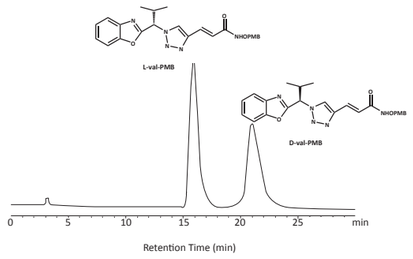


Silia*Chrom* Chiral Cellulose T-MB

Silia*Chrom* Chiral Amylose T-DPC Enantiomeric separation of L and D-val PMB

Chromatographic conditions

- **Column**: Silia*Chrom* Chiral Amylose T-DPC, 5 µm
- **Column size**: 4.6 x 250 mm
  SiliCycle P/N: H81005T-N250
- **Mobile phase**: Hexane/Isopropanol (*80/20*)
- **Flow rate**: 1.000 mL/min
- **Detector**: UV at 254 nm



Other Silia*Chrom* Products

Apart from the classic stationary phases, SiliCycle has also developped specific HPLC columns based on a silica matrix as our mixed-mode or phase-exclusion GF HPLC columns. To satisfy all HPLC needs, SiliCycle has polymer stationary phases in reversed phase applications (*RPC columns*) and ionic exchange HPLC applications (*IEC columns*).

Mixed-Mode Silia*Chrom*

Conjugate two surface function chemistries to optimize your separation in a single experiment. SiliCycle offers the following Silia*Chrom* Mixed-Mode HPLC columns:

- Silia*Chrom* C18/C8
- Silia*Chrom* C18/Amide
- Silia*Chrom* C18/Phenyl
- Silia*Chrom* C18/CN
- Silia*Chrom* C18/SCX
- Silia*Chrom* C18/SAX
- Silia*Chrom* C18/Nitrophenyl

Polymer-based Silia*Chrom* IEC

Silia*Chrom* IEC series are composed of polystyrene polymer-based packing bearing different functionalities such as weak or strong cationic and anionic functions. Silia*Chrom* IEC phases are compatible with most mobile phases and samples with a pH range from 1 to 14. Polymer-based columns tend to have lower efficiencies for small molecules compared to silica-based columns due to their smaller surface area. Nevertheless, Silia*Chrom* IEC packing is a good alternative for samples that require a mobile phase pH outside the normal operating range of standard silica-based columns. Silia*Chrom* IEC columns are generally used for ion-exchange separations, and are also useful for non-aqueous gel permeation chromatography size exclusion analyses and ion exclusion analyses of organic acids and carbohydrates.

This family is composed of 4 stationary phases;

- Silia*Chrom* IEC WA: Weak anion exchanger
- Silia*Chrom* IEC SA: Strong anion exchanger
- Silia*Chrom* IEC WC: Weak cation exchanger
- Silia*Chrom* IEC SC: Strong cation exchanger

Polymer-based Silia*Chrom* RPC:

Silia*Chrom* RPC phase is a hydrophobic copolymer based on polystyrene and divinylbenzene. The macroporous RPC reversed phase resins are available in different particle sizes within a very narrow size distribution. The chemically inert polymer matrix of the Silia*Chrom* RPC guarantees chemical stability and allows for use with applications in the range of pH 1 to 14. The K' values measured for aromatic and conjugated molecules on RPC columns are high due to the very pure uniform hydrophobic surface. The high efficiency and high selectivity of Silia*Chrom* RPC columns allow the separation of analytes in minutes. Even basic substances are separated efficiently without any peak tailing.

Silica-based Silia*Chrom* GF:

Size exclusion chromatography (*SEC*) also known as gel permeation chromatography (*GPC*) or gel filtration chromatography, separates molecules according to their size (*or, more accurately, according to their hydrodynamic diameter or hydrodynamic volume*). Smaller molecules are able to enter the pores of the media and, are therefore trapped and removed from the flow of the mobile phase. The average residence time in the pores depends upon the effective size of the analyte and the pore size itself. Larger molecules are excluded with essentially no retention. Silia*Chrom* GF column series are an appropriate set of phases to be used for size exclusion chromatography with silica-based material in normal phase conditions.

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Contact Us

Order Now

Ordering Information

Quote Form

General Information

Company: .....

SiliCycle Customer Number (*if known*): .....

Contact Name: .....

E-mail address: .....

Tel: ..... Fax: .....

Address Information .....

.....

Department: .....

Address: .....

ZIP/Postal Code: .....

Country: .....

Quote Request

Part Number	Product Description	Request Quantity

Confirm this request by

- ☐ FAX
- ☐ E-mail
- ☐ Phone

Comments

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Information Request Form

☐ **Metal Scavengers**

Metal of interest:

.....

.....

.....

☐ **Catalysts**

- ☐ Silia*Cat* catalysts
- ☐ Silia*Bond* catalysts
- ☐ Silia*Cat* + Silia*Bond* catalysts

☐ **Oxidants**

- ☐ Silia*Cat* oxidant
- ☐ Silia*Bond* oxidants
- ☐ Silia*Cat* + Silia*Bond* oxidants

☐ **Organic scavengers**

- ☐ Nucleophile scavengers
- ☐ Electrophile scavengers
- ☐ Genotoxic scavengers

☐ **SiliaBond Reagents**

- ☐ Reaction of interest
- ☐ .....
- ☐ .....
- ☐ .....

☐ **Others**

- ☐ .....
- ☐ .....
- ☐ .....
- ☐ .....

☐ **Chromatographic phases**

- ☐ Reversed phases
- ☐ Normal phases
- ☐ Fluorous phases
- ☐ Ion exchangers

☐ **HPLC Columns**

- ☐ Reversed phases
- ☐ Normal phases
- ☐ Ion exchangers
- ☐ Chiral phases

☐ **Application notes in**

- ☐ Bulk
- ☐ SPE cartridges
- ☐ Flash cartridges

General Information

Company: .....

SiliCycle Customer Number (*if known*): .....

Contact Name: .....

E-mail address: .....

Tel: ..... Fax: .....

Address Information .....

.....

Department: .....

Address: .....

ZIP/Postal Code: .....

Country: .....

Comments

.....

..... Please Copy and Fax this form to SiliCycle Inc. at **418-874-0355**

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Order Form

General Information

Company: .....

SiliCycle Client Number: ..... Purchase Order Number: .....

Contact Name: .....

E-mail address: .....

Tel: Fax: .....

Payment Details

Credit card number: .....

Expiry date: .....

Name as it appears on card: .....

Verification Code*

*The 3 last numbers in the back side of your card

VISA ☐ Master Card ☐ Amex ☐

Address Information for the Shipment

Department: .....

Address: .....

.....

ZIP/Postal Code: .....

Country: .....

SiliCycle Products

Part Number	Product Description	Packaging (g, Kg, box)	Quantity	Price (USD)

Confirm this request by

☐ FAX

☐ E-mail

☐ Phone

Comments

.....

..... Please Copy and Fax this form to SiliCycle Inc. at **418-874-0355**

SILICYCLE inc. UltraPure SILICA GELS SiliCycle®

Ordering Information

How to order

You can order any SiliCycle product on-line through the new SiliCycle e-commerce website: www.silicycle.com.

You can also order any product from this catalog, 365 days or nights a year. We hope you will enjoy this new and friendly way to order SiliCycle products.

Orders can also be placed by phone, fax, mail or e-mail. You will find an order form on page 222 of this catalog for fax (*1 418.874.0355*) and mail (*SiliCycle headquarters address at the bottom of this page)* orders. If you prefer, you can reach us by e-mail (*info@silicycle.com*) or by phone (*1 418.874.0054 or Toll free for North America only 1 877.745.4292*). Please have the following information on hand:

- Your name
- Company name, billing and shipping address
- Purchase order number
- Credit card information
- Catalog number and product description
- Size, quantity and unit of measure
- E.I.N. or F.I.N .for United States clients

Technical Support

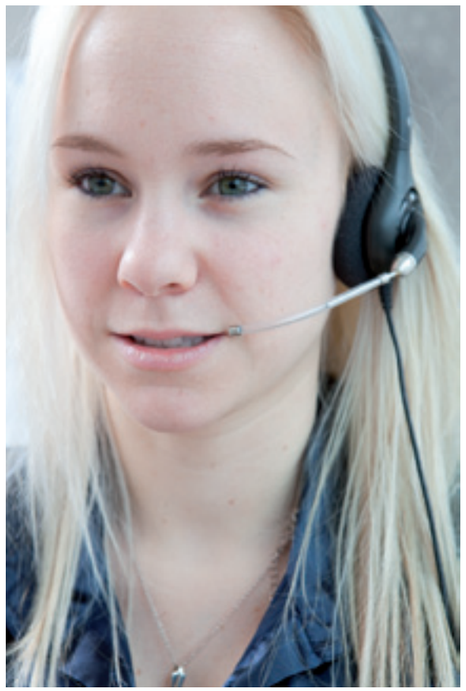
At SiliCycle, we are committed to providing the best technical support possible. Our worldwide Technical Support Group is comprised of a team of highly qualified M.Sc., Engineers and PhD Chemists, Technical Support Professionals and Service Coordinators who are prepared to troubleshoot, answer questions, and provide solutions for your service and applications needs.

In order to better respond to your technical inquiries, feel free to contact us in three different ways:

- E-mail: support@silicycle.com
- Phone: International 1 418.874.0054
- Canada USA 1 877.745.4292 (Toll-Free)
- Online forum at www.SiliCycle.com

SiliCycle headquarters address:

2500, Parc-Technologique Blvd
Quebec City, Quebec
G1P 4S6, CANADA



Terms and Conditions

General

Unless otherwise stated, all transactions are expressly subject to these Terms and Conditions. Modifications or additions will be recognized only if accepted in writing by an officer of SiliCycle Inc. (*hereinafter named SiliCycle*), or an officially designated representative. Provisions of Buyer's Purchase Order or other documents that add to or differ from these Terms and Conditions are expressly rejected. No waiver of these Terms and Conditions or acceptance of others shall be construed as failure of the Company to raise objections.

Privacy Policy

Because your clientele is our most vital asset, we take privacy very seriously and won't share your personal information with anyone. Your information is used only to personalize your profile and to facilitate the transaction. You can change or update your information at any time.

Quotation and Published Prices

Quotations automatically expire 30 calendar days from the date issued unless otherwise stated. Quotes are subject to withdrawal with notice within that period. Prices shown on the published price lists and other published literature issued by SiliCycle are not unconditional offers to sell, and are subject to change without notice.

Warranty

SiliCycle guarantees to the original Buyer that the products sold conform to the composition and purity described therein at the time of their shipment. The Buyer's sole remedy in the event that SiliCycle fails to meet said warranty shall be the replacement of the unused portion of the product(*s*), or if approved by SiliCycle, a refund (*at the purchase price*) provided that the Buyer returns the alleged non-conforming product(*s*) within 30 days after reception of product(*s*). SiliCycle makes no other guarantee 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.

Changes

The Buyer may, with the express written consent of SiliCycle, make changes in the specifications for products or work covered by the contract. In such an event, the contract price and delivery dates shall be equitably adjusted. SiliCycle shall be entitled to payment for reasonable profit plus costs and expenses incurred by work and materials rendered unnecessary as a result of such changes and for work and materials required to effect said changes.

If the Buyer has made a mistake on his/her purchase order, and the material has already been shipped and received, SiliCycle may approve the exchange of said material (*if price is identical*); however the Buyer will be responsible for all shipping costs. See return authorization policy section on the next page to obtain a return merchandize authorization form prior to returning goods.

Cancellation

Undelivered parts of any order may be cancelled by the Buyer only with the written approval of SiliCycle. If the Buyer makes an assignment for the benefit of creditors, or in the event that SiliCycle, for any reason feels insecure about Buyer's willingness or ability to perform, SiliCycle shall have the unconditional right to cancel the sales transaction or demand full or partial payment.

In the event of any cancellation of this order by either party, the Buyer shall pay SiliCycle for reasonable costs and expenses incurred by the SiliCycle prior to receipt of the cancellation notice, plus SiliCycle's usual rate of profit for similar work.

Taxes

The Company's prices do not include any applicable sales, goods and services, use, excise or similar taxes and the amount of any such tax SiliCycle may be required to pay or collect will be added to each invoice and paid by the Buyer.

Terms of Payment

All merchandise purchased remains the property of SiliCycle until such time as all invoices for the merchandise have been paid in full. Except for purchases paid online, or unless explicitly stated elsewhere in writing, terms are cash net 30 days from date of invoice. Additional fees of 2% per month (26.8% per year) will accrue on all accounts past due. If any payment is in default, and it becomes necessary to hire a recovery agency or lawyer, the client accepts to pay, in addition to the outstanding balance, recovery fees equal to 20% of the balance in capital and interests. By reason of the financial condition of Buyer or otherwise, SiliCycle may require full or partial payment in advance.

Certain orders may require a deposit or progressive payments as referenced in the quote. Such deposits may be increased upon receipt of purchase order based upon the Buyer's most current credit rating. Subject to the warranties stated in this policy, all sales are final without right of return.

Return Policy

Our Customer Service Department is available to assist you at any time should a problem arise with your order. Please make sure to inspect your packages immediately upon receipt and notify us within the next two (2) business days of any damage and/or discrepancies. Should a product be sent to you incorrectly, as the result of an error on our part, we will take quick and appropriate action to correct the problem at no charge to you.

In order to maintain the quality of our products and continue to provide competitive prices, some products may not be returned for credit. SiliCycle will not grant credit for:

(i) Shelf-worn, used or defaced products;
(ii) Scavengers, reagents, catalysts, or any other bounded silica whose containers have been opened;
(iii) Products that are personalized or customized;
(iv) Refrigerated or temperature-controlled products;
(v) Products that have been discontinued;
(vi) Products not directly purchased from SiliCycle

Products sold in distribution by SiliCycle will be subject to the Terms and Conditions Policy of the respective manufacturer.

Prior to any return, an authorization and a return material authorization (RMA) number must be obtained from our Customer Service Department. Shipping instructions will also be provided at this point. The RMA will ensure the safe and proper handling of material; it should therefore be referenced on all shipping labels.

The Buyer has 30 days from the issuance of the RMA to return the goods. Returns made without an authorization number will not be accepted and will be returned to the Buyer.

Returns are subject to a 50% restocking and/or disposal fee.

Shipping Policy

SiliCycle uses a two-day or five-day delivery (or equivalent) depending on weight and availability of product. Standard overnight delivery can also be arranged. Freight charges are prepaid and added to the invoice unless special instructions are requested by the customer. These conditions apply to all North American shipments. International delivery delays will vary according to orders and destination countries.

Delivery

Delivery dates indicated in the contract documents are approximate and based on prompt receipt of all necessary information regarding the product covered by the contract. SiliCycle will use reasonable efforts to meet the indicated delivery dates, but cannot be held responsible for its failure to do so.

In the event of any delivery delay caused by the Buyer, SiliCycle will store and handle all items ordered at Buyer's risk and will invoice Buyer for the unpaid portion of the contract price, plus storage, insurance, and handling charges on or after the date on which the product is ready for delivery. The invoice will be payable in full within 30 days from the invoice date, unless otherwise expressly agreed to in writing by SiliCycle.

SiliCycle will not hold orders unless specifically approved. SiliCycle has the right to make partial shipments and bill for those shipments; the buyer will make payment in accordance with the terms mentioned in this policy.

Shipping and Handling Charges

Shipping charges plus the applicable company handling charges will be prepaid and billed as a separate item on the product invoice. Title to the product and risk of loss shall pass to Buyer upon delivery to a carrier.

Application

All products are sold for laboratory or manufacturing uses. Only professional laboratory staff should handle the chemicals.

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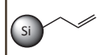
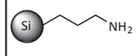
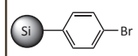
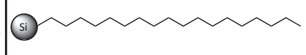
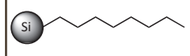
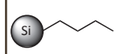
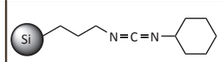
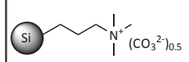
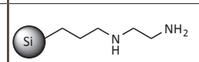
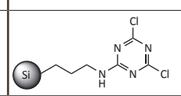
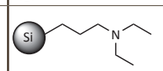
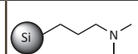
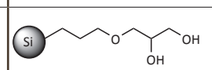
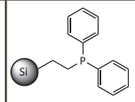
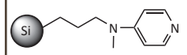
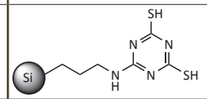
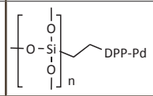
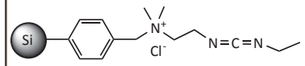
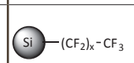
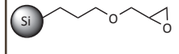
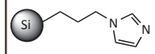
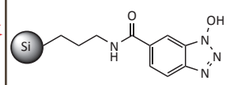
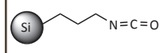
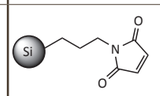
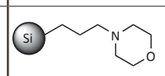
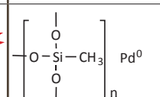
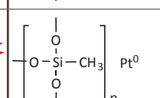
Silia*Bond* & Silia*Cat* Listing

Category Listing

Product (Number)	Structure	Function	Characteristics
Silia*Bond* Allyl (***Si*-Allyl**) R53530B		Solid Linker	Loading: 1.2 mmol/g Endcapping: yes Density: 0.613 g/mL
Silia*Bond* Aluminium Chloride (***Si*-AlCl$_x$**) R74530B		Catalyst & Reagent	Loading: 1.6 mmol/g Endcapping: no
Silia*Bond* Amine (***Si*-WAX or *Si*-NH$_2$**) R52030B		Base, Metal Scavenger Chromatographic Phase Ion Exchange Phase	Loading: 1.6 mmol/g Endcapping: yes Density: 0.700 g/mL
Silia*Bond* Bromophenyl (***Si*-BRP**) R55030B		Linker	Loading: 1.6 mmol/g Endcapping: yes Density: 0.742 g/mL
Silia*Bond* C18 R30030B, R30130B, R33230B, R33330B...		Chromatographic Phase	Loading: 11 to 23 %C Endcapping: yes & no
Silia*Bond* C12 R53030B		Chromatographic Phase	Loading: 16 %C Endcapping: yes Density: 0.665 g/mL
Silia*Bond* C8 R31030B & R31130B		Chromatographic Phase	Loading: 12 %C Endcapping: yes & no Density: 0.759 g/mL
Silia*Bond* C4 R32030B & R32130B		Chromatographic Phase	Loading: 8 %C Endcapping: yes & no Density: 0.656 g/mL
Silia*Bond* C1 R33030B		Chromatographic Phase	Loading: 5 %C Endcapping: yes Density: 0.599 g/mL
Silia*Bond* Carbodiimide (***Si*-DCC**) R70530B		Reagent	Loading: 0.9 mmol/g Endcapping: yes Density: 0.751 g/mL
Silia*Bond* Carbonate (***Si*-CO$_3$**) R66030B		Base Organic Scavenger	Loading: 0.7 mmol/g Endcapping: no Density: 0.608 g/mL
Silia*MetS* Diamine (***Si*-DIA**) R49030B		Metal Scavenger Base Ion Exchange Phase	Loading: 1.4 mmol/g Endcapping: yes Density: 0.728 g/mL
Silia*Bond* Dichlorotriazine (***Si*-DCT**) R52230B		Reagent	Loading: 0.7 mmol/g Endcapping: yes Density: 0.781 g/mL
Silia*Bond* Diethylamine (***Si*-WAX-2**) R76530B		Base Ion Exchange Phase	Loading: 1.2 mmol/g Endcapping: yes Density: 0.685 g/mL
Silia*Bond* Dimethylamine R45030B		Base	Loading: 1.4 mmol/g Endcapping: yes Density: 0.762 g/mL
Silia*Bond* Diol R35030B		Chromatographic Phase Organic Scavenger	Loading: 1.0 mmol/g Endcapping: no Density: 0.688 g/mL

Category Listing

Product (Number)	Structure	Function	Characteristics
Silia*Bond* Diphenylphosphine (***Si*-DPP**) R39030B		Reagent	Loading: 0.9 mmol/g Endcapping: yes Density: 0.692 g/mL
Silia*Bond* DMAP (***Si*-DMAP**) R75530B		Catalyst & Reagent	Loading: 0.8 mmol/g Endcapping: yes Density: 0.674 g/mL
Silia*MetS* DMT R79030B		Metal Scavenger	Loading: 0.5 mmol/g Endcapping: yes Density: 0.732 g/mL
Silia*Cat* DPP-Pd R390-100		Catalyst	Loading: > 0.2 mmol/g Endcapping: yes Density: 0.415 g/mL
Silia*Bond* EDC R70630B		Reagent	Loading: 0.8 mmol/g Endcapping: yes Density: 0.770 g/mL
Silia*Bond* Fluorochrom (***Si*-FCM**) R63730B		Fluorous Phase	Loading: 7 % Carbon Endcapping: yes Density: 0.738 g/mL
Silia*Bond* Glycidoxy (***Si*-GLY**) R36030B		Linker	Loading: 1.1 mmol/g Endcapping: no Density: 0.662 g/mL
Silia*MetS* Imidazole (***Si*-IMI**) R79230B		Base Metal Scavenger	Loading: 0.9 mmol/g Endcapping: no Density: 0.681 g/mL
Silia*Bond* HOBt R70730B NEW PRODUCT		Reagent	Loading: 0.8 mmol/g Endcapping: yes Density: TBD
Silia*Bond* Isocyanate (***Si*-ISO**) R50030B		Nucleophile Scavenger	Loading: 1.2 mmol/g Endcapping: yes Density: 0.741 g/mL
Silia*Bond* Maleimide (***Si*-MAL**) R71030B		Organic Scavenger	Loading: 0.7 mmol/g Endcapping: yes
Silia*Bond* Morpholine (***Si*-MOR**) R68030B		Base	Loading: 1.1 mmol/g Endcapping: yes Density: 0.666 g/mL
Silia*Cat* Pd^0 R815-100 NEW PRODUCT		Catalyst	N/A
Silia*Cat* Pt^0 R820-100 NEW PRODUCT		Catalyst	N/A

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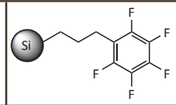
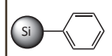
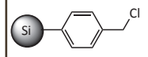
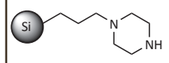
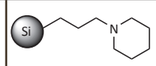
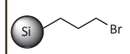
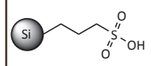
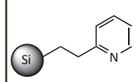
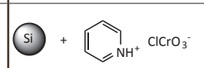
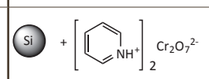
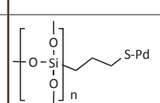
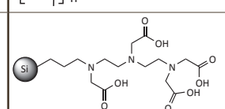
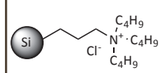
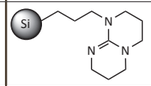
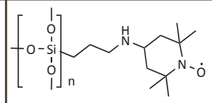
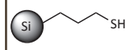
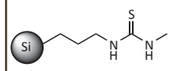
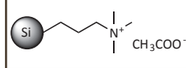
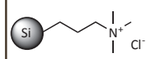
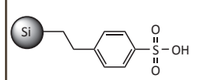
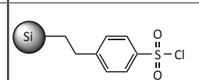
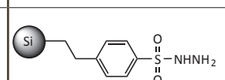
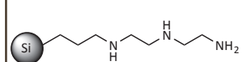
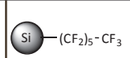
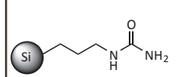
Silia*Bond* & Silia*Cat* Listing (con't)

Category Listing

Product (Number)	Structure	Function	Characteristics
Silia*Bond* Pentafluorophenyl (*Si*-PFP) R67530B		Fluorous Phase	Loading: 0.8 mmol/g Endcapping: yes Density: 0.666 g/mL
Silia*Bond* Phenyl (*Si*-PHE) R34030B		Chromatographic Phase	Loading: 1.2 mmol/g Endcapping: yes Density: 0.637 g/mL
Silia*Bond* Phenylmethylchloride R56530B		Linker	Loading: 0.5 mmol/g Endcapping: yes Density: 0.637 g/mL
Silia*Bond* Piperazine (*Si*-PPZ) R60030B		Base	Loading: 0.8 mmol/g Endcapping: yes Density: 0.671 g/mL
Silia*Bond* Piperidine (*Si*-PIP) R71530B		Base	Loading: 1.1 mmol/g Endcapping: yes Density: 0.660 g/mL
Silia*Bond* Potassium Permanganate R23030B		Oxidant	Loading: 10 % w/w Endcapping: no Density: 0.593 g/mL
Silia*Bond* Propyl Bromide (*Si*-PBR) R55530B		Linker	Loading: 1.5 mmol/g Endcapping: yes Density: 0.748 g/mL
Silia*Bond* Propylsulfonic Acid (*Si*-SCX-2) R51230B		Acid, Reagent Ion Exchange Phase Nucleophile Scavenger	Loading: 1.0 mmol/g Endcapping: yes Density: 0.728 g/mL
Silia*Bond* Pyridine (*Si*-PYR) R43030B		Base	Loading: 1.3 mmol/g Endcapping: yes Density: 0.727 g/mL
Silia*Bond* Pyridinium Chlorochromate (PCC) R24030B		Oxidant	Loading: 20 % w/w Endcapping: no Density: 0.693 g/mL
Silia*Bond* Pyridinium Dichromate (PDC) R24530B		Oxidant	Loading: 20 % w/w Endcapping: no Density: 0.651 g/mL
Silia*Bond* Silver Nitrate (*Si*-$AgNO_3$) R23530B		Chromatographic Phase	Loading: 10 % w/w Endcapping: no Density: 0.651 g/mL
SiliaCat S-Pd R510-100		Catalyst	Loading: >0.3 mmol/g Endcapping: yes Density: 0.550 g/mL
Silia*MetS* TAAcOH R69030B		Acid Metal Scavenger	Loading: 0.4 mmol/g Endcapping: yes Density: 0.632 g/mL
Silia*MetS* TAAcONa R69230B	*Same as TAAcOH but with Na*	Metal Scavenger	Loading: 0.4 mmol/g Endcapping: yes Density: 0.712 g/mL

Category Listing

Product (Number)	Structure	Function	Characteristics
Silia*Bond* TBA Chloride (*Si*-TBACl) R65530B		Ion Exchanger Phase	Loading: 0.5 mmol/g Endcapping: no Density: 0.751 g/mL
Silia*Bond* TBD R68530B		Base Metal Scavenger Reagent	Loading: 0.9 mmol/g Endcapping: yes Density: 0.730 g/mL
Silia*Cat* TEMPO R723-100		Catalyst	Loading: 0.7 mmol/g Endcapping: yes Density: 0.639 g/mL
Silia*MetS* Thiol R51030B		Metal Scavenger	Loading: 1.2 mmol/g Endcapping: yes Density: 0.682 g/mL
Silia*MetS* Thiourea (*Si*-THU) R69530B		Metal Scavenger	Loading: 1.2 mmol/g Endcapping: yes Density: 0.767 g/mL
Silia*Bond* TMA Acetate (*Si*-SAX-2) R66430B		Ion Exchange Phase	Loading: 1.0 mmol/g Endcapping: no Density: 0.665 g/mL
Silia*Bond* TMA Chloride (*Si*-SAX) R66530B		Ion Exchange Phase	Loading: 1.1 mmol/g Endcapping: no Density: 0.751 g/mL
Silia*Bond* Tosic Acid (*Si*-SCX) R60530B		Acid, Reagent Nucleophile Scavenger Ion Exchange Phase	Loading: 0.8 mmol/g Endcapping: yes Density: 0.743 g/mL
Silia*Bond* Tosyl Chloride (*Si*-TsCl) R44030B		Nucleophile Scavenger	Loading: 1.0 mmol/g Endcapping: yes Density: 0.761 g/mL
Silia*Bond* Tosyl Hydrazine (*Si*-$TsNHNH_2$) R61030B		Electrophile Scavenger	Loading: 1.5 mmol/g Endcapping: yes
Silia*MetS* Triamine (*Si*-TRI) R48030B		Base Metal Scavenger	Loading: 1.2 mmol/g Endcapping: yes Density: 0.736 g/mL
Silia*Bond* Tridecafluoro (*Si*-TDF) R63530B		Fluorous Phase	Loading: 0.5 mmol/g Endcapping: yes Density: 0.842 g/mL
Silia*Bond* Urea R67030B		Scavenger Chromatographic Phase	Loading: 1.3 mmol/g Endcapping: yes Density: 0.695 g/mL

SiliCycle Products and Mettler-Toledo MiniBlock®

An Ideal Partnership in North America

- The productivity enhancement of MiniBlock combined with the cutting-edge technology available from SiliCycle enable chemists to design reactions that eliminate tedious work-up and purification issues.
- The MiniBlock is compatible with the full range of SiliCycle products from the synthesis through the purification.
- All SiliCycle silicas (i.e.: Silia*MetS* Metal Scavengers, Silia*Cat* Heterogeneous Catalysts, and Silia*Bond* Functionalized silica gels) are available in MiniBlock prepacked SPE cartridges.

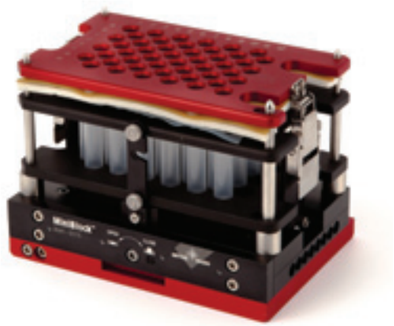
MiniBlock

The MiniBlock is an easy to use reaction block designed for parallel synthesis and screening. The unique valve body design of the MiniBlock enables processes where filtration is critical, including solid-phase organic synthesis, use of scavenger resins with solution phase synthesis and parallel purification via Solid Phase Extraction (SPE).

MiniBlock Reactors

Patented reactor with built-in value design. Available in 48, 24, 12, and 6-position arrays for reaction vessel volumes respectively of 4mL, 10mL, 20mL and 40mL.

13742044	MiniBlock Reactor Blue	48-position
13742043	MiniBlock Reactor Red	48-position
13742200	MiniBlock Reactor Blue	24-position
13742201	MiniBlock Reactor Red	24-position
13742210	MiniBlock Reactor Blue	12-position
13742211	MiniBlock Reactor Red	12-position
13742220	MiniBlock Reactor Blue	6-position
13742221	MiniBlock Reactor Red	6-position



Shaking and Washing Station

High performance orbital shaker with integrated basins for wash and rinse capability. Customized and configured to provide vigorous vortex mixing for up to 2 (compact) and 6 (high capacity) MiniBlocks.

13742071	Compact Shaking and Washing Station, 115V
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SiliCycle Products and MiniBlock: Great Compatibility

Catalysis using Silia*Cat*

- Suzuki Coupling (p. 25)
- Heck Coupling (p. 28)
- Sonogashira Coupling (p. 30)
- Stille Coupling (p. 32)
- Selective Hydrogenation (p. 36)
- Selective Debenzylation (p.38)
- Hydrosilylation Coupling (p.41)
- Oxidation (p.42)

Synthesis using Silia*Bond*

- Amide Coupling (p. 52)
- Baylis-Hillman & Acylations (p. 63)
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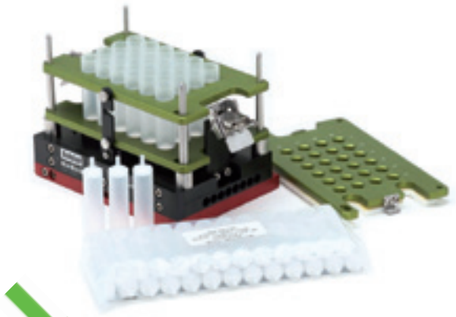
- Williamson Etherification (p. 68)

SiliCycle Products + MiniBlock: An Ideal Partnership



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Analysis

- Silia*Prep* SPE Sorbents (p. 171)

All SiliCycle Silia*Bond* & Silia*Cat* can be used with the MiniBlock and are available in prepacked SPE cartridges compatible with this system. Contact us for more details.

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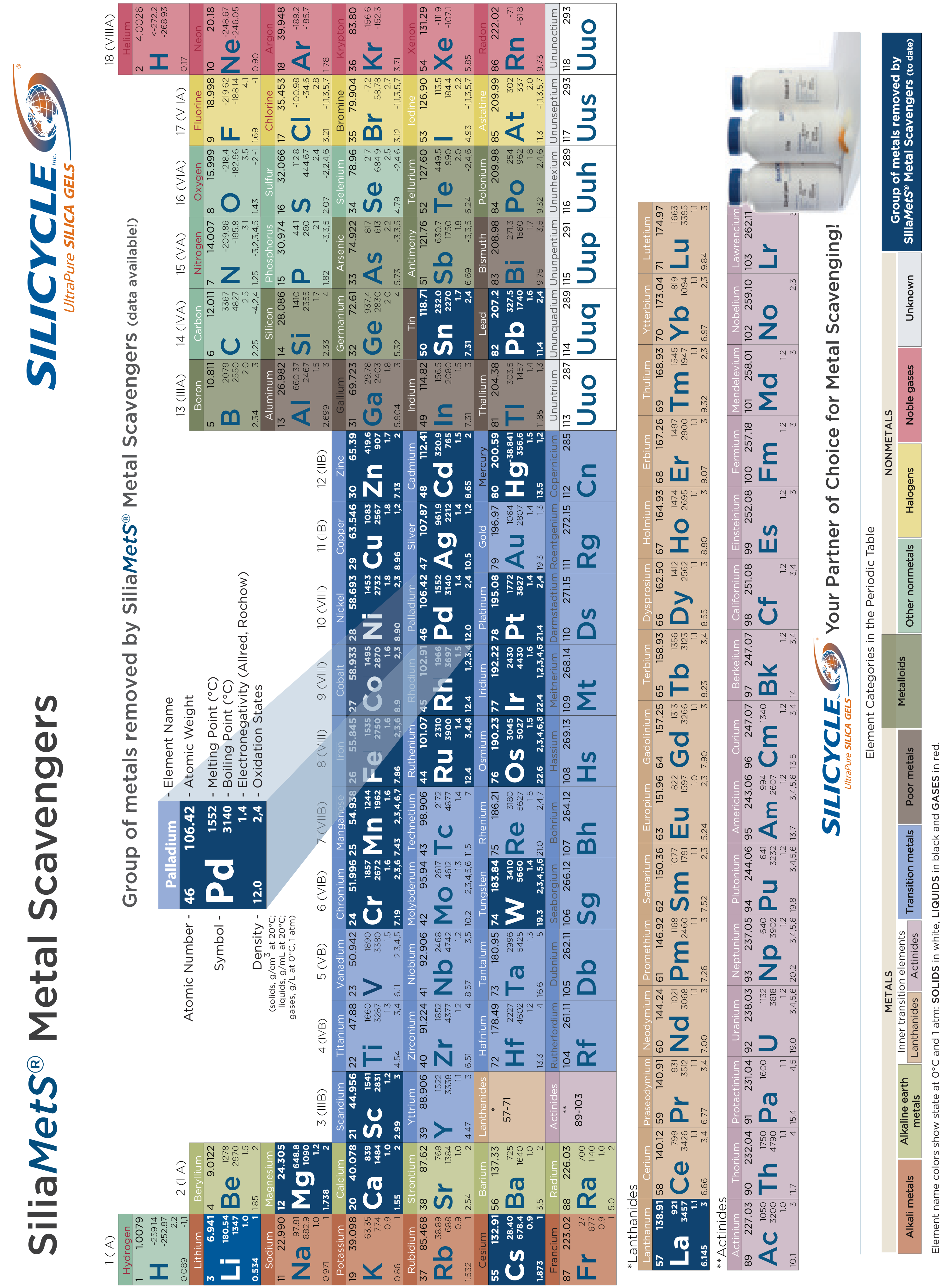

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