SOLUTIONS FOR CHROMATOGRAPHY AND PURIFICATION

SILICYCLE

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

Solutions for Chromatography and Purification

Bulk Silicas for Chromatography

SiliCycle is your partner of choice for your chromatography and purification needs! **Recognized as one of the leaders with an excellent quality silica gel, SiliCycle offers a wide range of products available in two different shapes:**

- **Silia*Flash*® Irregular silicas**
- **SiliaSphere™ PC Spherical silicas**

SiliCycle: Silica Expert

With pore diameters ranging from 30 to 1,000 Ångström (*Å*) and particle sizes up to 1,200 microns (*µm*), SiliCycle offers products to meet all your requirements. We offer one of the most reliable portfolios for flash and gravity grades for low to medium-high pressure. Our silica gels are ideal for preparative chromatography, from laboratory to pilot-plant processes and production scale.

Features and Benefits of Silia*Flash* & Silia*Sphere PC*

Features	Benefits
High purity silica gels	No contamination, consistency, reliability, reproducibility
Low level of fines	No contamination, lower back-pressure, good separation
Narrow particle and pore size distributions	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 and controlled water content	Symmetrical peaks without 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	In stock for fast delivery

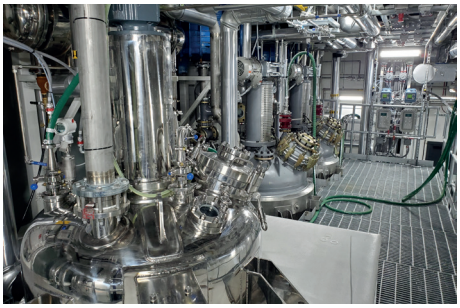
Together, all these benefits mean optimal and reproducible separation power, saving you time and money.

SiliCycle, the Silica Supplier for Every Need

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

Be confident in scaling-up your processes with our silica gels.

With SiliCycle, scale-up from laboratory to production without limitations!



Enjoy a virtual tour of SiliCycle's facility

Two Shapes Available: Irregular and Spherical

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 confident about your recoveries.

In chromatography, there are at least three physical properties that will influence your separation and that you need to consider when choosing your silica gel:

- **Particle shape** (*irregular or spherical*)
- **Particle size** distribution (*tight or large*)
- **Pore diameter** (*surface area*)

These caracteristics will directly influence crucial parameters involved in a successful chromatography:

- **Resolution** (*efficiency of separation and final purity*)
- **Retention** (*which allows separation*)
- **Capacity** (*maximal sample quantity and final recovery / yield*)
- **Back-pressure** (*speed and pumps related issues*)

At SiliCycle, we ensure consistency, reliability and reproducibility.

Our expertise and strong knowledge has been developed over many years of helping our customers find the best solutions to their particular needs.

How to Choose Between Silia*Flash* Irregular and Silia*Sphere* PC Spherical Gels?

Irregular silica gels are traditional in flash or gravity chromatography and have always been a spontaneous choice for preparative chromatography. Nowadays, spherical particles are used increasingly.

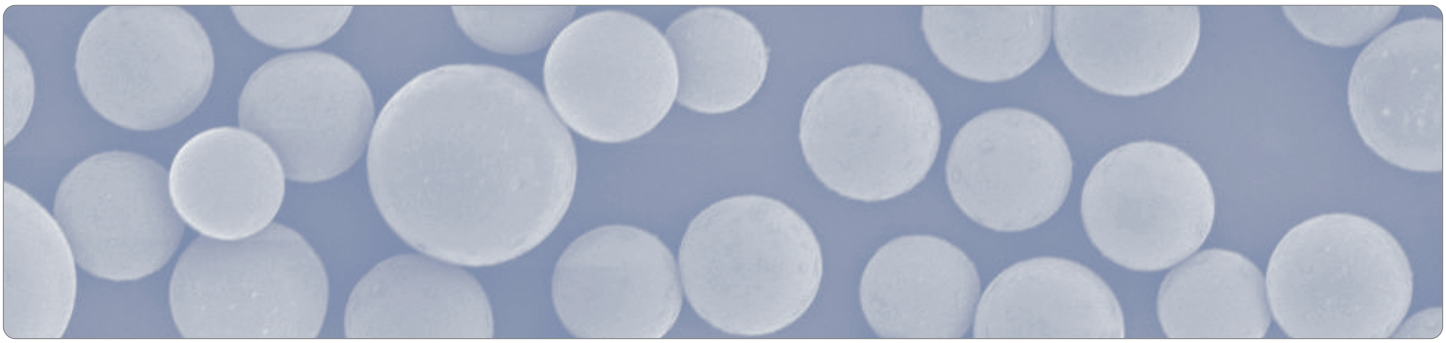
Cost is very important in preparative and process chromatography, and the use of monodisperse spherical particles with narrow particle size distribution is more expensive. It is possible in this case to use irregular silica but the separation may not provide the desired results. For these situations, SiliCycle has developed a more affordable class of spherical particles for preparative chromatography: Silia*Sphere* PC.

Advantages of using Silia*Sphere* PC materials over standard irregular silica gels include the following:

- Increased efficiency of the eluent's flow characteristics
- Higher resolution
- Ease of packing / better packing reproducibility
- Higher mechanical stability

Silia*Sphere* PC: Truly Spherical

Silica gel quality varies greatly between manufacturers. Even when advertised as being "spherical" this may not be the case. Please discover on next page a quick comparison of Scanning Electron Microscopy (*SEM*) pictures between SiliCycle Silia*Sphere* PC and the competition.

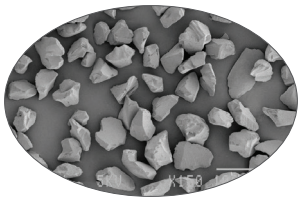


Silia*Flash* and Silia*Sphere* PC Characteristics

The importance of the particle and pore size distributions varies depending on the type of chromatography being done.

Importance of Tight Distributions in Chromatography	
Tight Particle Size Distribution	**Tight Pore Size Distribution**
Greater column performance and separation	Surface area (*Presence of bigger pore size leads to lower surface availability*)
Tighter peaks and better peak shape	Optimal peak shape (*Presence of smaller pore size leads to peak tailing*)
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 IRREGULAR silica gels 40 - 63 µm, 60 Å



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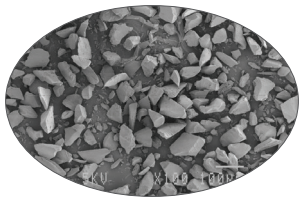
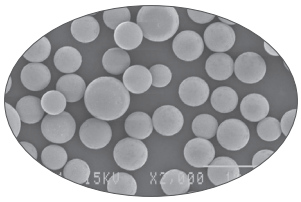
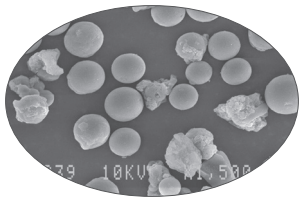


Competitor

Scanning Electron Microscopy (*SEM*) comparison of two SPHERICAL silica gels 50 µm, 60 Å



SiliCycle



Competitor

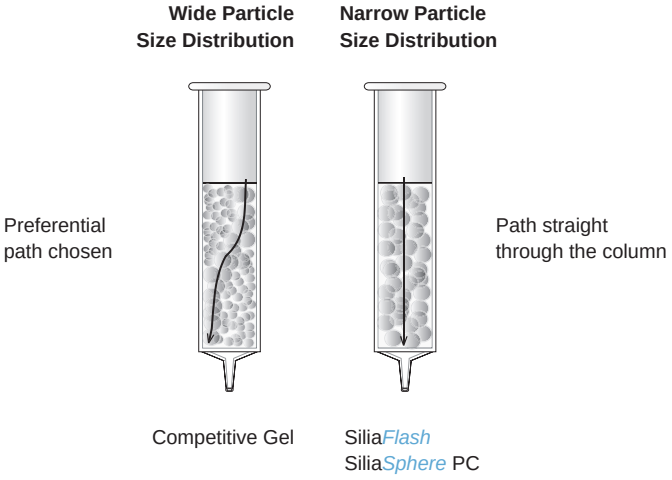
Effects of Homogeneous vs Uneven Packing

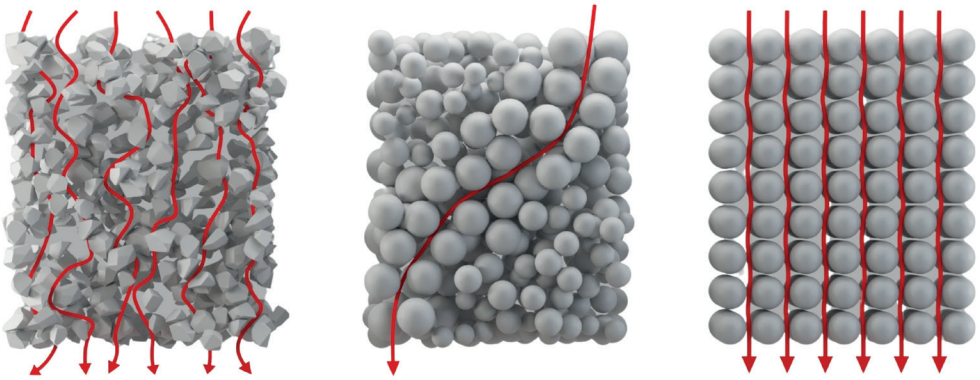
The connection between particle size distribution and column performance is very simple. When the distribution is broad, the packing is uneven. Some parts are composed of only large particles where the solvent will flow fast and meet little resistance, and there are sections composed of small particles 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 on the right illustrates the effect of a wide particle size distribution versus a narrow one. Narrower distribution gives a more homogenous packing and thus more concentrated fractions. And, by reducing solvent consumption, the process will be more cost-efficient.





Irregular flow

Preferential path

Homogeneous flow

High Purity Silica Gels

You can be sure of the outstanding quality of SiliCycle's silica gels because of the closely controlled manufacturing conditions. Our tight control of every manufacturing process step allows reproducible results (*chemical, physical and structural*) as well as ensuring the same chromatographic selectivity. Hence, Silia*Flash* and Silia*Sphere* PC are suitable for validated chromatographic processes.

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 (*COA*). Individual data sheets are also available directly from our website.

Stable Water Level Content

Water level of silica gel affects the selectivity of the silica. Silia*Flash* and Silia*Sphere* PC have generally a water content between 2 to 6 %. This is advantageous for you since other products have a water variation from 2 to 15 % depending on the manufacturer. SiliCycle can also adjust the water level upon request.

Neutral pH

Our silicas are pH-adjusted between 6 and 8 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 many gels on the market that are much more acidic.

Low Trace Metal Content

Silica, depending on its method of manufacturing, contains a certain amount of various 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. This ensures you will get optimal performance from your chromatography. Tight control of metals in every batch also improves your reproducibility and reduces risks of interaction between metals and desired compounds.

Typical Metal Content Comparison for 40 - 63 µm, 60 Å Silica Gels (*mg/kg*)

Metals		SiliCycle F60 R10030B	Manufacturer A	Manufacturer B
Aluminum	Al	33	262	280
Barium	Ba	9	60	33
Calcium	Ca	336	1,150	502
Chromium	Cr	0.5	0.6	0.4
Copper	Cu	0.2	0.2	0.2
Iron	Fe	32	75	41
Lead	Pb	0.41	5.3	0.95
Magnesium	Mg	61	149	104
Nickel	Ni	0.4	0.5	0.5
Silver	Ag	0.09	0.29	0.19
Sodium	Na	466	945	585
Tin	Sn	0.2	0.2	0.1
Titanium	Ti	147	250	179
Zirconium	Zr	32	75	56

Silia*Flash* Irregular Silica Gels

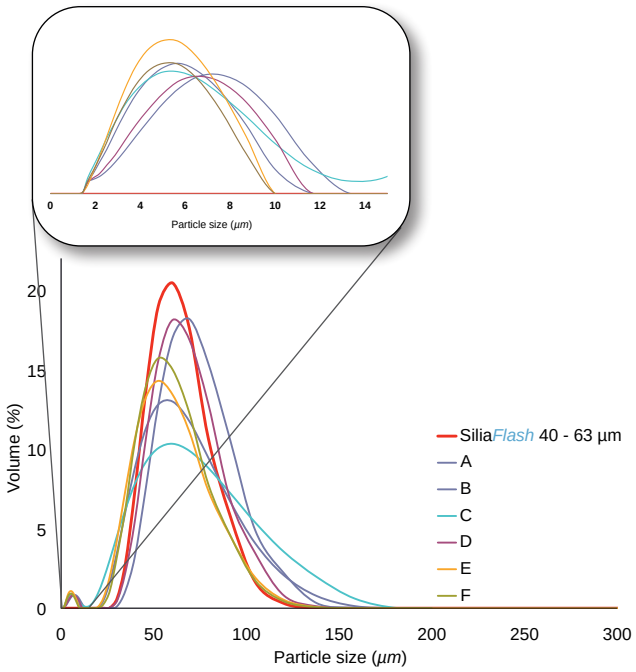
One of the Tightest Particle Size Distribution on the Market

Particle Size Distributions' Disparity

When selecting a silica gel, chemists need to take into account that not all 40 - 63 µm gels are the same.

In this example, the figure on the right shows the distribution curves of SiliCycle's Silia*Flash* gel (*PN: R10030B*) compared to other manufacturers of flash silica gels of same particle sizes. ***All products were sold as 40 - 63 µm 60 Å gels.***

As you can observe, SiliCycle's gel has a mean of 90 % of the particles in the nominal range compared to maximum 80 % for the competitor gels. The higher the curve, the tighter the particle size distribution.



Importance of the Absence of Fines

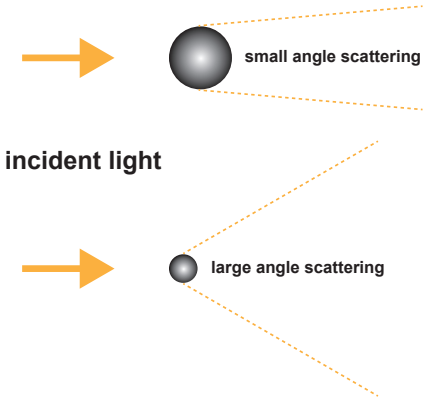
In chromatography, fine particles (*small particles under 10 microns*) increase back-pressure and can result in clogging, which is particularly dangerous when using glass columns. Fines can also pass through filters and contaminate final products. The lack of fines gives a more regular, stable and reproducible chromatography bed and a faster and more even flow rate for better separation.

The zoomed part of the figure shows that our most popular silica gel, Silia*Flash* 40 - 63 microns 60 Å, has total absence of fines unlike the six competitor gels analyzed.

Particle Size Analysis Methods

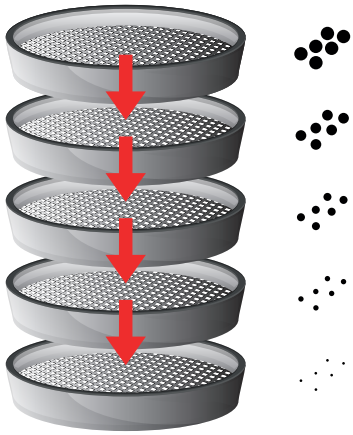
Laser Diffraction (*Malvern Analysis*)

Typically used for particle sizes below 40 microns. Particle size distribution is 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.



Video: Understanding particle size distribution

Two Different Grades for Different Needs

Over the years, SiliCycle has developed two different grades (*"Superior" and "Standard"*) for the two most popular irregular gels used in the industry:

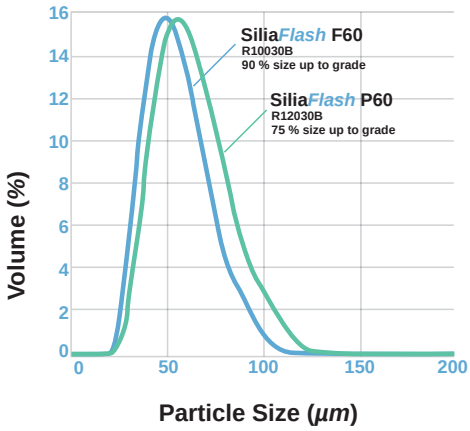
- 40 - 63 µm, 60 Å
- 60 - 200 µm, 60 Å

Those two grades of each gel are available to address all our customers' requirements, depending on their applications, areas of research, budgets and so on.

40 - 63 µm, 60 Å Gels: Silia*Flash* F60 (*R10030B*) VS Silia*Flash* P60 (*R12030B*)

Both compare favorably with the overall industry average of a 40 - 63 µm distribution, and each grade offers its own particle size distribution profile.

Two Different Grades of 40 - 63 µm, 60 Å Gel		
Grade	**Superior Grade**	**Standard Grade**
Name	F60	P60
PN	R10030B	R12030B
Particle Size	40 - 63 µm	40 - 63 µm
Pore Diameter	60 Å	60 Å
Particularities	• Extra step to reduce metal content to minimum level • Tighter particle size distribution • Fines have been removed	• Fines have been removed • Lower price

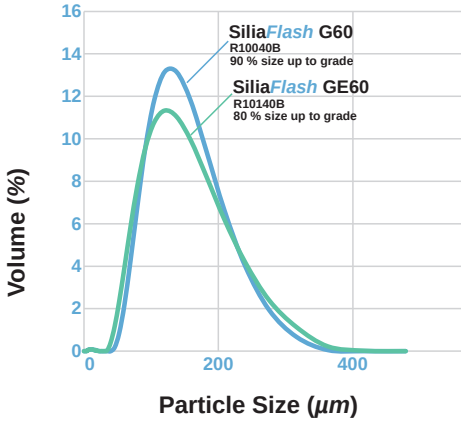


The figure on the right shows F60's tighter particle size distribution and the absence of fines for both gels.

60 - 200 µm, 60 Å Gels: Silia*Flash* G60 (*R10040B*) VS Silia*Flash* GE60 (*R10140B*)

Each grade offers its own particle size distribution profile.

Two Different Grades of 60 - 200 µm, 60 Å Gel		
Grade	**Superior Grade**	**Standard Grade**
Name	G60	GE60
PN	R10040B	R10140B
Particle Size	60 - 200 µm	60 - 200 µm
Pore Diameter	60 Å	60 Å
Particularities	• Extra step to reduce metal content to minimum level • Tighter particle size distribution • Fines have been reduced to minimal level	• Fines have been reduced to minimal level • Lower price



The figure on the right shows G60's tighter particle size distribution.

Typical Metal Content Comparison Between SiliCycle's Five Most Popular Gels

Typical Metal Content of Most Popular Irregular Silicas						
Product		**F60**	**P60**	**Acid Washed**	**G60**	**GE60**
Product Number		**R10030B**	**R12030B**	**R10530B**	**R10040B**	**R10140B**
Particle Size		40 - 63 µm			60 - 200 µm	
Pore Diameter		60 Å			60 Å	
Metal (*mg/kg*)						
Aluminum	Al	< 200	< 1,000	< 70	< 350	< 900
Antimony	Sb	< 0.2			< 0.2	
Arsenic	Ar	< 1			< 1	
Barium	Ba	< 40	< 40	< 5	< 40	
Beryllium	Be	< 0.1			< 0.1	
Bismuth	Bi	< 1			< 1	
Cadmium	Cd	< 0.01			< 0.01	
Calcium	Ca	< 200	< 500	< 10	< 250	< 500
Chromium	Cr	< 1			< 1	
Cobalt	Co	< 0.1			< 0.1	
Copper	Cu	< 1			< 1	
Iron	Fe	< 75	< 350	< 10	< 75	< 350
Lead	Pb	< 1			< 1	
Lithium	Li	< 0.1			< 0.1	
Magnesium	Mg	< 150	< 250	< 10	< 100	< 150
Manganese	Mn	< 1	< 2	< 1	< 1	
Molybdenum	Mo	< 0.1			< 0.1	
Nickel	Ni	< 1			< 1	
Potassium	K	< 500	< 30	< 2	< 750	< 30
Rubidium	Rb	< 0.2			< 0.2	
Selenium	Se	< 1			< 1	
Silver	Ag	< 0.1			< 0.1	
Sodium	Na	< 150	< 1,500	< 15	< 150	< 1,500
Strontium	Sr	< 4	< 15	< 1	< 4	< 15
Tellurium	Te	< 0.1			< 0.1	
Thallium	Tl	< 0.1			< 0.1	
Tin	Sn	< 0.4	< 0.4	< 0.2	< 0.4	
Titanium	Ti	< 200	< 250	< 90	< 250	
Uranium	U	< 0.1			< 0.1	
Vanadium	V	< 1			< 1	
Zinc	Zn	< 1			< 1	

Irregular Silica Selection Guide

Selecting the most appropriate sorbent for any given application can be difficult. To help you choose the right pore diameter and particle size, simply follow the two pathways to select the most suitable sorbent.

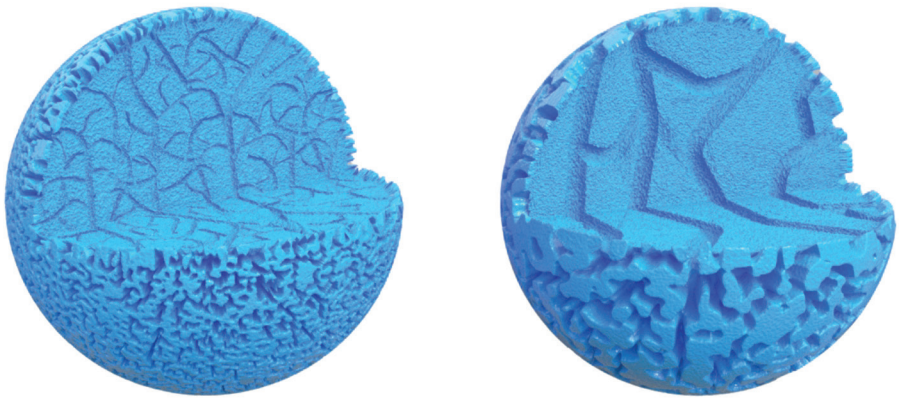
Selecting Pore Diameter

Sample Molecular Weight (*MW in Dalton*)			
	Organic Molecules	MW < 1,000 Dalton	30, 40 Å (< *400 Dalton*) 60, 70 Å (*400 - 1,000 Dalton*)
	Peptides	1,000 < MW < 10,000 Dalton	90, 100 Å 120, 150, 300 Å
	Proteins (*Polypeptides*) **Bio-polymers**	MW > 10,000 Dalton	500, 800 Å 1,000 Å

Selecting Particle Size

Generated Pressure (*GP*) or Selectivity (*α*)		
	GP ~950 psi (*65 bar*) **α < 1.2 (*very difficult separation*)**	From 0 to 30 µm
	GP ~220 psi (*15 bar*) **1.2 < α < 1.5 (*difficult separation*)**	From 15 to 40 µm
	GP ~73 psi (*5 bar*) **1.5 < α < 2.0 (*possible separation*)**	From 40 to 100 µm
	GP ~29 psi (*2 bar*) **α > 2.0 (*easy separation*)**	From 40 to 1,000 µm

Selectivity (α) is measured by the retention factor ratio between two similar compounds: $\alpha = \frac{Tr_1 - T_0}{Tr_2 - T_0}$



Small pores

Large pores

A Particle Size for Each Application

Most Popular Particle Size Applications		
Particle Size Distribution		Applications
Irregular Particles	Spherical Particles	
Particles for Preparative TLC Plates		
From 0 to 20 µm	-	• Contains neither binder (*organic or inorganic*) nor UV indicator (F_{254}) • Can also be used in flash chromatography if higher resolution is required (*higher back-pressure*)
Particles for Difficult Separations		
From 10 to 45 µm	From 15 to 45 µm	• High-resolution silica for difficult separations (*similar polarities*)
Particles for Flash Chromatography		
40 - 63 µm	From 40 to 75 µm	• Chromatography types: ▪ high-resolution flash chromatography ▪ low to medium-pressure preparative chromatography • Narrow particle size distribution • Easier to pack and more uniform packing • Superior resolution • Suitable for use with complex matrices Most popular particle size distribution
60 - 120 µm	From 60 to 150 µm	• Alternative to 40 - 63 µm silica for faster flow rate with lower pressure
Particles for Column (*or Gravity*) Chromatography		
From 60 to 200 µm	From 75 to 250 µm	• Most economical silica for open column chromatography (*gravity*) • Suitable for very dirty purification • Easier to handle
From 120 to 200 µm	From 100 to 200 µm	• Silica for standard open column chromatography • Narrow particle size distribution enables uniform packing • Suitable for mass overload purification
Other Application		
From 200 to 1,000 µm	From 200 to 500 µm	• Silica for plugs

Silia*Flash* & Silia*Sphere* PC Ordering Information

This is only an overview of gels we can provide. Please contact us if you are looking for a different product: support@silicycle.com.

Available formats: from 1 kg to 25 kg, even up to multi-ton scale!

Silia*Flash* Irregular Silica Gels Portfolio

Product Number	Particle Size µm	Particle Size mesh	Pore Diameter (Å)
R10137L	75 - 150	100 - 200	30
R10130A	40 - 63	230 - 400	40
R10150A	60 - 120	325 - 625	
R10140A	60 - 200	70 - 230	
R10160A	120 - 200	70 - 125	
R10170A	200 - 500	35 - 70	
R10180A	500 - 1,000	18 - 35	
R10117B	15 - 40	*	60
R10023B	20 - 45	*	
R10030B (*F60*)	40 - 63	230 - 400	
R12030B (*P60*)			
R10530B (*Acid-Washed*)			
R10050B	60 - 120	325 - 625	
R10040B (*G60*)	60 - 200	70 - 230	
R10140B (*GE60*)			
R10137B	75 - 150	100 - 200	
R10160B	120 - 200	70 - 125	
R10165B	150 - 250	60 - 100	
R10170B	200 - 500	35 - 70	
R10180B	500 - 1,000	18 - 35	
R10130D	40 - 63	230 - 400	90
R10140D	60 - 200	70 - 230	
R10170D	200 - 500	35 - 70	
R10180D	500 - 1,000	18 - 35	
R10181D	800 - 1,200	16 - 22	
R10130H	40 - 63	230 - 400	150
R10150H	60 - 120	325 - 625	
R10140H	75 - 250	60 - 200	
R10160H	120 - 200	70 - 125	
R10170H	200 - 500	35 - 70	
R10180H	500 - 1,000	18 - 35	
R10181H	800 - 1,200	16 - 22	
R10130M	40 - 63	230 - 400	300
R10140M	60 - 200	70 - 230	
R10170M	200 - 500	35 - 70	

Silia*Sphere* PC Spherical Silica Gels Portfolio

Product Number	Particle Size µm	Particle Size mesh	Pore Diameter (Å)
S10095W-A	25	*	50
S10030B-A	50	300	60
S10040B-A	100	150	
S10020C	20 - 45	*	70
S10030C	40 - 75	200 - 400	
S10040C	75 - 200	70 - 200	
S10070C	200 - 500	35 - 70	
S10095D-A	25	*	90
S10020E	20 - 45	*	100
S10030E	40 - 75	200 - 400	
S10040E	75 - 200	70 - 200	
S10070E	200 - 500	35 - 70	
S10027G-A	50	300	120
S10020M	20 - 45	*	300
S10030M	40 - 75	200 - 400	
S10040M	75 - 200	70 - 200	
S10070M	200 - 500	35 - 70	
S10020P	20 - 45	*	500
S10030P	40 - 75	200 - 400	
S10040P	75 - 200	70 - 200	
S10070P	200 - 500	35 - 70	
S10020S	20 - 45	*	800
S10030S	40 - 75	200 - 400	
S10040S	75 - 200	70 - 200	
S10070S	200 - 500	35 - 70	
S10020T	20 - 45	*	1,000
S10030T	40 - 75	200 - 400	
S10040T	75 - 200	70 - 200	
S10070T	200 - 500	35 - 70	

* Mesh equivalent too small to exist as real screen size.

R10530B: Acid-washed Silia*Flash* 40 - 63 µm, 60 Å irregular silica gel for extra purity

This product gel has been developed to ensure a pH-controlled media with even lower levels of trace metal contaminants and maximal purity.

Chromatographic Phases

Thanks to its high mechanical resistance, silica is the most widely used media in chromatography. With Silia*Bond* irregular silica gels, SiliCycle offers a large range of solutions for low pressure chromatography, to help cover many kinds of purification.

We guarantee quality and stability of our phases: no fines will appear when packing the media. Our gels will give you excellent performance and lifetime!

Silia*Bond* Phases for Low Pressure Chromatography

For all our listed Silia*Bond* sorbents, particle size is 40 - 63 µm and pore diameter is 60 Å. Contact us if you need a different particle size or pore diameter: support@silicycle.com.

All functionalized Silia*Bond* sorbents are available in bulk but also pre-packed in Silia*Sep* flash cartridges and Silia*Prep* SPE cartridges.

Reversed-Phases

In reversed-phase chromatography, the packing material is always hydrophobic (*non polar*) while the mobile phase is polar. The more hydrophobic the packing material, the more retention of non polar analytes.

Usual reversed-phases are standard alkyl chains grafted on silica (*C18, C8, C4, C1*) and cyclic or aromatic functions (*Phenyl, Pentafluorophenyl*).

Important parameters to keep in mind in reversed-phase chromatography:

- **Carbon load** (*% C*) will give the relative hydrophobicity of the packing media. Most of the time, it varies between 5 % and 17 %.
- **Endcapping**: when functionalizing silica, it is impossible to react with all available silanol groups (*free -OH groups on the silica surface*). But these free silanols are acidic and will react with basic compounds, so we endcap them with a capping agent to avoid non-specific bindings.

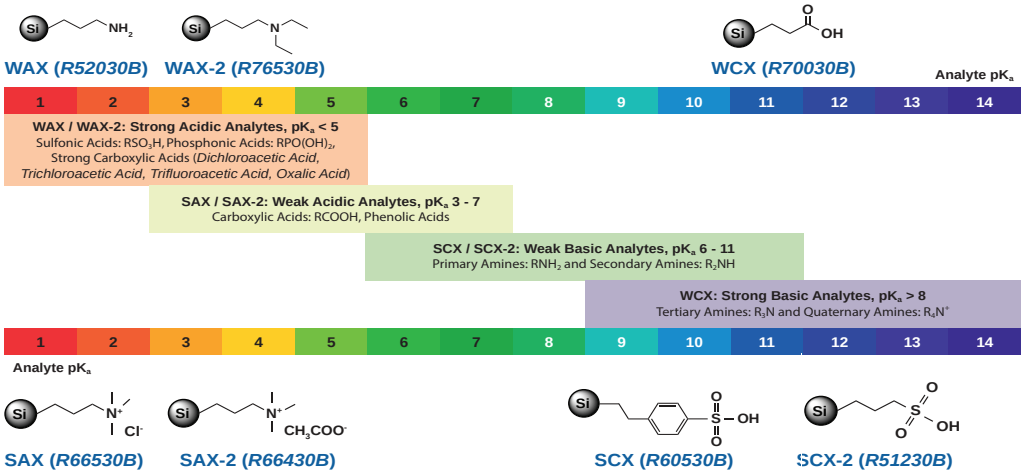
Normal Phases

In normal phase chromatography, the packing material is always polar while the mobile phase is non polar. The interactions between analytes and sorbent mainly take place on the highly polar silanols of the silica gel surface. Some hydrogen bonds can also happen on polar functionalized groups.

Usual normal phases are ungrafted silica, polar functions (*amine, cyano and diol*) or alternative adsorbents (*Alumina and Florisil® for example*).

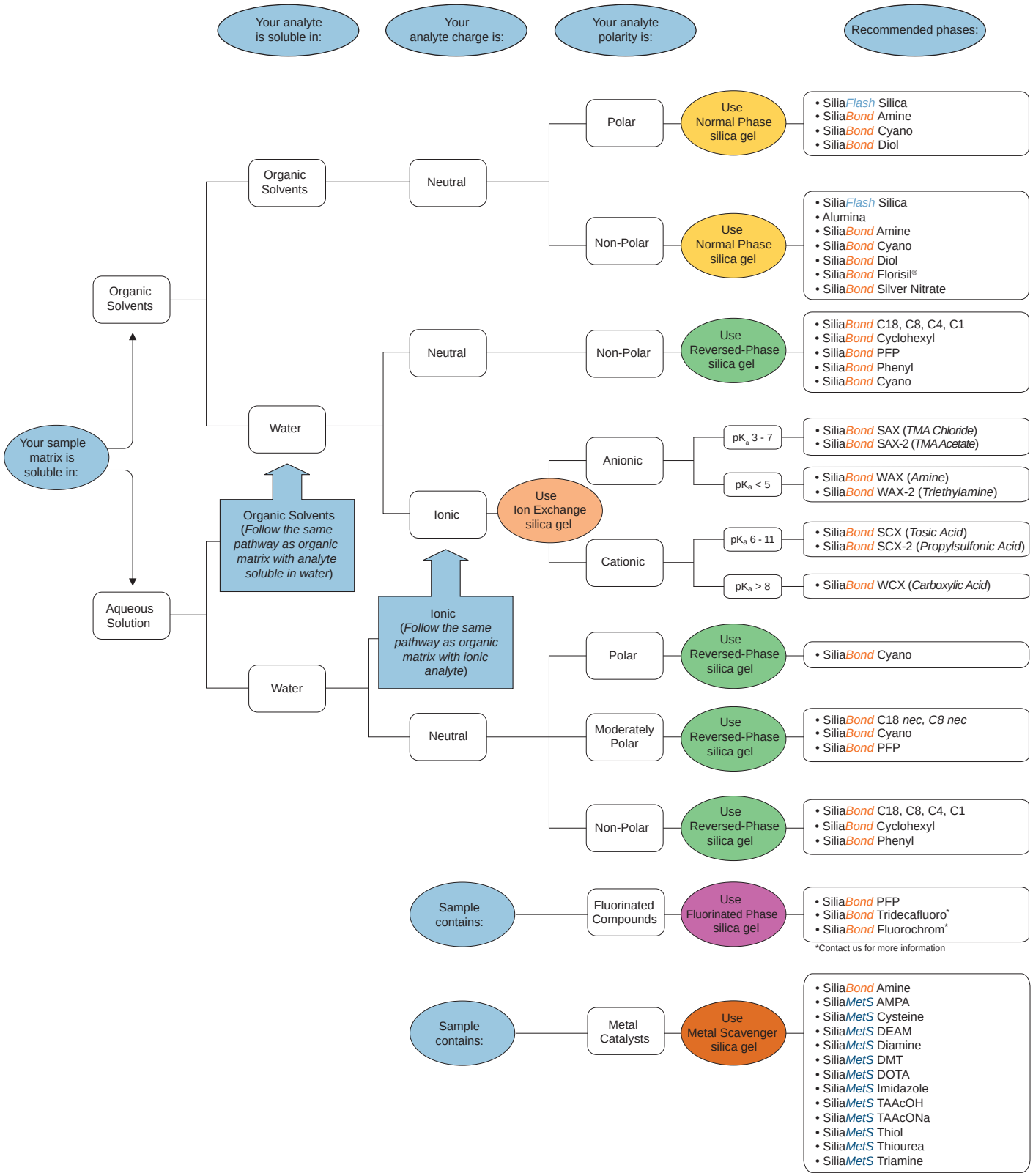
Ion Exchange Phases

In ion exchange chromatography, both silica support and analytes must be ionized. If the stationary phase (*packing material*) is positively charged, anionic analytes only will retain (*these phases are called WAX and SAX*). And in the contrary if the stationary phase is negatively charged, cationic analytes only will retain (*these phases are called WCX and SCX*). Hence, pH of the mobile phase is of crucial importance and needs to be chosen carefully, so that both functions are charged:



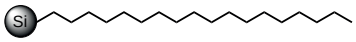
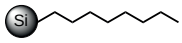
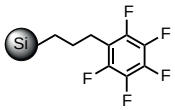
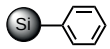
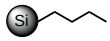
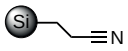
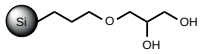
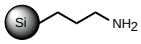
Sorbent Selection Chart

SiliCycle offers a wide range of Silia*Bond* sorbents to cover many kinds of purification. The following chart will guide you for the selection of the appropriated sorbent, based on the characteristics of the sample to purify.



Reversed & Normal Phases Portfolio

Available formats: from 5 g to 25 kg.

	Low Pressure Chromatography Reversed & Normal Phases Characteristics			
	Sorbent	**Structure**	**Typical Characteristics**	**Typical Applications**
Reversed-Phases	**C18** PN: R33230B		% C: ≥ 17.0 % Density: 0.639 g/mL	• Purification of low to high polarity compounds • Reproducible purification without complexity and cost of preparative HPLC
	C8 PN: R30830B		% C: ≥ 11.0 % Density: 0.586 g/mL	• Less retention compared to C18 • For highly hydrophobic pesticides, small peptides and large molecule drugs
	Pentafluorophenyl (*PFP*) PN: R67530B		% C: ≥ 9.0 % Density: 0.761 g/mL	• For an alternative selectivity, with aromatic ring interactions • For purification of conjugated compounds (*isomers*)
	Phenyl (*PHE*) PN: R34030B		% C: ≥ 8.0 % Density: 0.637 g/mL	• Moderately non-polar sorbent • Alternative selectivity for aromatic compounds, compared to other reversed-phases
	C4 PN: R32030B		% C: ≥ 8.0 % Density: 0.656 g/mL	• Less retention compared to C18 and C8 • For molecules with large hydrophobic regions
	C1 PN: R33030B		% C: ≥ 5.0 % Density: 0.559 g/mL	• Lower retention compared to other reversed-phases • For purification of polar and non-polar highly hydrophobic pharmaceutical products
Normal Phases	**Cyano** (*CN*) PN: R38030B		% C: ≥ 7.0 % % N: ≥ 1.93 % Loading: ≥ 1.38 mmol/g Density: 0.703 g/mL	• Versatile sorbent used either as normal or reversed-phase • Less polar than silica • For organic compounds with intermediate to extreme polarity
	Silica (*Si*) PN: R10030B		Density: 0.550 g/mL	• Most popular sorbent for day-to-day use • For purification of non-ionic polar organic compounds
	Silica Premium PN: S10095D-A		Particle size: 25 µm Pore size: 90 Å Density: 0.450 g/mL	• High performance sorbent for difficult separations (*isomers*) • Higher loading capacity, faster flow rate, less solvent used
	Diol *nec* PN: R35030B		Loading: ≥ 0.97 mmol/g Density: 0.687 g/mL	• For difficult separation of low to medium polarity samples • Can be used in HILIC mode • For mono and polysaccharides separation
	Amine (*NH_2, WAX*) PN: R52030B		Loading: ≥ 1.2 mmol/g Density: 0.700 g/mL	• For purification of compounds with basic properties, or for monosaccharides separation
	Acidic, Neutral & Basic Alumina PN: AUT-0053 AUT-0054 AUT-0055		Particle size: 75 - 150 µm	• For aromatic compounds, aliphatic amines & compounds containing electronegative functions
	Florisil® PN: AUT-0014		Particle size: ≤ 75 µm Pore size: 80 Å	• For separation of chlorinated pesticides, polychlorinated biphenyls (*PCBs*) & polysaccharides
	Silver Nitrate (*$AgNO_3$*) PN: R23530B		Loading: 10 % w/w Density: 0.604 g/mL	• For separation of cis / trans isomers of unsaturated compounds (*alkenes, lipids, steroids and terpenes*)

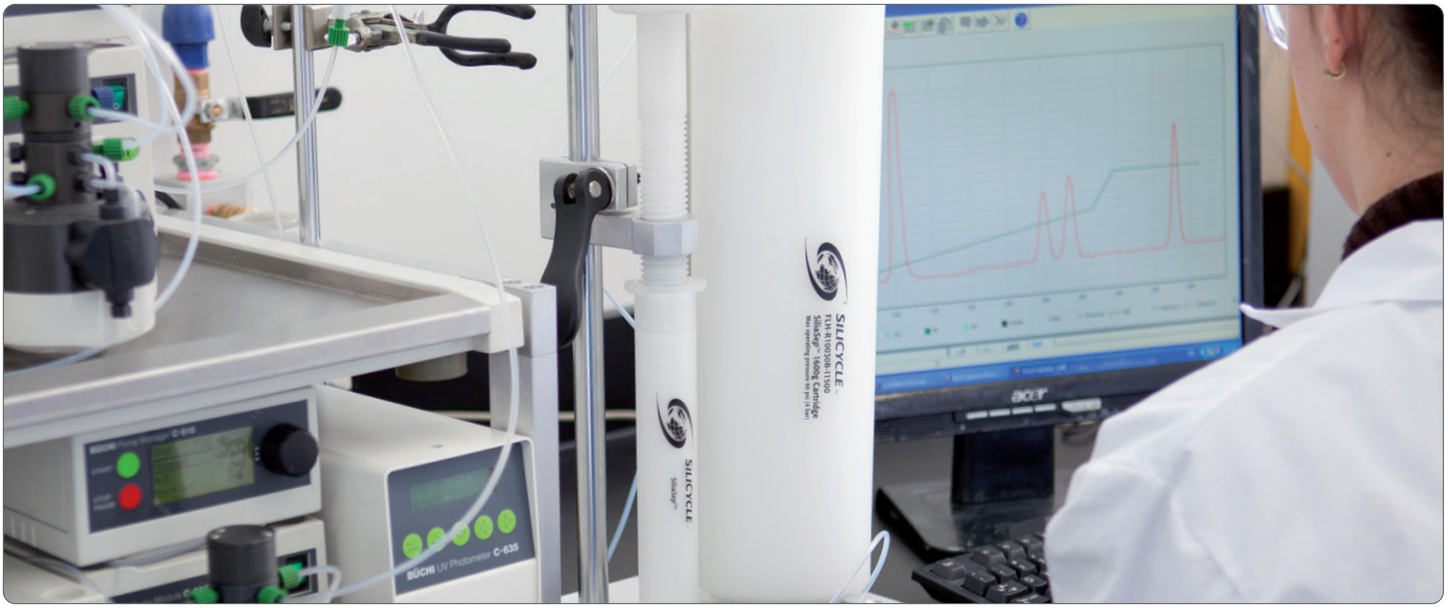
If not otherwise stated, particle size is 40 - 63 µm and pore diameter is 60 Å.
All phases are available endcapped and non-endcapped.
Other phases could be offered on a custom basis, contact us for more information: info@silicycle.com.

Typical Reversed and Normal Phases Applications

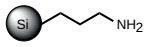
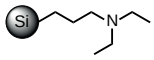
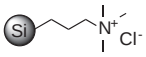
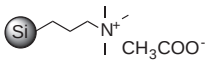
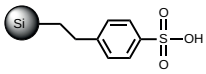
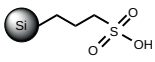
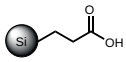
The table below will help you select the right media to purify your compounds of interest. All phases are available either in bulk or pre-packed cartridges.

Typical Applications Using Reversed and Normal Phases												
Analytes	**Examples**	**C18**	**C8**	**C6**	**PFP**	**PHE**	**C4**	**C1**	**CN**	**NH_2**	**Si**	**Diol**
Biomolecules	Peptides, proteins	✓	✓	✓			✓	✓				✓
Nucleotides	Deoxyribonucleotides, ribonucleotides	✓								✓		
Lipids	Phospholipids		✓	✓			✓	✓		✓		
Carbohydrates	Sugars								✓	✓		✓
Glycosides	Glucosides, fructosides								✓	✓		✓
Oligosaccharides	Malto-oligosaccharides									✓		✓
Pesticides	Organophosphates	✓	✓									
PCBs	Dichlorobiphenyl, trichlorobiphenyl	✓			✓	✓						
PAHs	Anthracene, pyrene	✓	✓		✓	✓						
Drugs	Basic drugs, metabolites	✓	✓	✓					✓	✓	✓	
Alkaloids	Cocaine, morphine, nicotine, quinine	✓	✓						✓		✓	
Analgesics	Aspirin, acetaminophen, ibuprofen	✓	✓		✓				✓			
Cyclosporine	-	✓							✓			
Conjugated Compounds	Phenols, chloroanilines, steroids, caffeine	✓	✓	✓	✓	✓	✓	✓				
Natural Compounds	Tannins, aflatoxins, flavonoids, carotenoids	✓	✓	✓	✓	✓	✓	✓				
Fat-Soluble Vitamins	Vitamins A, D, E and K	✓	✓									
Water-Soluble Vitamins	Vitamins B and C									✓	✓	
Heterocyclic Compounds	Dioxins, furans	✓										

$AgNO_3$ is particularly useful to separate isomers that present unsaturated groups.
Neutral Alumina is used for the separation of aldehydes, ketones, quinines, esters, lactones and glucosides.
Florisil® will help analyze pesticides, PCBs and PAHs.



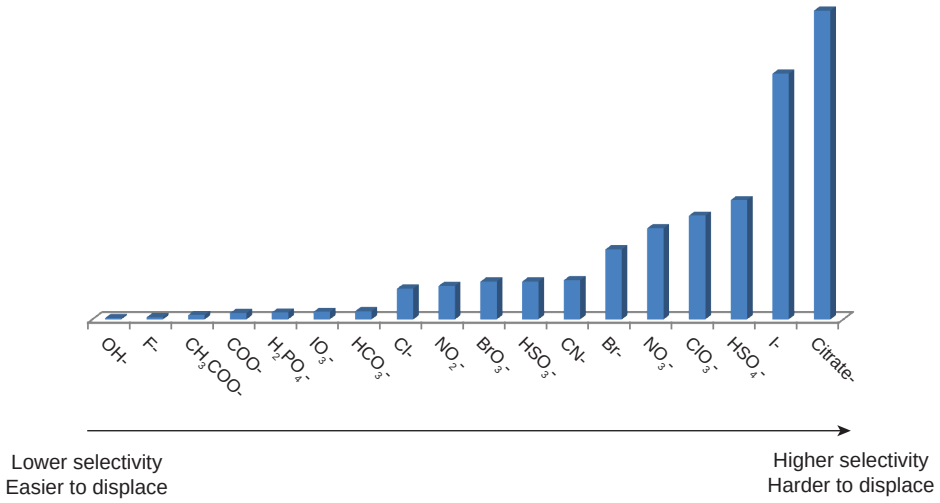
Ion Exchange Phases Portfolio

Low Pressure Chromatography Ion Exchange Phases Characteristics				
	Sorbent	Structure	Typical Characteristics	Typical Applications
Ion Exchange Phases	**Amine** (*NH_2, WAX*) PN: R52030B		Loading: ≥ 1.2 mmol/g Density: 0.700 g/mL	• Weak anion exchanger (*pK_a of 9.8*), positively charged at pH below 7.8 • For very strong anions (*such as sulfonic acids*), that may be too strongly retained on SAX phases
	WAX-2 (*Triethylamine*) PN: R76530B		Loading: ≥ 1.04 mmol/g Density: 0.761 g/mL	• Weak anion exchanger (*pK_a of 10.5*), positively charged at pH below 8.5 • For catch & release of compounds bearing a permanent negative charge (*like salts of sulfonic acids*)
	SAX *nec* (*TMA Chloride*) PN: R66530B		Loading: ≥ 0.90 meq/g Density: 0.700 g/mL	• Strong anion exchanger, permanently positively charged (*pH independant*) • For weak anions (*such as carboxylic acids*) that may not bind strongly enough on WAX phases • For analysis of acidic drugs / analgesics, biomolecules & water-soluble vitamins
	SAX-2 *nec* (*TMA Acetate*) PN: R66430B		Loading: ≥ 0.71 mmol/g Density: 0.665 g/mL	• Strong anion exchanger, with easily exchangeable acetate counter-ion (*more than chloride ion*) • For compounds with pK_a < 5 (*such as carboxylic acids*)
	SCX (*Tosic Acid*) PN: R60530B		Loading: ≥ 0.54 meq/g Density: 0.698 g/mL	• Strong cation exchangers (*pK_a < 1*), permanently negatively charged (*pH independant*) • For catch and release purification of weak cations (*basic drugs / analgesics, biomolecules & water-soluble vitamins*)
	SCX-2 (*Propylsulfonic Acid*) PN: R51230B		Loading: ≥ 0.63 meq/g Density: 0.728 g/mL	
	WCX (*Carboxylic Acid*) PN: R70030B		Loading: ≥ 0.92 meq/g Density: 0.687 g/mL	• Weak cation exchanger (*pK_a of 4.8*), neutralized at pH below 2.8 • For strong cationic species, that may bind too strongly on SCX phases

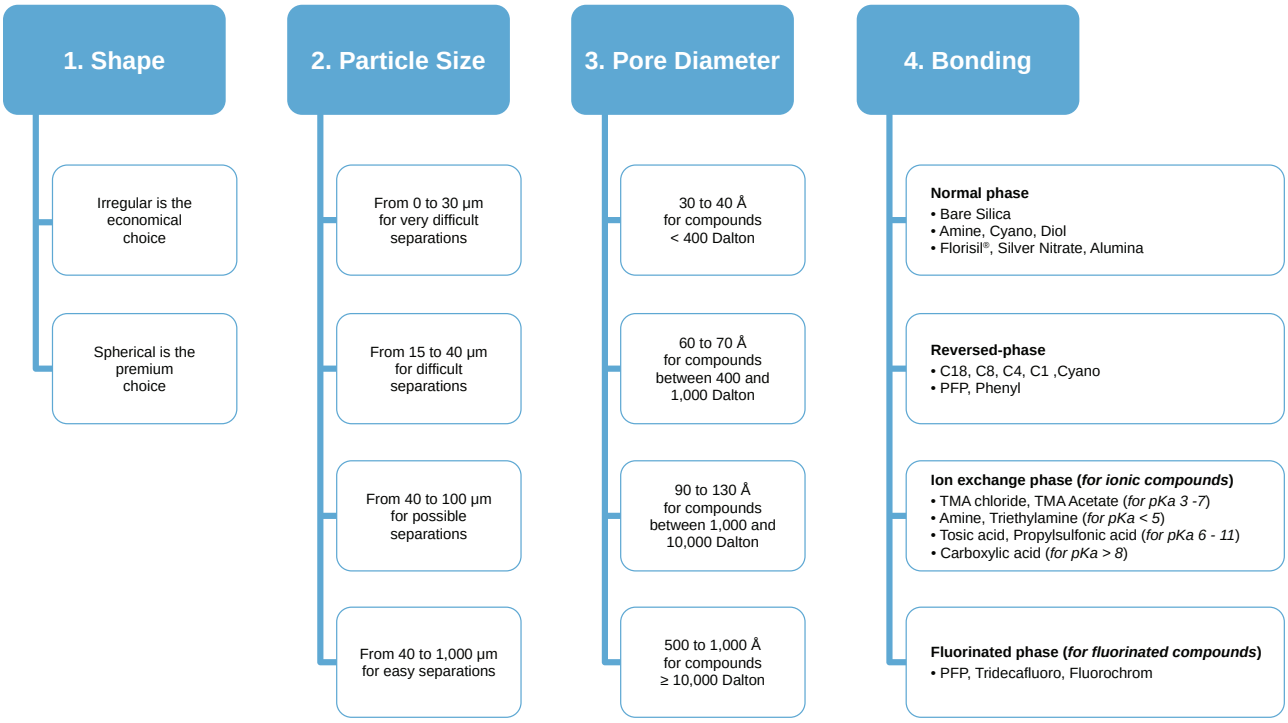
For all sorbents, particle size is 40 - 63 µm and pore diameter is 60 Å.
All bonded phases are available endcapped and non-endcapped.
Other phases could be offered on a custom basis, contact us for more information: info@silicycle.com.

Counter-Ion Selectivity in Ion Exchange Mode

SAX phases are always paired with a counter-ion to neutralize the quaternary amine charge. But counter-ions have different selectivites and some are more easily removed from the silica gel by the analyte. You will find below the relative selectivity of standard counter-ions, compared to the hydroxyl ion OH^- (*lowest selectivity*). Always choose a phase paired with a counter-ion less selective than the analyte.



Steps to Choose the Sorbent



Silia*Bond* Bulk Ordering Information

To build your own product number, just choose the right Codes for the desired Phase, Physical Characteristics, and Format:
[Phase Code][Properties Code]-[Quantity Code]
Example: 100 g of C18 silica gel, 40 - 63 µm, 60 Å: R33230B-100G.

Silia*Bond* phases are available on all irregular Silia*Flash* silicas (*R100-*) and on all spherical Silia*Sphere* PC silicas (*S100-*).
See page 11 for all available irregular & spherical silica types and corresponding codes.

You will find below the most common bare & bonded silica gels ordered in bulk.
Please note product numbers begin by **R-** for irregular silicas and by **S-** for spherical silicas.

Silia*Bond* Phases

Phase	Code
Silia*Bond* Silica	R100
Silia*Bond* Amine	R520
Silia*Bond* Diol *nec*	R350
Silia*Bond* Cyano	R380
Silia*Bond* C18	R332
Silia*Bond* C8	R308
Silia*Bond* Phenyl	R340
Silia*Bond* PFP	R675
Silia*Bond* SCX	R605
Silia*Bond* SCX-2	R512
Silia*Bond* SAX *nec*	R665
Silia*Bond* SAX-2 *nec*	R664

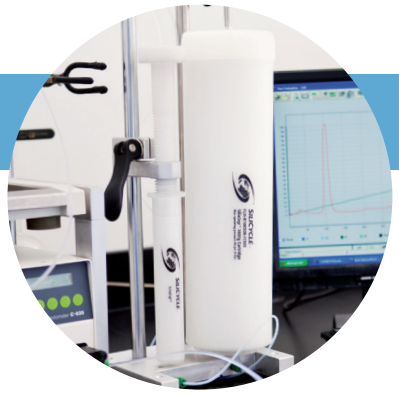
Silia*Bond* Characteristics

Properties	Code
25 µm, 90 Å	95D-A
40 - 63 µm, 60 Å	30B
40 - 63 µm, 300 Å	30M
40 - 75 µm, 100 Å	30E
40 - 200 µm, 60 Å	40B
50 µm, 120 Å	27G-A
200 - 500 µm, 60 Å	70B

Silia*Bond* Bulk Formats

Quantity	Code
5 g	5G
10 g	10G
25 g	25G
50 g	50G
100 g	100G
250 g	250G
500 g	500G
1 kg	1KG
5 kg	5KG
10 kg	10KG
25 kg	25KG

Flash Cartridges



Flash chromatography is one of the most used methods for compounds purification.

Several types of flash chromatography are available, and it has been showed that the use of pre-packed flash cartridges improves purification efficiency compared to conventional flash, by offering superior reproducibility and productivity due to its tightly and homogeneously packed silica bed.

With Silia*Sep*, benefit from SiliCycle's renown quality: selectivity, speed and reliability.

Cartridge Design

Features and Benefits

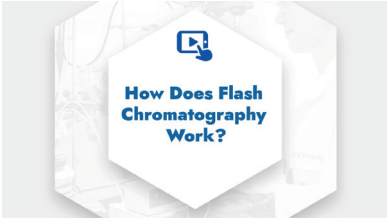
High silica gel quality, with low level of fines
- No product contamination
- Homogeneous packing, no channelling (*no peak tailing*)
- High loading capacity (*high surface area*)
- Direct transfer from TLC to flash chromatography

Reproducibility, reliability and safety
- Leak-free guaranteed by unique one-piece cartridge design
- Batch-to-batch reproducibility (*stringent quality control*)
- Excellent durability to withstand high pressures
- Universal luer fittings for compatibility with any flash system

Versatility
- Wide choice of cartridge sizes from 4 g to 10 kg
- Purification scale-up from milligrams to kilograms
- Variety of sorbents to meet any separation needs

Effective packing technology
- Consistent packing for reproducible high plate count (*N*)
- Excellent performance and separation
- High resolution with tight band definition (*no tailing*)
- Great compound purity and recovery

Cost effectiveness
- Excellent performance / price ratio
- Readily available, even for large volumes



Video: How flash chromatography works

Portfolio

All our bare and bonded silica gels are available to be packed in Silia*Sep* flash cartridges to accomodate your chemistry.

Silia*Sep* Flash Cartridges Adsorbents	
Adsorbent Type	**Adsorbent**
Backbone	• Standard Silia*Flash* Irregular Silica, 40 - 63 µm, 60 Å • PREMIUM Spherical Silica, 25 µm, 90 Å • Acidic, Neutral and Basic Alumina, 50 - 75 µm, 55 Å
Bonded phases	• Silia*Bond* Chromatographic Phases (*reversed, normal and ion exchange phases*) • Silia*MetS* Metal Scavengers (*Thiol, DMT, etc.*) • Silia*Bond* Organic Scavengers (*Amine, Tosic Acid, etc.*)

Formats

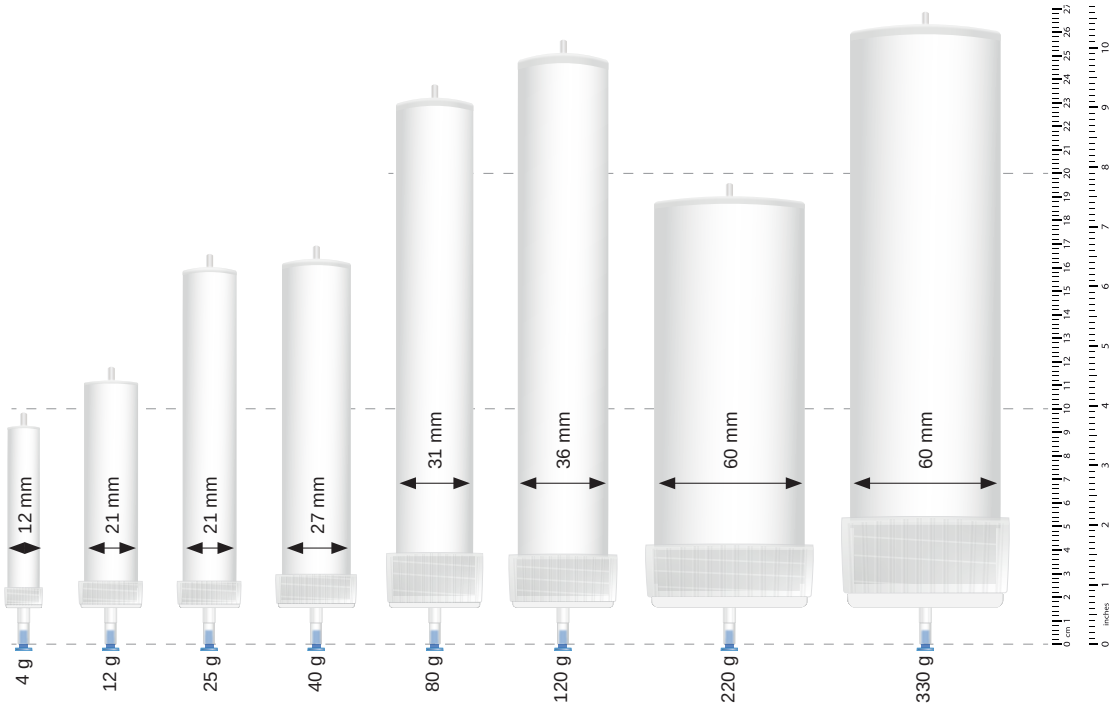
	Silia*Sep* Flash Cartridges Portfolio							
	Cartridge Format [Code]	**Silica Weight (*g*)**	**Qty / Box**	**Dimensions (*Diam. × Length**) (*mm*)**	**Column Volume (*mL*)**	**Recommended Flow Rate (*mL/min*)**	**Loading Capacity (*g*)**	**Max Operating Pressure (*psi / bar*)**
Discovery and R&D	**Silia*Sep* 4 g** [ISO04]	Bare: 4	20	12 × 98	6	15 - 25	Bare: 0.04 - 0.4 Bonded: 0.02 - 0.2	225 / 16
		Bonded: ≥ 5	2					
	Silia*Sep* 12 g [ISO12]	Bare: 12	20	21 × 117	20	20 - 40	Bare: 0.12 - 1.2 Bonded: 0.06 - 0.6	
		Bonded: ≥ 15	1					
	Silia*Sep* 25 g [ISO25]	Bare: 25	15	21 × 165	32	20 - 45	Bare: 0.25 - 2.5 Bonded: 0.125 - 1.25	
		Bonded: ≥ 30	1					
	Silia*Sep* 40 g [ISO40]	Bare: 40	15	27 × 169	50	25 - 50	Bare: 0.4 - 4 Bonded: 0.2 - 2	
		Bonded: ≥ 45	1					
	Silia*Sep* 80 g [ISO80]	Bare: 80	12	31 × 237	110	40 - 80	Bare: 0.8 - 8 Bonded: 0.4 - 4	
		Bonded: ≥ 90	1					
	Silia*Sep* 120 g [IS120]	Bare: 120	10	36 × 256	155	60 - 120	Bare: 1.2 - 12 Bonded: 0.6 - 6	205 / 13
		Bonded: ≥ 130	1					
	Silia*Sep* 220 g [IS220]	Bare: 220	4	60 × 195	280	60 - 190	Bare: 2.2 - 22 Bonded: 1.1 - 11	160 / 11
		Bonded: ≥ 230	1					
	Silia*Sep* 330 g [IS330]	Bare: 330	4	60 × 268	430	80 - 190	Bare: 3.3 - 33 Bonded: 1.65 - 16.5	
		Bonded: ≥ 360	1					
Development and Process	**Silia*Sep* XL 800 g**** [IS750]	Bare: 800	2	78 × 382	1,050	200 - 300	Bare: 8 - 80 Bonded: 4 - 40	125 / 8
		Bonded: ≥ 870	1					
	Silia*Sep* XL 1,600 g** [I1500]	Bare: 1,600	2	104 × 429	2,000	300 - 450	Bare: 16 - 160 Bonded: 8 - 80	100 / 7
		Bonded: ≥ 1,700	1					
	Silia*Sep* XL 3 kg** [ISO3KG]	Bare: 3,000	1	128 × 510	3,850	200 - 500	Bare: 30 - 300	100 / 7
	Silia*Sep* XL 5 kg** [ISO5KG]	Bare: 5,000	1	128 × 770	6,500	200 - 500	Bare: 50 - 500	100 / 7
	Silia*Sep* XL 10 kg** [ISO10KG]	Bare: 10,000	1	128 × 850	13,000	300 - 600	Bare: 100 - 1,000	100 / 7

* Cartridge length includes luer-lock and connection tip.

** For Silia*Sep* XL formats, you may need to use an XL Adapter to connect the cartridge onto your system. Part number AUT-0127-2.

Note: a higher flow rate will generate higher pressure, especially with spherical silica. Be careful to always respect the recommended pressure limit.

Silia*Sep* Relative Dimensions



Silia*Sep* Flash Cartridges System Compatibility

Silia*Sep* cartridges are compatible with a variety of flash systems. They are a slip tip connection type, but they can be used with many leur lock systems. With some systems, and adapter kit might be required. The Table below is a guide to determine if an adapter is required to use Silia*Sep* cartridges with your flash system.

Silia*Sep* System Compatibility		
System	**Silia*Sep* Cartridges**	**Comments**
Teledyne Isco™ CombiFlash®	COMPATIBLE	100 % compatible
Biotage Isolera™ & Selekt®	COMPATIBLE	100 % compatible
Büchi Pure™ & Sepacore™	COMPATIBLE	100 % compatible
Gilson PLC	COMPATIBLE	100 % compatible
Grace Reveleris™	COMPATIBLE	100 % compatible
Interchim PuriFlash™ & Spot II (Armen®)	COMPATIBLE	100 % compatible
Varian® (Analogix®) IntelliFlash® & SimpliFlash®	COMPATIBLE	100 % compatible
Biotage Horizon™	WITH ADAPTER	Use the Biotage Adapter Kit (PN: KAD-1006) or the Solvent Line Replacement (PN: KAD-1014)
Biotage SP1 & SP4	WITH ADAPTER	Use Support Rings to allow the Silia*Sep* cartridge to sit on the instrument (Support Ring Kit PN: KAD-1008)
Biotage FlashMaster™	WITH ADAPTER	Use the FlashMaster Adapter Kit (PN: KAD-1016) or connect a Silia*Sep* OT cartridge

Method Development

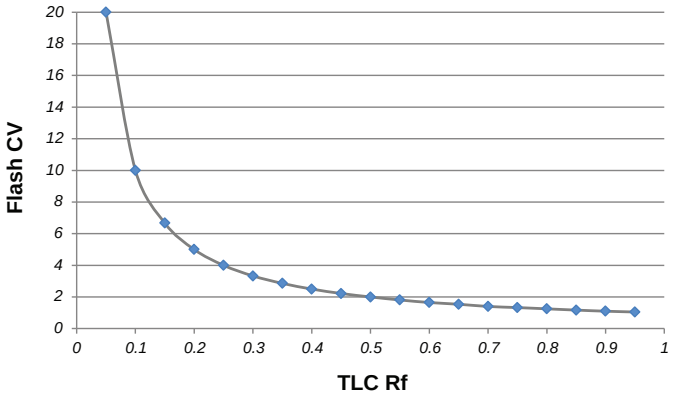
Prediction of Column Volumes (*CV*)

TLC data can be used to predict flash purification, based on the relationship between TLC retention factor (*Rf*) and flash retention time (*measured in column volume, CV*). CV is the number of column volumes required to elute the component from the column, regardless of column dimensions.

So the first step to convert a TLC method in flash chromatography is to convert Rf into CV.

Rf and CV are inversely proportional: **CV = 1 / Rf**

The graph below shows that lower Rfs in TLC means greater CVs in flash (*so better analyte retention*). On the right is a chart giving CV values according to typical Rf values.



Video: Relationship between retention factor and column volume

As CV is a measure of analyte retention, then ΔCV is a measure of two analytes separation and resolution: **$\Delta CV = CV_2 - CV_1 = (1 / Rf_2) - (1 / Rf_1)$**

ΔCV Values According To Rf_1 And Rf_2 Values

Rf_1 \ Rf_2	0.05	0.10	0.15	0.20	0.25	0.30	0.35	0.40	0.45	0.50	0.55	0.60	0.65	0.70	0.75	0.80	0.85	0.90	0.95
0.05	0.00																		
0.10	10.00	0.00																	
0.15	13.33	3.33	0.00																
0.20	15.00	5.00	1.67	0.00															
0.25	16.00	6.00	2.67	1.00	0.00														
0.30	16.67	6.67	3.34	1.67	0.67	0.00													
0.35	17.14	7.14	3.81	2.14	1.14	0.47	0.00												
0.40	17.50	7.50	4.17	2.50	1.50	0.83	0.36	0.00											
0.45	17.78	7.78	4.45	2.78	1.78	1.11	0.64	0.28	0.00										
0.50	18.00	8.00	4.67	3.00	2.00	1.33	0.86	0.50	0.22	0.00									
0.55	18.19	8.19	4.86	3.16	2.16	1.52	1.05	0.69	0.41	0.19	0.00								
0.60	18.35	8.35	5.02	3.35	2.35	1.68	1.21	0.85	0.57	0.35	0.16	0.00							
0.65	18.46	8.46	5.13	3.46	2.46	1.79	1.32	0.98	0.68	0.46	0.27	0.11	0.00						
0.70	18.60	8.60	5.27	3.60	2.60	1.93	1.46	1.10	0.82	0.60	0.41	0.25	0.14	0.00					
0.75	18.67	8.67	5.34	3.67	2.67	2.00	1.53	1.17	0.89	0.67	0.48	0.32	0.21	0.07	0.00				
0.80	18.75	8.75	5.42	3.75	2.75	2.08	1.61	1.25	0.97	0.75	0.56	0.40	0.29	0.15	0.08	0.00			
0.85	18.83	8.83	5.50	3.83	2.83	2.16	1.69	1.33	1.05	0.83	0.64	0.48	0.37	0.23	0.16	0.08	0.00		
0.90	18.90	8.90	5.57	3.90	2.90	2.23	1.76	1.40	1.12	0.90	0.71	0.55	0.44	0.30	0.23	0.15	0.07	0.00	
0.95	18.95	8.95	5.62	3.95	2.95	2.28	1.81	1.45	1.17	0.95	0.76	0.60	0.49	0.35	0.28	0.20	0.12	0.05	0.00

From TLC to Low Pressure Chromatography

It is now understood that TLC methods should be optimized so that compounds of interest elute with lower Rfs, ideally between 0.1 and 0.4, to maximize retention and analytes separation. To obtain these Rfs values, you can adjust the TLC solvent mixture by using different solvents with different polarities, and change the composition of the final TLC solvent mixture.

An optimized TLC method will assure you a better separation and purification of your compounds in low pressure chromatography, with optimal loading capacity (*you will be able to load more on the cartridge if your compounds are well separated*).

We recommend using a flash cartridge phase matching the TLC plate, for a more linear and easy method conversion. You should also run your flash chromatography with the same solvent conditions as your TLC method (*in isocratic mode*).

Case Study

We need to separate two analytes, 1 and 2. We will study two different TLC configurations.

FROM TLC ...

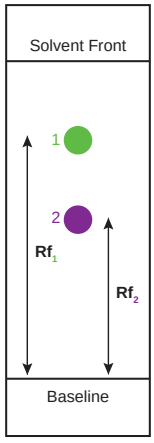


Plate 1

1 and 2 TLC spots are high and well distinct

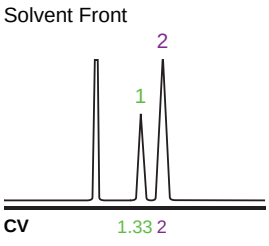
Converting TLC to Low Pressure Chromatography

	Rf_1	CV_1	Rf_2	CV_2	ΔCV
Plate 1	0.75	1.33	0.5	2	**0.67**
Plate 2	0.2	5	0.1	10	**5**

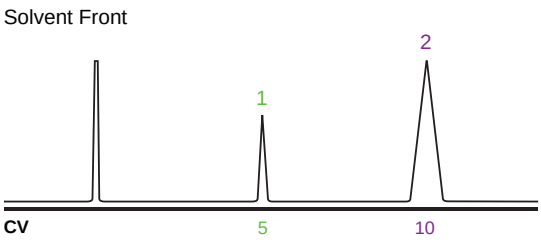
1 and 2 TLC spots are low and quite close

Plate 2

... TO LOW PRESSURE CHROMATOGRAPHY



1 and 2 are poorly retained and separated in flash chromatography



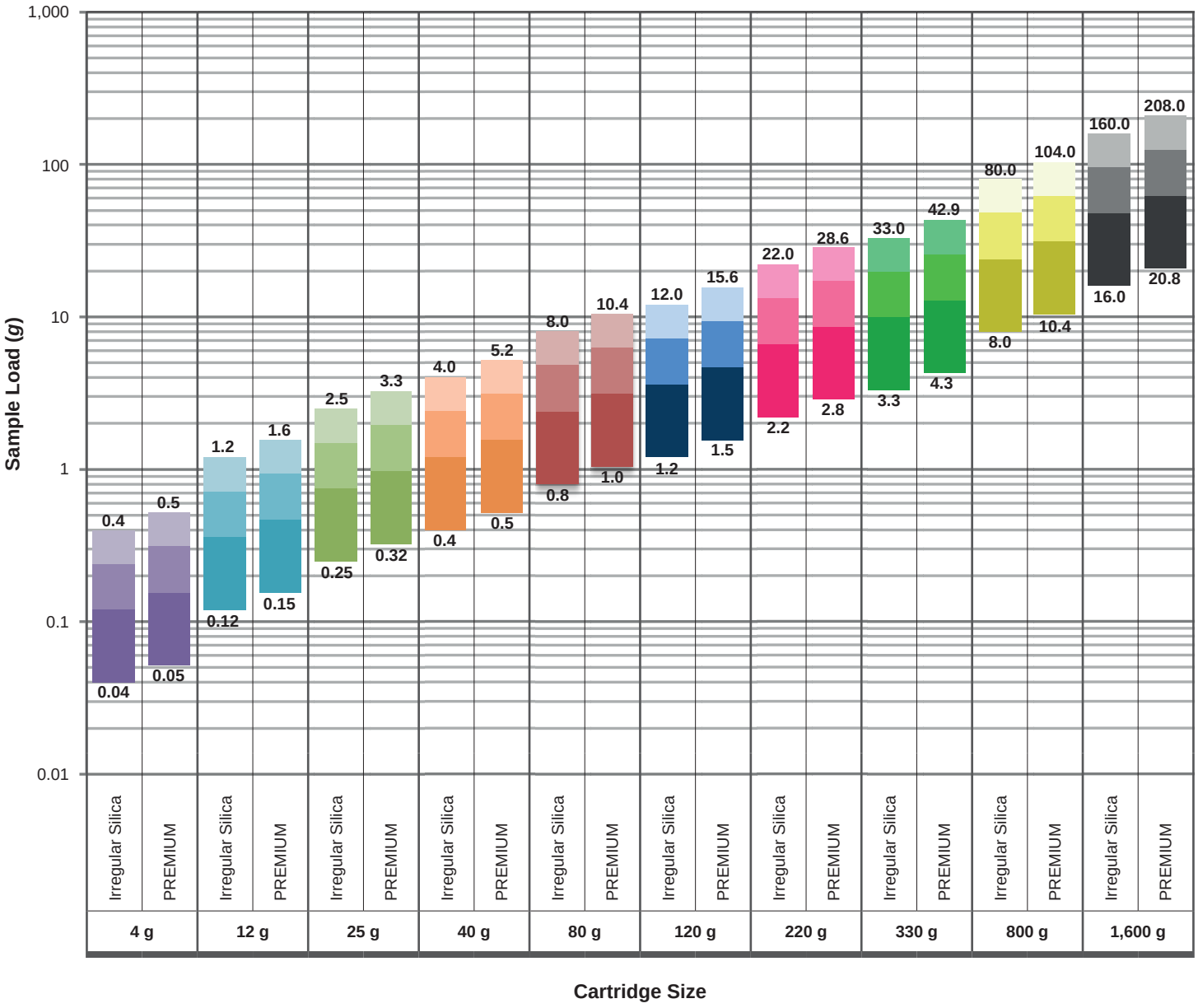
1 and 2 are well retained and separated in flash chromatography

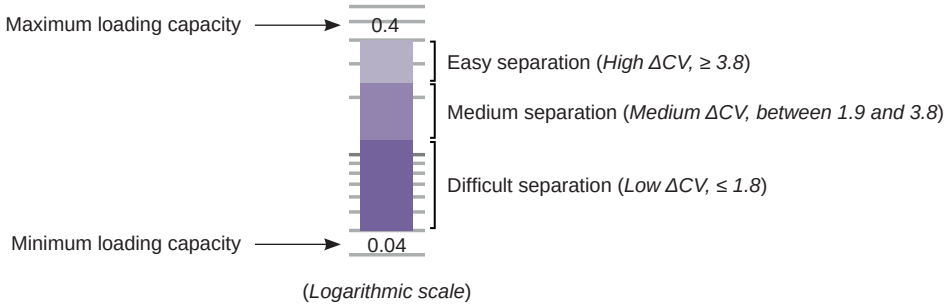
<u>In summary</u>:

- **The lower the Rfs, the greater ΔCV**
- **The greater the ΔCVs, the greater the separation and resolution between the spots (*easier separation*)**
- **The greater the ΔCVs, the more sample can be loaded onto the column**

Low Pressure Chromatography Loading Chart

The chart below will help you choose the right cartridge size according to your sample size and your TLC results.





The loading capacity depends on the sample itself, the column dimensions and the silica type. You will find below the sample loading we recommand with our Silia*Sep* flash cartridges. For easily separated compounds (*ΔCV > 6*) we suggest to load up to 5 % on irregular bonded phases, up to 10 % on bare irregular silica and up to 15 % on bare spherical silica.

Low Pressure Chromatography Loading Chart

Dimensions ID × Length	Silia*Sep* Format	Silia*Sep* Phase	Load (*g*)									
			Difficult Separation			Medium Separation			Easy Separation			
			ΔCV = 0.1 - 0.6	ΔCV = 0.7 - 1.2	ΔCV = 1.3 - 1.8	ΔCV = 1.9 - 2.4	ΔCV = 2.5 - 3.1	ΔCV = 3.2 - 3.8	ΔCV = 3.9 - 4.5	ΔCV = 4.6 - 5.2	ΔCV = 5.3 - 6.0	ΔCV > 6
12 × 98 mm	4 g	Irregular Silica	0.040	0.080	0.120	0.160	0.200	0.240	0.280	0.320	0.360	0.400
		PREMIUM	0.052	0.104	0.156	0.208	0.260	0.312	0.364	0.416	0.468	0.520
		Bonded	0.020	0.040	0.060	0.080	0.100	0.120	0.140	0.160	0.180	0.200
21 × 117 mm	12 g	Irregular Silica	0.120	0.240	0.360	0.480	0.600	0.720	0.840	0.960	1.080	1.200
		PREMIUM	0.156	0.312	0.468	0.624	0.780	0.936	1.092	1.248	1.404	1.560
		Bonded	0.060	0.120	0.180	0.240	0.300	0.360	0.420	0.480	0.540	0.600
21 × 165 mm	25 g	Irregular Silica	0.250	0.500	0.750	1.000	1.250	1.500	1.750	2.000	2.250	2.500
		PREMIUM	0.325	0.650	0.975	1.300	1.625	1.950	2.275	2.600	2.925	3.250
		Bonded	0.125	0.250	0.375	0.500	0.625	0.750	0.875	1.000	1.125	1.250
27 × 169 mm	40 g	Irregular Silica	0.400	0.800	1.200	1.600	2.000	2.400	2.800	3.200	3.600	4.000
		PREMIUM	0.520	1.040	1.560	2.080	2.600	3.120	3.640	4.160	4.680	5.200
		Bonded	0.200	0.400	0.600	0.800	1.000	1.200	1.400	1.600	1.800	2.000
31 × 237 mm	80 g	Irregular Silica	0.800	1.600	2.400	3.200	4.000	4.800	5.600	6.400	7.200	8.000
		PREMIUM	1.040	2.080	3.120	4.160	5.200	6.240	7.280	8.320	9.360	10.400
		Bonded	0.400	0.800	1.200	1.600	2.000	2.400	2.800	3.200	3.600	4.000
36 × 256 mm	120 g	Irregular Silica	1.200	2.400	3.600	4.800	6.000	7.200	8.400	9.600	10.800	12.000
		PREMIUM	1.560	3.120	4.680	6.240	7.800	9.360	10.920	12.480	14.040	15.600
		Bonded	0.600	1.200	1.800	2.400	3.000	3.600	4.200	4.800	5.400	6.000
60 × 195 mm	220 g	Irregular Silica	2.200	4.400	6.600	8.800	11.000	13.200	15.400	17.600	19.800	22.000
		PREMIUM	2.860	5.720	8.580	11.440	14.300	17.160	20.020	22.880	25.740	28.600
		Bonded	1.100	2.200	3.300	4.400	5.500	6.600	7.700	8.800	9.900	11.000
60 × 268 mm	330 g	Irregular Silica	3.300	6.600	9.900	13.200	16.500	19.800	23.100	26.400	29.700	33.000
		PREMIUM	4.290	8.580	12.870	17.160	21.450	25.740	30.030	34.320	38.610	42.900
		Bonded	1.650	3.300	4.950	6.600	8.250	9.900	11.550	13.200	14.850	16.500
78 × 382 mm	800 g	Irregular Silica	8.000	16.000	24.000	32.000	40.000	48.000	56.000	64.000	72.000	80.000
		PREMIUM	10.400	20.800	31.200	41.600	52.000	62.400	72.800	83.200	93.600	104.000
		Bonded	4.000	8.000	12.000	16.000	20.000	24.000	28.000	32.000	36.000	40.000
104 × 429 mm	1,600 g	Irregular Silica	16.000	32.000	48.000	64.000	80.000	96.000	112.000	128.000	144.000	160.000
		PREMIUM	20.800	41.600	62.400	83.200	104.000	124.800	145.600	166.400	187.200	208.000
		Bonded	8.000	16.000	24.000	32.000	40.000	48.000	56.000	64.000	72.000	80.000
			Difficult Separation			Medium Separation			Easy Separation			

For alumina sorbent, refer to the bare silica loading capacity.

It is worth noting that unlike with normal phases where it is easy to determine and optimize separation conditions using TLC plates, in reversed-phase chromatography the same cannot be said. First, the functions are impregnated on a TLC plate while they are bounded on SiliaBond products, thus the compounds do not have the exact same behaviors interacting with these products. Secondly, the plates are dry when the product is applied. However, the silica is already wetted in a chromatographic column. Since a reversed-phase is harder to wet than a normal phase, less contact area is available which tends to cause the products to migrate with the solvent front. Finally, the sample application is harder on a hydrophobic surface.

With this in mind, a general idea of the elution conditions can be found with some adjustments needed when transposing these onto a flash cartridge.

Silia*Sep* Cartridges Cleaning and Re-Use

Pre-packed flash cartridges are designed and typically used for a single purification run (*1-injection*). Single-use gives the highest purification performance and the lowest solvent consumption. It is typically the easiest process to validate and it may give the lowest purification process cost.

It is possible to develop and validate a cleaning process that meets FDA requirements, so the flash cartridge can be used for multiple runs. This cleaning process is the client's responsibility. SiliCycle does not warranty any flash cartridge for multiple injections and all process validation is under the client's (*sole*) responsibility.

Guidelines for Flash Cartridge Use in cGMP Environments	
Silia*Sep* Phase	**Recommended Use and Cleaning Procedure**
Bare Silica **(*normal phase separations*)**	Porous silica is used in adsorption chromatography processes, where the product and its impurities "bind" to the surface with varying degrees of affinity. The solvent polarity is increased to desorb the product and its impurities at different elution volumes. While it is possible to elute nearly all the product from silica, some impurities typically remain at the end of each separation. If the cartridge is not fully cleaned, this remaining material may reduce the purification effectiveness and these impurities may elute in a subsequent run. Clearly, if the cartridge is planned to be used for a second or subsequent run, the process will require a validated cleaning protocol. Some guidelines are given below: • **Single injection of a single batch of one API** In this case, the cartridge is eluted and the purified product is collected. The cartridge is flushed and then discarded. This single-use process has the minimum solvent consumption and no-risk of cross-contamination. • **Multiple injections of a single batch of one API** In this process, the full batch is too large to purify in a single run, and therefore multiple runs are required. Each injection is from a single batch or lot, and therefore the product and its impurities are identical in each injection or sample load. The cartridge must be cleaned between runs, but no cross-contamination is possible between batches. Re-using silica cartridges for multiple injections within a single batch is a well accepted process decision. It must be demonstrated that each of the multiple injections gives the same elution profile and that the product purity is consistent in each of the sequential runs. Typically, the process control points are set to ensure that the impurity profile does not change more than 0.1 %. This process can be modeled at the lab or pilot scale and then demonstrated at full production volume. In this process, the cleaning solvent is often 100 % of the most polar solvent in the elution mixture and is often carried out in reverse flow mode. The cartridge must be re-equilibrated, in normal flow mode, with the initial solvent conditions prior to the next injection. The cleaning step and re-equilibration step will each use a minimum of 3 column volumes of each solvent. • **Multiple batches of one API with single or multiple injections** Silica is rarely used for multiple batches of a single API, due the high cost and technical risk of batch-to-batch contamination. If a multiple lot strategy is nonetheless considered, the cleaning process will require a high level of data to support the decision.* • **Multiple batches of multiple APIs** This multiple product cleaning protocol would require an extremely high level of data and would still have significant risks of cross contamination. The cost of cleaning and validation would also be very high. It is recommended that flash cartridges be dedicated to an individual API and never be used for multiple API compounds.
C18 **(*reversed-phase separations*)**	Reversed-phase media is often used for multiple batches of a single API. However, due to the high cost and technical risk of batch-to-batch contamination, a fully validated cleaning procedure must be implemented. If a multiple lot strategy is nonetheless considered, the cleaning process will require a high level of data to support the decision.* The cleaning protocol can be modeled at the lab or pilot scale and then demonstrated at full production volume. In this process, the cleaning solvent is often 100 % of the most polar solvent in the elution mixture (*typically methanol, ethanol or acetonitrile*), and is often carried out in reverse flow mode. The cartridge must be re-equilibrated, in normal flow mode, with the initial solvent conditions prior to the next injection. The cleaning step and re-equilibration step will each use a minimum of 3 column volumes of each solvent. It is recommended that C18 flash cartridges be dedicated to an individual API and never be used for multiple API compounds.

* The data set must include analytical methods, such as HPLC and/or GC, and data to determine residue analysis. The standard assay is Total Organic Carbon (*TOC*) analysis. The upper and lower control limits must be set and defined for this process. The FDA does not set a number, but many organizations have used 1/1000 of a therapeutic dose of Product A in Product B as a guideline. This is a very challenging requirement, and the cost of cleaning solvents and time invested may be prohibitive.

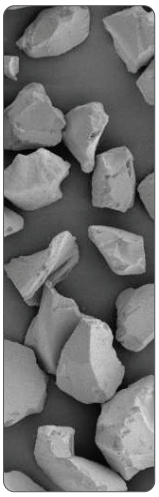
SiliaSep Cartridges Ordering Information

To build your own product number, just add the Format Code to the Phase PN: **FLH-[Phase PN]-[Format Code]**
Example: SiliaSep C18 40 - 63 µm irregular grade, 4 g cartridge: FLH-R33230B-ISO04.

SiliaSep Phases

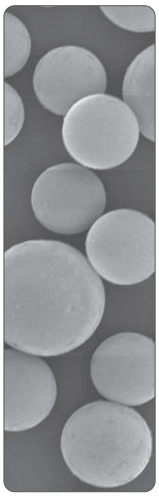
SiliaSep 40 - 63 µm Irregular Silica

SiliaSep Phases		
Phases		Phase PN
	Bare Irregular Silica	R10030B
	Acidic Alumina	AUT-0053
	Neutral Alumina	AUT-0054
	Basic Alumina	AUT-0055
FUNCTIONALIZED IRREGULAR SILICA	C18	R33230B
	C8	R30830B
	Phenyl	R34030B
	PFP	R67530B
	Amine	R52030B
	Diol *nec*	R35030B
	Cyano	R38030B
	SCX	R60530B
	SCX-2	R51230B
	SAX *nec*	R66530B
	SAX-2 *nec*	R66430B



SiliaSep PREMIUM 25 µm Spherical Silica

SiliaSep PREMIUM Phases		
Phases		Phase PN
	Bare Spherical Silica	10095D-A
FUNCTIONALIZED SPHERICAL SILICA	C18	03295D-A
	C8	30895D-A
	Phenyl	34095D-A
	PFP	67595D-A
	Amine	52095D-A
	Diol *nec*	35095D-A
	Cyano	38095D-A
	SCX	60595D-A
	SCX-2	51295D-A
	SAX *nec*	66595D-A
	SAX-2 *nec*	66495D-A



Note: Other phases could be offered, like metal scavengers. Contact us for more information: info@silicycle.com.

Video: Timelapse of a separation of dyes

SiliaSep Formats

SiliaSep Formats				
Formats	Qty / Box Bare Silica	Qty / Box Functionalized Silica*		Format Code
		Standard Box	Big Box	
4 g	20	2	10	ISO04
12 g	20	1	10	ISO12
25 g	15	1	10	ISO25
40 g	15	1	5	ISO40
80 g	12	1	5	ISO80
120 g	10	1	5	IS120
220 g	4	1	4	IS220
330 g	4	1	4	IS330
XL 800 g	2	1	-	IS750
XL 1,600 g	2	1	-	I1500
XL 3 kg	1	-	-	ISO3KG
XL 5 kg	1	-	-	ISO5KG
XL 10 kg	1	-	-	ISO10KG

* Add -B to the part number for the big boxes (*eg: FLH-03295D-A-ISO04-B*).

Notes:

- For bigger columns, please contact us.
- For SiliaSep XL formats, you may need to use an XL Adapter to connect the cartridge onto your system. Part number: AUT-0127-2.

Silia*Sep* Solid-Load Cartridges and Plungers

The use of solid-load technique (*also called dry-load*) is known to improve chromatography resolution, especially for compounds soluble only in strong solvents or in large volumes. Our plungers and solid-load cartridges are designed to allow injections in a dry form on Silia*Sep* flash cartridges.

Silia*Sep* Solid-Load Cartridges

Four formats of solid-load cartridges are available: 10, 55, 150 and 700 mL (*SiliCycle offers adapted plungers for the smaller ones - 10 and 55 mL- but not for the larger ones - 150 and 700 mL**). The open top of the cartridge receives the bottom end of the plunger, and the luer-lock bottom connects to the flash cartridge.

Our solid-load cartridges are available either empty (*to be filled with your own media*) or pre-packed (*with 2, 5, 10, 20, 65* or 270* g of media*), whichever best suits your needs.

* These two solid-load cartridges formats (*150 and 700 mL*) are compatible with the large-scale plunger of the CombiFlash Torrent® system.

Silia*Sep* Solid-Load Cartridge Installation

A. EMPTY solid-load cartridges (*to concentrate the sample and eliminate any solvent effect on the purification*)

1. Mix your sample with bulk silica to make a silica-sample slurry.
   For a dry sample use a 1:1 ratio (*1 g of silica for 1 g of dry sample*) and for an oily sample use a 3:1 ratio (*3 g of silica for 1 g of oily sample*).
2. Dry the slurry by evaporating the solvent.
3. Transfer the dried silica-sample powder in the empty solid-load cartridge and insert the lose frit.
4. Connect the solid-load cartridge to the plunger (*top*) and the flash cartridge (*bottom*).

55 mL empty solid-load cartridge

10 mL empty solid-load cartridge

B. PRE-PACKED solid-load cartridges (*to allow direct injection of liquid samples*)

1. Inject your liquid sample into the solid-load cartridge.
   You should be able to dilute your sample in 1 column volume at the most.
   If not, choose a bigger pre-packed solid-load cartridge.
2. Remove the dissolution solvent by gravity and / or heating.
3. Connect the solid-load cartridge to the plunger (*top*) and the flash cartridge (*bottom*).

55 mL pre-packed solid-load cartridge

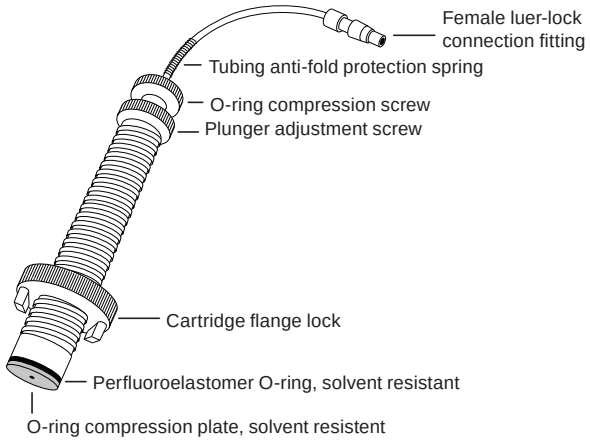
These pre-packed solid-load cartridges are available with various SiliCycle's standard stationary phases, mainly: Silica, C18, Amine, Cyano and Diol (*see Table on the following page for more information*).

10 mL pre-packed solid-load cartridge

SiliaSep Plungers

SiliCycle offers two plunger sizes: 10 and 55 mL. Connect the female luer-lock connection fitting to the flash system (*solvent inlet*) and insert the bottom part of the plunger inside the solid-load cartridge.

Both the compression plate and the perfluoroelastomer O-ring are solvent resistant, which permits the use of any mobile phase for the purification.



SiliaSep Solid-Load Cartridges and Plungers Ordering Information

SiliaSep Solid-Load Cartridges				
Product Number	**Sorbent**	**Weight / Volume**	**Description**	**Qty / Box**
AUT-0060-010	-	-	Plunger for 10 mL Solid Load Cartridge (*16 mm*)	1
AUT-0060-055	-	-	Plunger for 55 mL Solid Load Cartridge (*23 mm*)	1
SPL-R10030B-10U	Silica (*40 - 63 µm*)	2 g / 10 mL	SiliaSep Silica Pre-packed Solid-Load Cartridge, 2 g, 10 mL	20
SPL-R10030B-10X	Silica (*40 - 63 µm*)	5 g / 10 mL	SiliaSep Silica Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R10030B-55Y	Silica (*40 - 63 µm*)	10 g / 55 mL	SiliaSep Silica Pre-packed Solid-Load Cartridge, 10 g, 55 mL	16
SPL-R10030B-55Z	Silica (*40 - 63 µm*)	20 g / 55 mL	SiliaSep Silica Pre-packed Solid-Load Cartridge, 20 g, 55 mL	16
SPL-R10030B-065*	Silica (*40 - 63 µm*)	65 g / 150 mL	SiliaSep Silica Pre-packed XL Solid-Load Cartridge, 65 g, 150 mL*	12
SPL-R10030B-270*	Silica (*40 - 63 µm*)	270 g / 700 mL	SiliaSep Silica Pre-packed XL Solid-Load Cartridge, 270 g, 700 mL*	6
SPL-R52030B-10X	Amine	5 g / 10 mL	SiliaSep Amine Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R52030B-55Z	Amine	20 g / 55 mL	SiliaSep Amine Pre-packed Solid-Load Cartridge, 20 g, 55 mL	16
SPL-R35030B-10X	Diol	5 g / 10 mL	SiliaSep Diol Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R35030B-55Z	Diol	20 g / 55 mL	SiliaSep Diol Pre-packed Solid-Load Cartridge, 20 g, 55 mL	16
SPL-R38030B-10X	Cyano	5 g / 10 mL	SiliaSep Cyano Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R38030B-55Z	Cyano	20 g / 55 mL	SiliaSep Cyano Pre-packed Solid-Load Cartridge, 20 g, 55 mL	16
SPL-R33230B-10X	C18	5 g / 10 mL	SiliaSep C18 Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R33230B-55Z	C18	20 g / 55 mL	SiliaSep C18 Pre-packed Solid-Load Cartridge, 20 g, 55 mL	16
SPL-0009-010	Empty	- / 10 mL	SiliaSep Empty Solid-Load Cartridge, 10 mL (*with 200 frits*)	100
SPL-0012-055	Empty	- / 55 mL	SiliaSep Empty Solid-Load Cartridge, 55 mL (*with 200 frits*)	100
AUT-0090-150*	Empty	- / 150 mL	SiliaSep Empty Solid-Load Cartridge, 150 mL (*with 24 frits*)*	12
AUT-0090-700*	Empty	- / 700 mL	SiliaSep Empty Solid-Load Cartridge, 700 mL (*with 12 frits*)*	6

* These two solid-load cartridges formats (*150 and 700 mL*) are compatible with the large-scale plunger of the CombiFlash Torrent® system.

Notes: For optimal purification performance, solvent removal under vacuum is highly recommended.

Other phases can be offered pre-packed in our solid-load cartridges, contact us for more information: info@silicycle.com.

Silia*Sep* OT (*Open Top*) Flash Cartridges

Silia*Sep* OT cartridges are mainly used with vacuum manifolds and automated SPE equipments.
They are also directly compatible with FlashMaster™ systems.

Ordering Information

To build your own product number, just add the Format Code to the Phase PN: **FLH-[Phase PN]-[Format Code]**
Example: Silia*Sep* OT C18, 15 g cartridge: FLH-R00230B-70i.

Silia*Sep* OT Phases

Phase	Phase PN
Bare Silica	**R10030B**
C18	**R00230B**
C8	**R31030B**
Phenyl	**R34030B**
PFP	**R67530B**
Amine	**R52030B**
Diol *nec*	**R35030B**
Cyano	**R38030B**
SCX	**R60530B**
SCX-2	**R51230B**
SAX *nec*	**R66530B**
SAX-2 *nec*	**R66430B**

Silia*Sep* OT Formats

Format	Qty / Box	Dimensions (*ID* × *length*) (*mm*)	Format Code	
			Bare Silica	Functionalized Silica
2 g / 12 mL	20	15.8 × 90	**15U**	**SPE-[Phase PN]-12U**
5 g / 25 mL	20	20.5 × 100	**25X**	**SPE-[Phase PN]-20X**
10 g / 70 mL	16	26.8 × 154	**70Y**	
15 g / 70 mL	16	26.8 × 154	**70i**	
20 g / 70 mL	16	26.8 × 154	**70Z**	
25 g / 150 mL	10	38.2 × 170	**95K**	
50 g / 150 mL	10	38.2 × 170	**95M**	
70 g / 150 mL	10	38.2 × 170	**95N**	

Notes:

- Other phases could be offered, contact us for more information: info@silicycle.com.
- Silia*Sep* OT cartridges are also available with bar code for automation purposes.
- Maximum operating pressure: 60 psi.

Thin Layer Chromatography (*TLC*)

- **Wide choice of sizes, sorbents, and thicknesses available**
- **Excellent reproducibility between Silia*Plate* TLC plates and bulk silicas or flash cartridges**

The hardness of our silica layer, combined to a homogeneous coating and layer thickness, allows excellent separations. Each TLC batch is chemically and physically controlled by our Quality Control department to ensure lot-to-lot and layer-to-layer reproducibility.

Silia*Plate* TLC Plates

Thin-layer chromatography (*TLC*) is a quick, simple and inexpensive analytical technique frequently used in various laboratories as it is one of the most verstatile. It is used for reaction monitoring, screening, and compound purity evaluation.

Rapid and cost-efficient selection and optimization of chromatographic conditions prior to flash chromatography purification or HPLC analysis.

Besides speed and low cost, TLC analysis presents other non-negligible advantages like the small quantity of compound required and high sample throughput capability (*up to 20 samples simultaneously*).

Like column chromatography, TLC is a solid-liquid partitioning technique, in which the sample is applied to the plate as a small spot near the base of the plate. The moving liquid phase is then allowed to ascend the plate, causing the sample to partition between moving and stationary phase.

Silia*Plate* Features and Benefits

For more than 20 years, SiliCycle has been offering a wide selection of TLC plates in various sizes (*plate size, thickness, backing*) and chemistries (*10 % Silver Nitrate, CN, C18, NH_2*). Silia*Plate* represents an efficient and economical alternative to other TLC plate manufacturers while demonstrating high separation power, which is due to our narrow particle size distribution silica gel.

The extraordinary silica layer hardness combined to a homogeneous coating and layer thickness allows excellent separation. Each TLC batch is chemically and physically controlled by our Quality Control department to ensure lot-to-lot and layer-to-layer reproducibility.

Selection Guide

Plate Types

SiliCycle offers different types of plates for thin-layer chromatography applications: classical TLC and preparative TLC (*PLC*). The plate types are selected based on the analysis required and the available budget.

Differences Between Classical TLC and PLC

Properties	Classical TLC	Preparative (*PLC*)
Applications	Quick, inexpensive, flexible and classical separations	Purification on a TLC plate
Analysis	Qualitative	Quantitative
Detection	UV - Stains	UV
Distribution [*Mean Particle Size*]	5 - 20 µm [*10 - 14 µm*]	5 - 40 µm [*22 - 25 µm*]
Layer Thickness	200 - 250 µm	500 - 2,000 µm
Typical Sample Volume	1 - 5 µL	5 - 20 µL

TLC Backings

TLC plates are available with different backings: rigid (*glass-backed*) and flexible (*aluminum & plastic-backed*).

TLC Backings Comparison

Properties	Glass	Aluminum	Plastic
Advantages	• Rigid • High chemical resistance • High heating stability and charring resistance • Transparent	• Thin and low fragility • Low weight & consequent shipping costs • High heating stability • Possible to cut with scissors • Can be stored in notebook	• Thin • Low fragility • Possible to cut with scissors • High chemical resistance • Can be stored in notebook
Disadvantages	• Thick and high fragility • Impossible to cut with scissors • Cannot be stored in lab notebook • High weight & consequent shipping costs • Large shelf space	• Low chemical resistance • Opaque	• Medium weight • Opaque • Heating stability up tp 175°C • Possible cracking of matrix due to high flexibility
Thickness (*approx.*)	2.0 - 2.5 mm	1.5 - 2.0 mm	1.5 - 2.0 mm
Heating Stability	High	High	Below 175°C
Fragility	High	Low	Low
Cutting with Scissors	Impossible	Easily	Possible
Chemical Resistance	High	Low	High

Layer Thicknesses

The layer thickness is related to the nature of the analysis (*analytical or preparative*) as well as the performance of the plate. The most common layer thicknesses are 200 - 250 µm (*analytical TLC plates*), and 500 - 2,000 µm (*preparative TLC plates*)

UV Indicator

Silia*Plate* TLC Plates could contain inorganic fluorescent indicator for UV detection of colorless substances: F254 (*manganese activated zinc silicate with absorption maximum at 254 nm; green fluorescence*). Therefore, samples which absorb short-wave UV at 254 nm (*F254*) can be viewed under UV light. As a matter of fact, substances absorbing in the respective region of wavelength cause a quenching of the fluorescence and appear as dark spots on the TLC.

Channeled

Those TLC plates have ‘channels’ (*alternating zones with and without silica*) which allows to separate several samples and prevent cross contamination from a sample to another.

Preadsorption Zone

Silia*Plate* TLC Plates with preadsorption zone allow to load quickly and easily samples, even large volumes of diluted samples. The adsorbent in the preadsorption zone is a large pore concentrating adsorbent and the other one is a selective layer for separation.

With this kind of plates, the sample always concentrate in a narrow band (*no matter the way sample is loaded; spots do not have to be the exact same size or on the exact same line*).

Matrices (*or Adsorbents*)

Various adsorbents can be used for TLC coating; silica, aluminum oxide, Florisil®, etc. For more than 20 years, SiliCycle has been offering a wide selection of TLC plates in various sizes (*plate size, thickness, backing*) and chemistries (*Silver Nitrate, CN, C18, NH_2*).
More than 80 % of all purifications are performed using silica gel as the adsorbent.

Available Matrices

Silica Gel	Aluminum Oxide
Can be unmodified or functionalized, and suitable for a myriad of molecules of functionalities & polarities, such as aflatoxins, alkaloids, barbiturates, fatty acids, flavonoids, glycosides, lipids, nucleosides, proteins, pesticides, sweeteners, vitamins and so on.	Aluminum oxide (*commonly called Alumina*) is the second most commonly used matrix, and shows similar selectivity to that of silica, with 3 different pH ranges (*basic, neutral, acidic*). Popular applications include the separation for alkaloids, aliphatic compounds, aromatics, steroids, etc.

Binder

SiliCycle offers two types of binder, with different sensitivities and areas of applications: 'B' and 'BK'

- **B**'s layer is polymeric: a small percentage of inorganic, hardening agent has been added for a uniform and hard surface, smooth and dense, that will not crack, blister or swell up. They were designed for maximum robustness of the binder: they are very easy to handle and to write on, as well as completely wettable. They are compatible with all solvents, yet, they might oxidize a bit faster when dipped into $KMnO_4$ (*fading in a few minutes from flashy purple to yellow ocher*). Also, spots are a bit less definite when using CAM as a revelatory. Such binder also contains a higher percentage of fluorescent indicator for greater brilliance of spots and less background noise from the silica layer.

- **BK's** layer is gypsum (*calcium sulfate*), and do not contain the polymeric additive that provides the former plates a harder surface and ruggedness. This means that the layer is softer, so spots can be easily scrapped off from the glass support, and are particularly recommended for aggressive visualization methods (*strong charring, CAM staining solution*) or, if dipped into $KMnO_4$, ought to remain bright-purple a longer period of time.

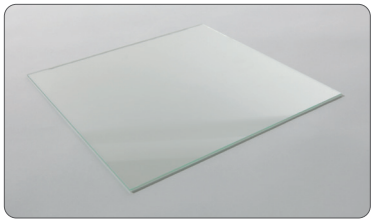
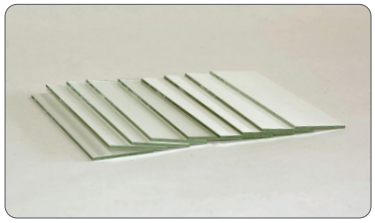
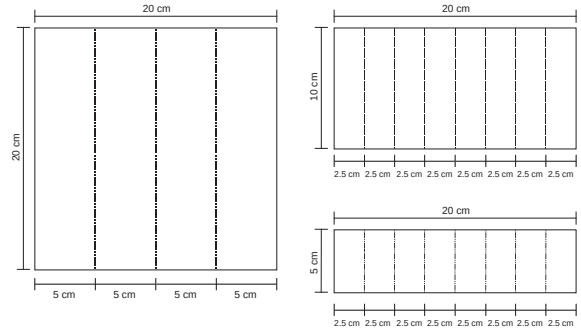
Here is a chart which can hopefully help you quickly choose the right plate for your specific application.

Silia*Plate* TLC Plates Binders		
TLC Plate Binder	**B**	**BK**
Example	**TLG-R10014B-323**	**TLG-R10014BK-323**
UV Fluorescence (F_{254})	Higher brightness Less background noise from layer	Yes
Binder Sensitivity	Stable in almost all organic solvents	
	Increased separation efficiency	Resistant to aggressive visualization methods
Surface Layer	Robust and rugged	Easily scratched off
Water Tolerance	Up to 80 %	Up to 40 %
Specific Surface (*BET*)	≈ 500 m^2/g	
Mean Pore Size	60 Å	
Mean Pore Volume	0.75 mL/g	
Distribution (*Mean Particle Size*)	5 - 20 µm [10 - 14 µm]	
Layer Thickness	≈ 250 µm	
Stain Compatibility		
$KMnO_4$	Compatible	**Highly compatible**
CAM	Compatible	
p-Anisaldehyde	Compatible	**Highly compatible**
Ninhydrin	**Highly compatible**	
Vanilin	**Highly compatible**	

Sorbents

Available Sorbents		
Classical Silica Gel	**Reversed-Phases**	**Special Phases**
A universal matrix for daily, fast, reliable analysis of the largest spectra of molecules The particle size distribution used for the silica is related to the nature of the plate. For standard TLC, silica gel with a mean particle size of 10 - 14 µm is used. In both cases, pore diameter is always 60 Å.	**The two most popular modes of separation employed in TLC are normal and reversed-phases.** In normal phase separation, the mobile phase is less polar than the stationary phase. Inversely, in reversed mode, the mobile phase (*usually a mixture of water and organic solvent*) is more polar than the stationary phase (*C18*). When satisfactory separations cannot be achieved by unmodified silica, other functionalized matrices have been designed for specific applications:	
	C2, C8 and C18 phases are functionalizations of silica performed using organosilanes of various chain lengths. Retention of molecules & ability to tolerate water in the moving phase are directly dependent on the chains length.	Diol and Cyano (*CN*) are moderately polar. They can thus be suitable for both normal and reversed-phase chromatography, depending on your application. Amino phases (NH_2) show weak anion exchange characteristics, great for charged compounds.

Plate Sizes

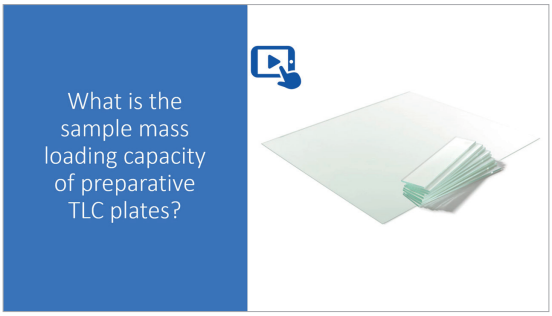
Available Sizes		
Standard TLC Plates	**Micro TLC Plates**	**Scored TLC Plates**
Silia*Plate* TLC plates are available in the following standard sizes depending on the coating used: 20 × 20 cm 10 × 20 cm 5 × 20 cm 5 × 10 cm 10 × 10 cm Example: 	Also for your convenience, SiliCycle provides ready-to-use micro TLC plates in the following formats: 2.5 × 10 cm 2.5 × 7.5 cm 2.5 × 5 cm Example: 	An interesting compromise between standard and micro plate sizes is our Scored Silia*Plate* *(glass backing)*. Three different formats are available and possible cut combinations are shown in the image below. 20 × 20 cm plates scored to four 5 × 20 cm plates *(or multiple of 5 cm width)* 10 × 20 cm plates scored to eight 2.5 × 10 cm plates *(or multiple of 2.5 cm width)* 5 × 20 cm plates scored to eight 2.5 × 5 cm plates *(or multiple of 2.5 cm width)* 

Silia*Plate* Ordering Information

Please note that this is an overview of plates that SiliCycle offers.
Different sizes are available, as well as more exotic layers for special separations (*chiral layers, layers for surfactant separations, for PAH analysis, layers for basic or acidic ion exchange, cellulose layers, etc.*). Contact us: info@silicycle.com.

Various combinations are possible with Silia*Plate* TLC plates and are summarized in the table below.

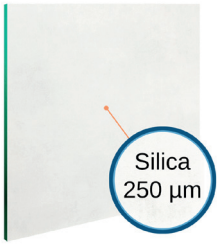
Silia*Plate* TLC Plates Portfolio		
Properties	**Analytical**	**Preparative**
Available Backings		
Glass	Yes	Yes
Aluminum	Yes	No
Plastic	Yes	No
Available Adsorbents		
Bare Silica	Yes	Yes
Functionalized Silica	No	Yes
Silica Specifications		
Mean Particle Size	10 - 14 µm	22 - 25 µm
Mean Pore Diameter	60 Å	60 Å
Type of Plate Available		
Scored Plate	Yes	Yes
Channeled Plate	Yes	No
Layer Thickness	Glass: 250 µm Flexible: 200 µm	Glass: • 500 µm • 1,000 µm Flexible: • 1,500 µm • 2,000 µm
Plate Size	• 2.5 × 5 cm • 2.5 × 7.5 cm • 2.5 × 10 cm • 5 × 10 cm • 5 × 20 cm • 10 × 20 cm • 20 × 20 cm	• 20 × 20 cm



Video: Sample mass loading rule of thumb for 20 × 20 cm plates

Glass-backed TLC Plates

Glass-backed Analytical TLC Plates					
PN	**Sorbent**	**Plate Size**	**Thickness**	**UV Indicator**	**Qty / Box**
Silica					
TLG-R10014B-124	Silica, Hard Layer	2.5 × 7.5 cm	250 µm	F254	100
TLG-R10014B-424	Silica, Hard Layer	5 × 20 cm	250 µm	F254	100
TLG-R10014B-323	Silica, Hard Layer	20 × 20 cm	250 µm	F254	25
TLG-R10014B-323N	Silica, Hard Layer	20 × 20 cm	250 µm	None	25
TLG-R10014BK-417	Silica, optimized for $KMnO_4$ revelation	2.5 × 5 cm	250 µm	F254	200
TLG-R10014BK-124	Silica, optimized for $KMnO_4$ revelation	2.5 × 7.5 cm	250 µm	F254	100
TLG-R10014BK-527	Silica, optimized for $KMnO_4$ revelation	5 × 10 cm	250 µm	F254	200
TLG-R10014BK-424	Silica, optimized for $KMnO_4$ revelation	5 × 20 cm	250 µm	F254	100
TLG-R10014BK-725	Silica, optimized for $KMnO_4$ revelation	10 × 20 cm	250 µm	F254	50
TLG-R10014BK-323	Silica, optimized for $KMnO_4$ revelation	20 × 20 cm	250 µm	F254	25
TLG-R10014BK-323N	Silica, optimized for $KMnO_4$ revelation	20 × 20 cm	250 µm	None	25
TLG-R10014BKB-323	Silica, optimized for $KMnO_4$ revelation	20 × 20 cm	250 µm	F254 , F366	25
Channeled with preadsorbent zone					
TLGCZ-R10011B-423	Silica	5 × 20 cm	250 µm	F254	25
TLGCZ-R10011B-723	Silica	10 × 20 cm	250 µm	F254	25
TLGCZ-R10011B-323	Silica	20 × 20 cm	250 µm	F254	25
TLGCZ-R10011B-323N	Silica	20 × 20 cm	250 µm	None	25
Scored TLC plates					
TLGSR10011B-423	Silica	5 × 20 cm, scored to 2.5 × 5 cm	250 µm	F254	25
TLGSR10011B-424	Silica	5 × 20 cm, scored to 2.5 × 5 cm	250 µm	F254	100
TLGSR10011B-723	Silica	10 × 20 cm, scored to 2.5 × 10 cm	250 µm	F254	25
TLGSR10011B-323	Silica	20 × 20 cm, scored to 5 × 20 cm	250 µm	F254	25
Functionalized silica & other adsorbents					
TLG-AUT0014-423	Florisil®	5 × 20 cm	250 µm	F254	25
TLG-AUT0014-723	Florisil®	10 × 20 cm	250 µm	F254	25
TLG-AUT0014-323	Florisil®	20 × 20 cm	250 µm	F254	25
TLG-AUT0337-323B	Basic Alumina	20 × 20 cm	250 µm	F254	25
TLG-AUT0337B-424N	Neutral Alumina	5 × 20 cm	250 µm	F254	100
TLG-AUT0337-323N	Neutral Alumina	20 × 20 cm	250 µm	F254	25
TLG-AUT0337-323NF	Neutral Alumina	20 × 20 cm	250 µm	None	25
TLG-AUT0337B-323N	Neutral Alumina	20 × 20 cm	250 µm	F254	25



Glass-backed TLC Plates

Glass-backed Analytical TLC Plates					
PN	**Sorbent**	**Plate Size**	**Thickness**	**UV Indicator**	**Qty / Box**
Functionalized silica & other adsorbents					
TLG-R30314BK-213	C18 (*100 %*), optimized for $KMnO_4$ revelation	10 × 10 cm	150 µm	F_{254}	25
TLG-R30314BK-213N	C18 (*100 %*), optimized for $KMnO_4$ revelation	10 × 10 cm	150 µm	None	25
TLG-R30411B-213	C18 (*13 %*)	10 × 10 cm	150 µm	F_{254}	25
TLG-R30411B-303	C18 (*13 %*)	20 × 20 cm	150 µm	F_{254}	25
TLG-R30414B-313	C18 (*13 %*)	20 × 20 cm	200 µm	F_{254}	25
TLG-R30411B-323	C18 (*13 %*)	20 × 20 cm	250 µm	F_{254}	25
TLG-R31011B-203	C8	10 × 10 cm	150 µm	F_{254}	25
TLG-R31011B-303	C8	20 × 20 cm	150 µm	F_{254}	25
TLG-R32611B-203	C2	10 × 10 cm	150 µm	F_{254}	25
TLG-R32611B-303	C2	20 × 20 cm	150 µm	F_{254}	25
TLG-R32614BK-313	C2, optimized for $KMnO_4$ revelation	20 × 20 cm	200 µm	F_{254}	25
TLG-R32614BK-713	C2, optimized for $KMnO_4$ revelation	10 × 20 cm	200 µm	F_{254}	25
TLG-R35011B-713	Diol	10 × 20 cm	200 µm	F_{254}	25
TLG-R35014BK-213	Diol, optimized for $KMnO_4$ revelation	10 × 10 cm	200 µm	F_{254}	25
TLG-R35014BK-713	Diol, optimized for $KMnO_4$ revelation	10 × 20 cm	200 µm	F_{254}	25
TLG-R35014BK-313	Diol, optimized for $KMnO_4$ revelation	20 × 20 cm	200 µm	F_{254}	25
TLG-R38011B-203	Cyano (*CN*)	10 × 10 cm	150 µm	F_{254}	25
TLG-R38011B-723	Cyano (*CN*)	10 × 20 cm	150 µm	F_{254}	25
TLG-R38011B-303	Cyano (*CN*)	20 × 20 cm	150 µm	F_{254}	25
TLG-R38014BK-213	Cyano (*CN*), optimized for $KMnO_4$ revelation	10 × 10 cm	200 µm	F_{254}	25
TLG-R38014BK-713	Cyano (*CN*), optimized for $KMnO_4$ revelation	10 × 20 cm	200 µm	F_{254}	25
TLG-R38014BK-313	Cyano (*CN*), optimized for $KMnO_4$ revelation	20 × 20 cm	200 µm	F_{254}	25
TLG-R52011B-203	Amine (NH_2)	10 × 10 cm	150 µm	F_{254}	25
TLG-R52011B-723	Amine (NH_2)	10 × 20 cm	150 µm	F_{254}	25
TLG-R52011B-303	Amine (NH_2)	20 × 20 cm	150 µm	F_{254}	25
TLG-R52014BK-213	Amine (NH_2), optimized for $KMnO_4$ revelation	10 × 10 cm	200 µm	F_{254}	25
TLG-R52014BK-713	Amine (NH_2), optimized for $KMnO_4$ revelation	10 × 20 cm	200 µm	F_{254}	25
TLG-R52014BK-313	Amine (NH_2), optimized for $KMnO_4$ revelation	20 × 20 cm	200 µm	F_{254}	25
TLG-R23511B-423	$AgNO_3$ (*10 %*)	5 × 20 cm	250 µm	F_{254}	25
TLG-R23511B-303	$AgNO_3$ (*10 %*)	20 × 20 cm	250 µm	F_{254}	25
TLG-R23611B-423	$AgNO_3$ (*15 %*)	5 × 20 cm	250 µm	F_{254}	25
TLG-R23611B-323	$AgNO_3$ (*15 %*)	20 × 20 cm	250 µm	F_{254}	25
TLG-R23711B-423	$AgNO_3$ (*20 %*)	5 × 20 cm	250 µm	F_{254}	25
TLG-R23711B-323	$AgNO_3$ (*20 %*)	20 × 20 cm	250 µm	F_{254}	25
TLG-R23M11B-323	$AgNO_3$ (*5-10-15-20 %, 5 TLC each*)	5 × 20 cm	250 µm	F_{254}	5 × 4
TLGSR1234511B-723	Trial Packing of Functionalized Silica	10 × 20 cm, scored to 2.5 × 10 cm	150 µm	F_{254}	25
TLG-AUT0308-203	RP Silanized	10 × 10 cm	150 µm	F_{254}	25

Glass-backed Preparative TLC Plates

Glass-backed Preparative TLC Plates					
PN	**Sorbent**	**Plate Size**	**Thickness**	**UV Indicator**	**Qty / Box**
Silica					
TLG-R10011B-333	Silica	20 × 20 cm	500 µm	F_{254}	25
TLG-R10011B-341	Silica	20 × 20 cm	1,000 µm	F_{254}	25
TLG-R10011B-353	Silica	20 × 20 cm	2,000 µm	F_{254}	25
Functionalized silica & other adsorbents					
TLG-AUT0337-343N	Neutral Alumina	20 × 20 cm	1,000 µm	F_{254}	25
TLG-AUT0337-343NF	Neutral Alumina	20 × 20 cm	1,000 µm	None	25
TLG-AUT0337-443	Neutral Alumina	5 × 20 cm	1,000 µm	F_{254}	25
TLG-AUT0337-443F	Neutral Alumina	5 × 20 cm	1,000 µm	None	25
TLG-AUT0337B-341N	Neutral Alumina	20 × 20 cm	1,000 µm	None	15
TLG-R23511B-433	$AgNO_3$ (*10 %*)	5 × 20 cm	500 µm	F_{254}	25
TLG-R23511B-333	$AgNO_3$ (*10 %*)	20 × 20 cm	500 µm	F_{254}	25
TLG-R23611B-433	$AgNO_3$ (*15 %*)	5 × 20 cm	500 µm	F_{254}	25
TLG-R23611B-333	$AgNO_3$ (*15 %*)	20 × 20 cm	500 µm	F_{254}	25
TLG-R23711B-433	$AgNO_3$ (*20 %*)	5 × 20 cm	500 µm	F_{254}	25
TLG-R23711B-333	$AgNO_3$ (*20 %*)	20 × 20 cm	500 µm	F_{254}	25
TLG-R30411B-341	C18 (*13 %*)	20 × 20 cm	1,000 µm	F_{254}	15
TLG-R30414BK-341	C18 (*15 %*), optimized for $KMnO_4$ revelation	20 × 20 cm	1,000 µm	F_{254}	15
Scored preparative TLC plates					
TLGSR10011B-333	Silica	20 × 20 cm, scored to 5 × 20 cm	500 µm	F_{254}	25
TLGSR10011B-341	Silica	20 × 20 cm, scored to 5 × 20 cm	1,000 µm	F_{254}	25
TLGSR10011B-353	Silica	20 × 20 cm, scored to 5 × 20 cm	2,000 µm	F_{254}	25

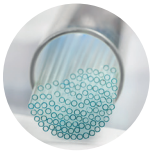
Aluminum-backed TLC Plates

Aluminum-backed Analytical TLC Plates					
PN	**Sorbent**	**Plate Size**	**Thickness**	**UV Indicator**	**Qty / Box**
Silica					
TLA-R10011B-005	Silica	4 × 8 cm	150 µm	F_{254}	50
TLA-R10011B-124	Silica	2.5 × 7.5 cm	200 µm	F_{254}	200
TLA-R10011B-323	Silica	20 × 20 cm	200 µm	F_{254}	25
TLA-R10011B-323N	Silica	20 × 20 cm	200 µm	None	25
TLA-R10011B-415	Silica	5 × 20 cm	200 µm	F_{254}	50
TLA-R10011B-515	Silica	5 × 10 cm	200 µm	F_{254}	50
TLA-R10011B-712	Silica	10 × 20 cm	200 µm	F_{254}	20
TLA-R10014BK-1112	Silica, optimized for $KMnO_4$ revelation	5 × 7.5 cm	200 µm	F_{254}	20
Functionalized silica & other adsorbents					
TLA-AUT0337-323N	Neutral Alumina	20 × 20 cm	200 µm	F254	25
TLA-AUT0337-323NF	Neutral Alumina	20 × 20 cm	200 µm	None	25
TLA-R30411B-005	Silica C18 (*13 %*)	4 × 8 cm	150 µm	F254	50
TLA-R30411B-303	Silica C18 (*13 %*)	20 × 20 cm	150 µm	F254	25
TLA-R30411B-405	Silica C18 (*13 %*)	5 × 20 cm	150 µm	F254	50
TLA-R30414BK-303	Silica C18 (*13 %*), opt. for $KMnO_4$ revelation	20 × 20 cm	150 µm	F254	25
TLA-R52014BK-005	Amine (NH_2), optimized for $KMnO_4$ revelation	4 × 8 cm	150 µm	F254	50

Plastic-backed TLC Plates

Plastic-backed Analytical TLC Plates					
PN	Sorbent	Plate Size	Thickness	UV Indicator	Qty / Box
Silica					
TLP-R10011B-005N	Silica	4 × 8 cm	150 µm	None	50
TLP-R10011B-117	Silica	2.5 × 7.5 cm	200 µm	F254	200
TLP-R10011B-323	Silica	20 × 20 cm	200 µm	F254	25
TLP-R10011B-323N	Silica	20 × 20 cm	200 µm	None	25
TLP-R10014B-0115	Silica	5 × 6.7 cm	200 µm	F254	50
TLP-R10014BK-0116	Silica, optimized for KMnO4 revelation	3.3 × 6.6 cm	200 µm	F254	50

TLC Accessories

TLC Accessories			
PN	Accessory		Qty / Box
AUT-0161	Rectangular TLC Developing Chamber		1
AUT-0161B	Replacement Lid for Rectangular Developing Chamber		1
AUT-0162	TLC Adsorbent Scraper		1
AUT-0163	TLC Spotting Capillaries		300
AUT-0182	Cutter for glass-baked TLC Plates (*up to 20 × 20 cm*)		1
AUT-0183	Replacement Scriber for TLC Plate Cutter		1
AUT-1182	TLC Plate Pencil Glass Cutter		1

Thin Layer Chromatography Practical Guide

Select a Stationary Phase

As almost 80 % of all separations can be performed using silica gel plates, it is suggested to try using this coating first. However, for acid sensitive compounds, alumina is probably a better choice (*useful for amine purification*). If you are working with highly polar compounds, reversed-phase mode is more suitable.

Select a Mobile Phase (*Solvent Systems*)

The selection of the mobile phase (*also called solvent system or eluent*) is perhaps the most important parameter to achieve efficient thin-layer chromatography separation. It is based on the compound's solubility with the solvent and the difference in the affinity for the mobile phase versus the stationary adsorbent (*silica or alumina*).

In normal phase chromatography, where non-polar solvents such as hexane or pentane are used, non-polar compounds will move up the plate while most polar compounds will stay on the baseline. Inversely, polar solvents will allow polar compounds to move off the origin. The most suitable solvent system is the one that moves all components off the baseline with Rf values between 0.15 and 0.85 (*ideally, close to 0.2 - 0.4*).

For most applications, a common solvent system to start with is EtOAc / Hexane (*1:1*). Varying the ratio can have a pronounced effect on the Rf. If it is not working, then try: *MeOH* / *DCM* (*2:8 - 10:90*); or toluene with acetone, EtOAc, or DCM.

Remember: in normal phases, to increase the compound's Rf, increase the polarity of the mobile phase; increase the ratio of the polar solvent or choose another solvent. Inversely, to decrease Rf, decrease the polarity of the eluent.

Rules of thumb

- Standard compounds (*most popular solvent system*): 10 - 50 % EtOAc / Hexane
- Polar compounds: 100 % EtOAc or 5 - 10 % MeOH / DCM
- Non-polar compounds: 5 % EtOAc (*or ether*) / Hexane or 100 % Hexane
- For basic compounds: (*amine or nitrogen containing*), it could be useful or required to add a small quantity of triethylamine (Et_3N) to the solvent mixture (*0.1 - 2.0 % but typical quantity is 0.1 %*) or 1 - 10 % ammonia (NH_3) in MeOH / DCM.
- For acidic compounds: it could be useful to add acetic (*AcOH*) or formic acid (*FA*) to the solvent mixture (*0.1 - 2.0 %*).

Reversed-phase mode

In reversed-phase chromatography, the typical solvent systems are:

- Mixtures of water or aqueous buffers and water miscible organic solvents such as acetonitrile (*ACN*), methanol and tetrahydrofuran (*THF*). Other solvents can be used such as ethanol (*EtOH*) and isopropanol (*IPA*).
- MeOH, to improve peak shape in flash chromatography, 0.1 % of acetic, formic or trifluoroacetic acid (*TFA*) can be added to the solvent system.

TLC Preparation & Interpretation

TLC Plate Preparation

Using a pencil, lightly draw a straight-line parallel to the width of the plate at about 1 cm from the base end of the plate. Sample application will be done on this line called baseline or origin.

Note: never use a pen because ink can move with some solvents used as eluent.

Sample Preparation

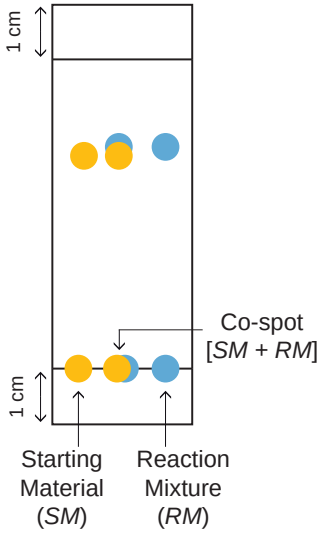
Thorough sample preparation is a prerequisite for an optimal and efficient TLC separation. Typical sample preparation processes could consist in a sample crushing, filtration, extraction or concentration of the product of interest.

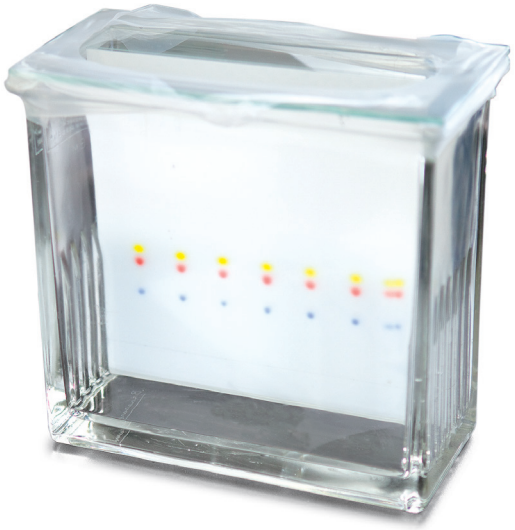
Sample Application

Sample preparation will differ depending on the nature of the plate (*analytical or preparative*). For analytical plates, because thin layer chromatography is extremely sensitive, it is really important to apply a small quantity using a glass capillary (*or a micro pipette*) to get optimal resolution. For preparative plates, apply a series of small adjacent spots to form a band or a streak using a glass capillary (*or a microliter syringe*). In both cases, a spotting guide can be used to facilitate sample application.

Co-spotting

For analytical chromatography, co-spotting is frequently used for similar polarity products.
This consists to apply on the same spot the starting material and reaction mixture, as shown by the image below.





TLC Plate Development

The most commonly used method to perform thin layer chromatography separation is to place vertically the TLC plate inside a sealed developing chamber to ensure solvent saturation. Place approximately 0.5 cm of the suitable solvent system inside the chamber. Slowly place the TLC inside the chamber and allow the eluent to travel up the plate until it gets to 1 cm from the top of the plate. Immediately remove the plate and draw a line along the solvent front.

Note: for optimal solvent saturation, a filter paper can be added inside the TLC chamber. This also prevents eluent evaporation. The solvent level needs to be below the baseline; otherwise the spots will be dissolved.

TLC Plate Visualization

If components of the reaction are colored, no visualization method is required (*spots can be seen directly on the silica layer*). However, most of the time it is not the case, therefore one of the methods described below should be used to reveal the spots.

Non-destructive methods

As a general visualization procedure, before treating the TLC plate with any destructive methods, UV-active compounds can be viewed under an ultraviolet lamp (*usually for polyconjugated compounds like benzophenones and anthracenes*). Furthermore, an iodine chamber can be useful for thiols, phosphines and alkenes but it works in about 50 % of cases for alkanes. It is recommended to circle the spots with a pencil on the TLC plate prior to visualization by destructive methods.

Destructive methods

For compounds that are not UV-active, there are several varieties of stains that can be used depending on the nature of the compound of interest. To use a stain, simply dip the TLC plate into the staining solution as quickly as possible, and then immediately absorb the excess stain with paper and heat carefully with a heat gun or on a hot plate at 110°C until spots are revealed.

Chromatogram Interpretation

Retention factor (*Rf*) definition

Retention factor analysis is used to evaluate if the solvent system is adequate. Rf is defined as the distance traveled by the compound divided by the distance traveled by the solvent front. This means: the larger the Rf value of a compound, the larger is the distance traveled by the compound. In other words, when comparing Rf values of various compounds under identical chromatography conditions, the compound with the larger Rf is less polar because it interacts less strongly with the polar adsorbent on the plate.

Remember, a good solvent system is one that moves all components off the baseline with Rf values between 0.15 and 0.85 (*ideal Rf is 0.2 - 0.4*). Otherwise, when possible, it is preferable to chose another solvent system.

$$\textbf{Retention factor (}Rf\textbf{)} = \frac{\textbf{distance traveled by the compound}}{\textbf{distance traveled by the solvent front}}$$

Rf calculation based on the example shown here:
Rf = 4.0 cm / 5.5 cm = 0.73

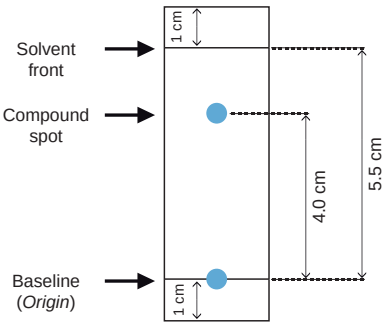
Prediction of Column Volumes *(CV)*

TLC data can be used to predict column elution based on the relationship between the retention factor and the column volume. CV is the number of column volumes required to elute the component from the column regardless of column dimensions [(*bed volume*) - (*volume of packing*)].

CV = 1 / Rf & **$\Delta CV = 1 / Rf_1 - 1 / Rf_2$**

The greater the ΔCV, the greater will be the separation and resolution between the spots (easier separation).

A bigger ΔCV will therefore allow more sample to be loaded onto the column.



TLC Visualization Methods

Described below are the most frequently used TLC visualization methods (*also called stains*) in alphabetical order.

Stains for Thin Layer Chromatography			
Name	**Visualization of...**	**Stain Recipe**	**Comments**
p-Anisaldehyde #1	**Universal stain** *Good for nucleophiles and oxygenated compounds* **Note***:* Tends to be insensitive to alkenes, alkynes and aromatic compounds unless other functional groups are present.	**Prepare stain as follows** • 2 mL of glacial acetic acid • 5 mL of p-anisaldehyde • 7 mL of conc. sulfuric acid • 185 mL of 95 % ethanol **Tip:** *add dropwise the acid at the end and stir vigorously.*	**Visualization Colors** • Spots: Various colors • BG: Orange to pink **Appropriate Storage** • Aluminum wrapped at 0°C
p-Anisaldehyde #2	*Acronycine* *Cineoles* *Terpenes*	**Prepare stain as follows** • 1 mL of p-anisaldehyde • 10 mL of perchloric acid • 20 mL of acetone • 80 mL of water	**Visualization Colors** • Spots: Various colors • BG: Orange to pink **Appropriate Storage** • Aluminum wrapped at 0°C
Bromocresol Green	*Acidic groups* ($pK_a < 5$) *Carboxylic acids*	**Prepare stain as follows** • 0.04 g of bromocresol green • 100 mL of 95 % ethanol • 0.1 M solution of sodium hydroxide **Tip***: add the base slowly at the end until the solution turns pale blue.*	**Visualization Colors** • Spots: Yellow to green • BG: Blue **Appropriate Storage** • Aluminum wrapped at 0°C ***Heating NOT required***
Cerium Molybdate (*CAM or Hanessian's Stain*)	**Universal stain** *Good for peptides* **Note***:* Highly sensitive stain; very low concentration of product may appear as a significant impurity.	**Prepare stain as follows** • 12 g of ammonium molybdate • 0.5 g of ceric ammonium molybdate • 15 mL of conc. sulfuric acid • 235 mL of water	**Visualization Colors** • Spots: Blue • BG: White **Appropriate Storage** • Aluminum wrapped
Cerium Sulfate ($Ce(SO_4)_2$)	**Difficultly stainable compounds**	**Prepare stain as follows** • 15 % aqueous sulfuric acid saturated with ceric sulfate	**Visualization Colors** • Spots: Black • BG: Yellow to white
Cobalt Chloride ($CoCl_2$)	**Universal stain** *Used in conjunction with PMA when this one is not effective enough*	**Prepare stain as follows** • 2 g of cobalt chloride • 100 mL of water • 10 mL of conc. sulfuric acid **Tip***: simply dip PMA treated plate in* $CoCl_2$ *solution.*	**Visualization Colors** • Spots: Various colors • BG: Pink ***Heating NOT required***
p-Dimethylamino-benzaldehyde (*PDAB or Ehrlich's Reagent*)	*Amines* *Indoles*	**Prepare stain as follows** • 0.5 g of p-dimethylamino-benzaldehyde • 10 mL of conc. hydrochloric acid • 40 mL of acetone (*or 95 % ethanol*)	**Visualization Colors** • Spots: Blue • BG: White
2,4-Dinitrophenyl-hydrazine (*DNP*)	*Aldehydes* *Ketones*	**Prepare stain as follows** • 12 g of 2,4-dinitrophenylhydrazine • 60 mL of conc. sulfuric acid • 80 mL of water • 200 mL of 95 % ethanol	**Visualization Colors** • Spots: Yellow to red • BG: Light orange ***DO NOT HEAT dipped plate***

Abbreviation: BG stands for "background".

Stains for Thin Layer Chromatography

Name	Visualization of...	Stain Recipe	Comments
Dragendorff Reagent	*Nitrogenous compounds (alkaloids, amines, organics bases, etc.)* *Phenols*	**Prepare stain as follows:** **Solution A** • 1.7 g of bismuth nitrate • 80 mL of water • 20 mL of acetic acid **Solution B** • 40 g of potassium iodide • 100 mL of water **Tip:** *mix 5 mL of each solution A and B to a solution of 20 mL of acetic acid in 70 mL of water.*	**Visualization Colors** • Spots: Orange to red • BG: Yellow **Appropriate Storage** • Aluminum wrapped **Stain Shelf-Life** • One or two weeks • Solutions A and B are long term storable ***DO NOT HEAT dipped plate***
Ferric Chloride (*$FeCl_3$*)	*Phenols*	**Prepare stain as follows** • 2 g of ferric chloride • 102 mL of 0.5 N hydrochloric acid	**Visualization Colors** • Spots: Red • BG: Yellow
Iodine	*Unsaturated and aromatic compounds*	**Prepare stain as follows** • Iodine crystals in an amber bottle	**Visualization Colors** • Spots: Dark brown • BG: Light brown **Note**: Iodine stain can be removed by heating.
Morin Hydrate (*Hydroxy Flavone*)	**Universal stain** *Fluorescently active compounds*	**Prepare stain as follows** • 0.1 % w/w of morin hydrate in methanol	**Visualization Colors** • Spots: Various colors • BG: White
Ninhydrin (*Indanetrione Hydrate*)	*Amino acids* *Amino sugars* *Amines*	**Prepare stain as follows** • 1.5 g of ninhydrin • 3 mL of acetic acid • 100 mL of n-butanol	**Visualization Colors** • Spots: Various colors • BG: White
Phosphomolybdic Acid (*PMA*)	**Universal stain** *Very effective against diluted sample*	**Prepare stain as follows** • 10 % of PMA solution in ethanol • or 10 g of PMA in 100 mL of ethanol	**Visualization Colors** • Spots: Dark green to black • BG: Light green
Potassium Permanganate* (*$KMnO_4$*)	*Olefins* *Readily oxidized groups* *Alcohols, aldehydes, alkenes, alkynes, etc.* *Can be used for detection of alcohols, amines, sulfides and mercaptans groups when gently heated.*	**Prepare stain as follows** • 1.5 g of potassium permanganate • 10 g of potassium carbonate • 1.25 mL of 10 % sodium hydroxide • 200 mL of water	**Visualization Colors** • Spots: Yellow to light brown • BG: Purple to pink **Stain Shelf-Life** • Three months
Vanillin	**Universal stain** *Very effective for same polarity products (Rf)*	**Prepare stain as follows** • 15 g of vanillin • 250 mL of 95 % ethanol • 2.5 mL of conc. sulfuric acid	**Visualization Colors** • Spots: Various colors • BG: Light tan

Abbreviation: BG stands for “background”.

Note: Occasionally, spots can be seen more clearly from glass side with glass backed TLC plate.
Otherwise mentioned, stains are long-term stable when stored in a tightly-closed container to prevent solvent evaporation

Silia*Plate* TLC Troubleshooting

Streaking or elongated spot rather than a defined spot?

Possible Solutions

- Sample was overloaded: run the TLC again using a more diluted solution of your sample.
- In presence of a base sensitive compound: try to add acetic or formic acid to the eluent (*0.1 - 2.0 %*).
- In presence of an acid sensitive compound: try to add triethylamine to the eluent (*0.1 - 2.0 %*) or 1 - 10 % ammonia in MeOH / DCM. If it is not working use Alumina as TLC coating.
- In presence of too highly polar compounds: try using a specialized silica TLC plate like reversed-phase (*C18 for example*).

Unable to see any spots on the TLC?

Possible Solutions

- If you have not been able to visualize any spots on your TLC using UV light, try another method; maybe your compound is not UV-active.
- Maybe your sample is too diluted. Try to apply several times your sample on the same spot (*do not forget to dry solvent between each application for optimal results*) or to concentrate your solution.
- Make sure the solvent level inside the tank is lower than the spotting line to avoid sample dissolution by the eluent.

How to monitor a reaction in presence of similar Rfs for both starting materials and product of interest?

Possible Solutions

- Try the co-spotting method.
- Try to visualize the plate using anisaldehyde or molybdene. Spot color or brightness differ for two compounds when using these stains.
- If none of the two previous solutions work, change solvent systems (*use another class of solvent*).

Tips: in chromatography, there are three classes of solvent systems providing significantly different results:

1. Mixture of polar / hydrocarbon solvents (*i.e.: EtOAc / Hexane; Ether / Petroleum ether*).
2. Mixture of polar / dichloromethane solvents (*examples of polar solvent: Ether, EtOAc, MeOH*).
3. Mixture of polar / benzene (*or toluene*) solvents (*examples of polar solvent: Ether, EtOAc, MeOH*).

Compounds stay too close to the baseline or solvent front.

Possible Solutions

- Too close to the baseline: your eluent is not polar enough; increase the proportion of polar solvent in the same solvent system or chose a more polar solvent.
- Too close to the solvent front: inversely, your eluent is too polar; decrease the proportion of polar solvent in the same solvent system or chose a less polar solvent.

Resource Center

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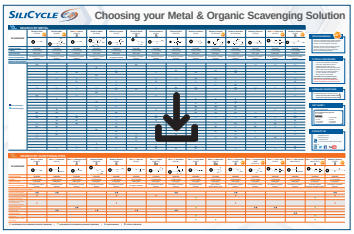
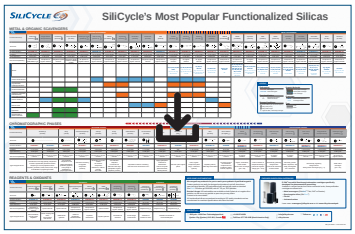
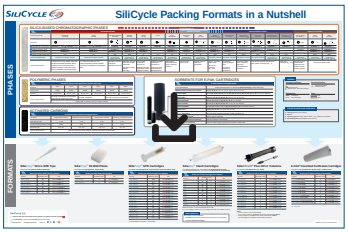


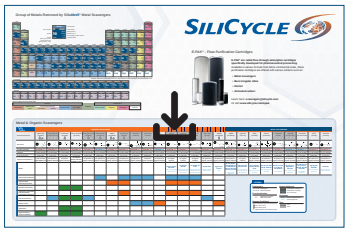
Choosing your metal & organic scavenging solution



SiliCycle's most popular functionalized silicas



SiliCycle packing formats in a nutshell



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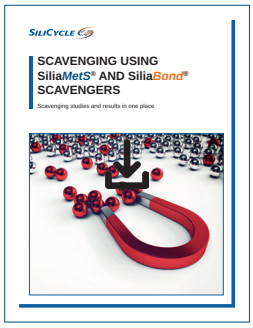
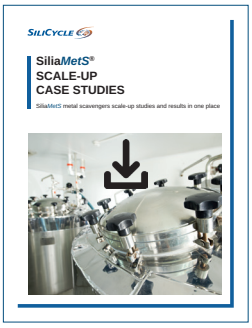


A collection of various case studies and application notes using scavengers



A collection of scale-up case studies and application notes using scavengers

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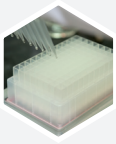


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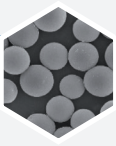


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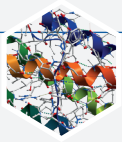

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Email: **info@silicycle.com**

Phone: **+1 418.874.0054**

Toll Free: **+1 877.745.4292** (*North America only*)

Canadian Headquarters

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

Overseas Offices

SiliCycle Europe / europe@silicycle.com

SiliCycle Shanghai / contact@silicyclechina.com