SOLUTIONS FOR PURIFICATION & CHROMATOGRAPHY

SILICYCLE

SILICYCLE

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

Solutions for Purification & Chromatography

SiliCycle is your partner of choice for your purification and chromatography 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
- Silia*Sphere*™ 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
Exceptional 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 & 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, No Scale-up Limitations!

Scaling-up from laboratory to production scale

Two Shapes Available: Irregular & 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 & 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 & 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 or 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 now 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.

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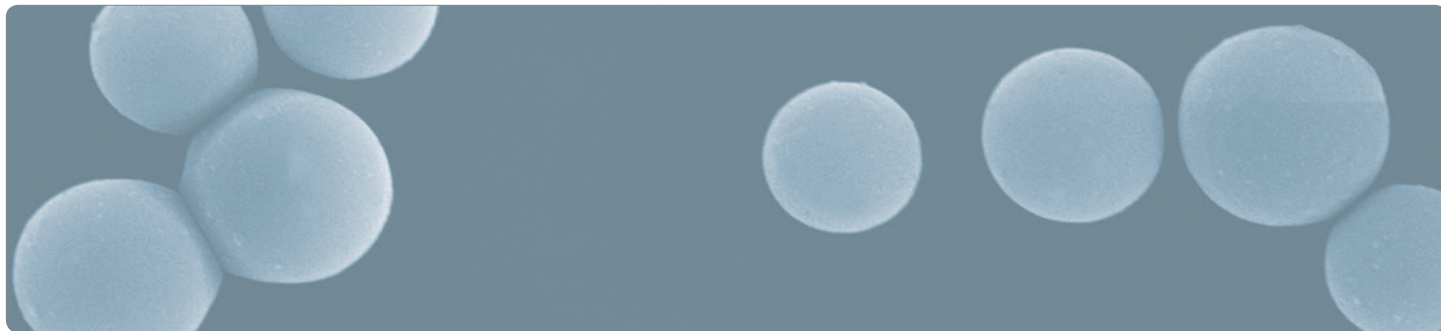
Silia*Flash* & Silia*Sphere* PC Characteristics

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
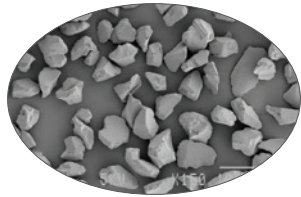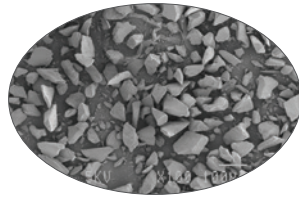
SiliCycle

Competitor

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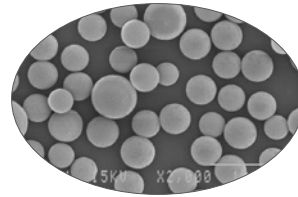
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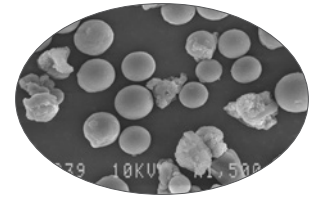
Competitor

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.

Wide Particle Size Distribution   **Narrow Particle Size Distribution**

Preferential path chosen

Path straight through the column

Competitive Gel

Silia*Flash*
Silia*Sphere* PC
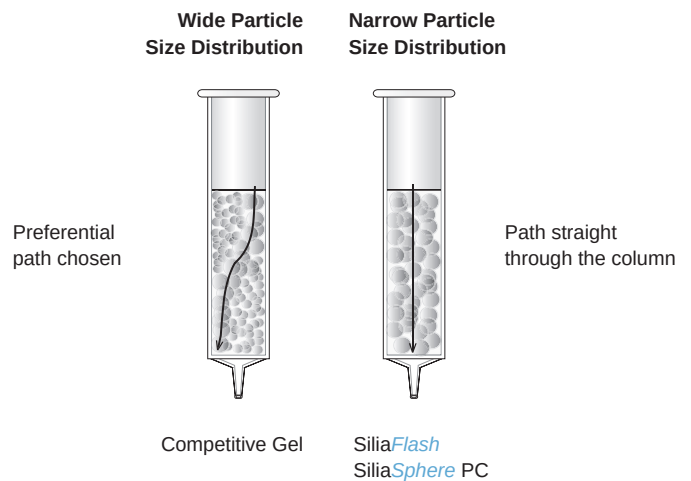
High Purity Silica Gels

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 (*CofA*). 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
Iron	Fe	32	75	41
Magnesium	Mg	61	149	104
Sodium	Na	466	945	585
Titanium	Ti	147	250	179
Zirconium	Zr	32	75	56

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Silia*Flash* Irregular Silica Gels

One of the Tightest Particle Size Distribution on the Market

Of 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.

Of the 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.
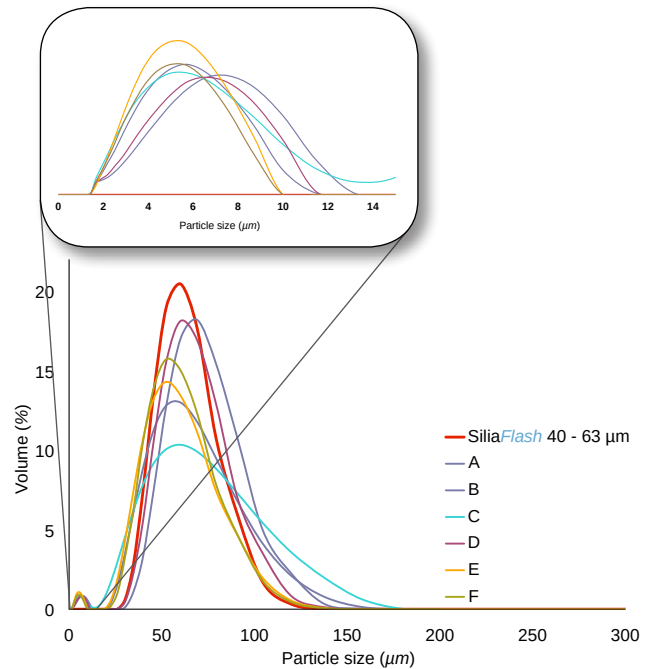
Particle Size Analysis Methods

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.

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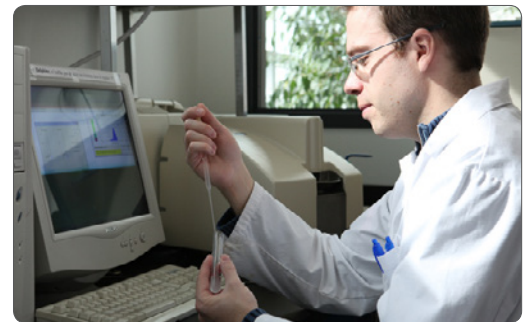
Two Different Grades for Different Needs

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 Gels		
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

Silia*Flash* F60 R10030B 90 % size up to grade
Silia*Flash* P60 R12030B 75 % size up to grade
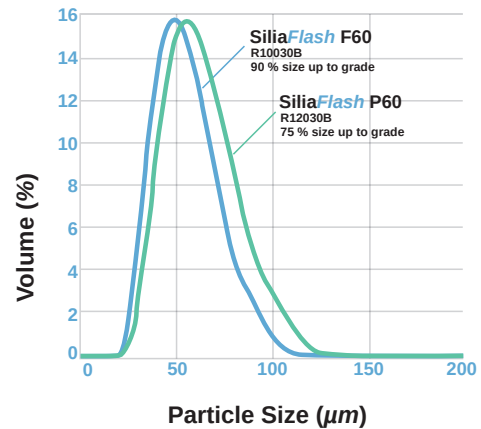
The figure on the right shows F60's tighter particle size distribution and the absence of fines for both gels.

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 Gels		
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

Silia*Flash* G60 R10040B 90 % size up to grade
Silia*Flash* GE60 R10140B 80 % size up to grade

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

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

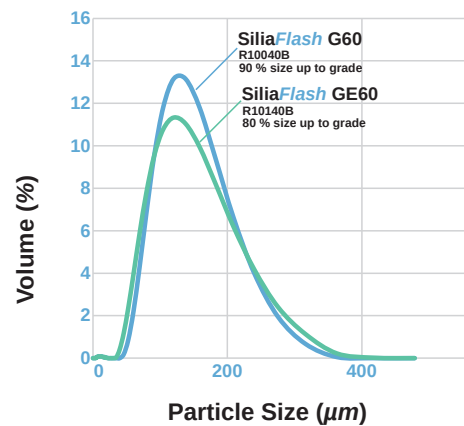| Typical Metal Content of Most Popular Irregular Silicas						
Product		**F60**	**P60**	**Acid Washed***	**G60**	**GE60**
Product Number		**R10030B**	**R12030B**	**R10530B**	**R10040B**	**R10140B**
Particle Size		40 - 63 µm			60 - 200 µm	
Pore Diameter		60 Å			60 Å	
Metal (*mg/kg*)						
Aluminum	Al	< 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	

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

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

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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
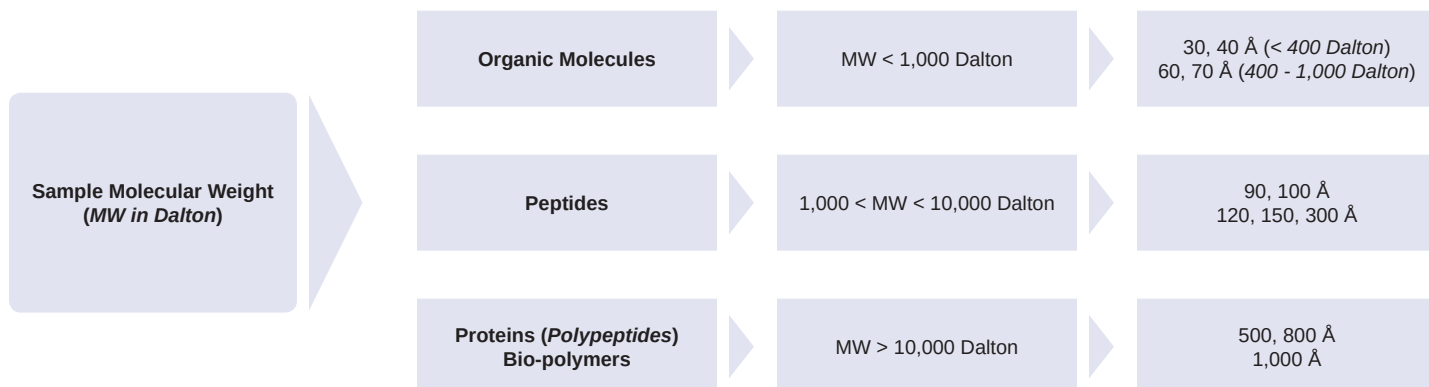
Selecting Particle Size

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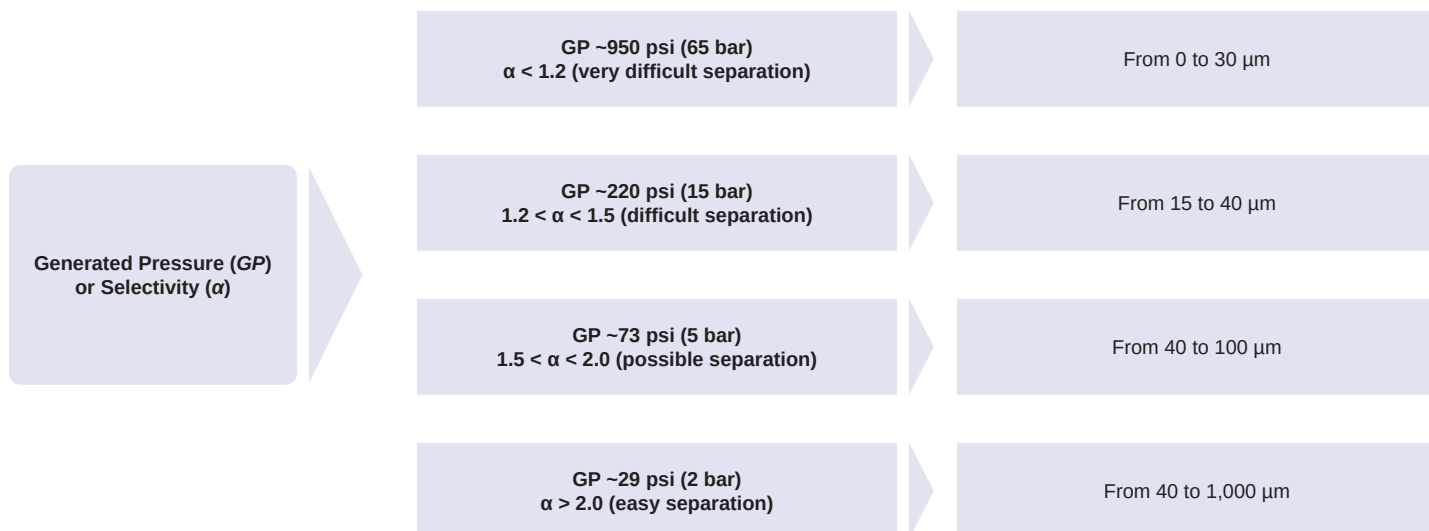
Selectivity ($α$) is measured by the retention factor ratio between two similar compounds: $\alpha = \dfrac{Tr_1 - T_0}{Tr_2 - T_0}$

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A Particle Size for Each Application

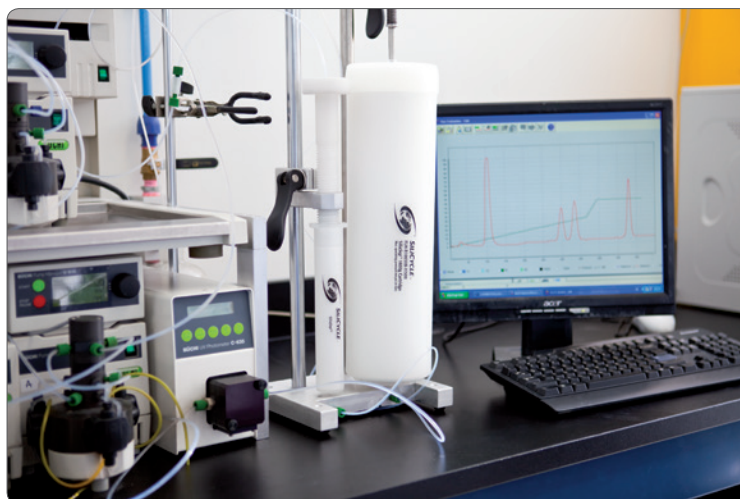| Most Popular Particle Size Applications		
Particle Size Distribution		**Applications**
Irregular Particles	**Spherical Particles**	
Particles for Preparative TLC Plates		
From 0 to 20 µm	-	• Contains neither binder (*organic or inorganic*) nor UV indicator (F_{254}) • Can also be used in flash chromatography 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

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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: info@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**		**Pore Diameter (Å)**
	µm	**mesh**	
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*)			
R10150B	60 - 120	325 - 625	
R10040B (*G60*)	60 - 200	70 - 230	
R10140B (*GE60*)			
R10137B	75 - 150	100 - 200	
R10157B	105 - 175	86 - 140	
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	
R10157D	105 - 175	86 - 140	
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	
R10157H	105 - 175	86 - 140	
R10160H	120 - 200	70 - 125	
R10170H	200 - 500	35 - 70	
R10072H	250 - 500	35 - 60	
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**		**Pore Diameter (Å)**
	µm	**mesh**	
S10095W-A	25	*	50
S10030B-A	50	300	60
S10027B-A	60	250	
S10034B-A	75	200	
S10040B-A	100	150	
S10063B-A	150	100	
S10066B-A	200	70	
S10068B-A	300	50	
S10020C	20 - 45	*	70
S10040C	75 - 200	70 - 200	
S10030C	40 - 75	200 - 400	
S10070C	200 - 500	35 - 70	
S10095D-A	25	*	90
S10020E	20 - 45	*	100
S10030E	40 - 75	200 - 400	
S10040E	75 - 200	70 - 200	
S10065E	150 - 250	60 - 100	
S10070E	200 - 500	35 - 70	
S10030G-A	50	300	120
S10034G-A	70	200	
S10040G-A	100	150	
S10063G-A	150	100	
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.

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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. But we can graft any irregular Silia*Flash* or spherical Silia*Sphere* PC silica gel, with the function of your choice. Contact us for more information.

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, Cyclohexyl*).

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 4 % and 19 %.
• **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.
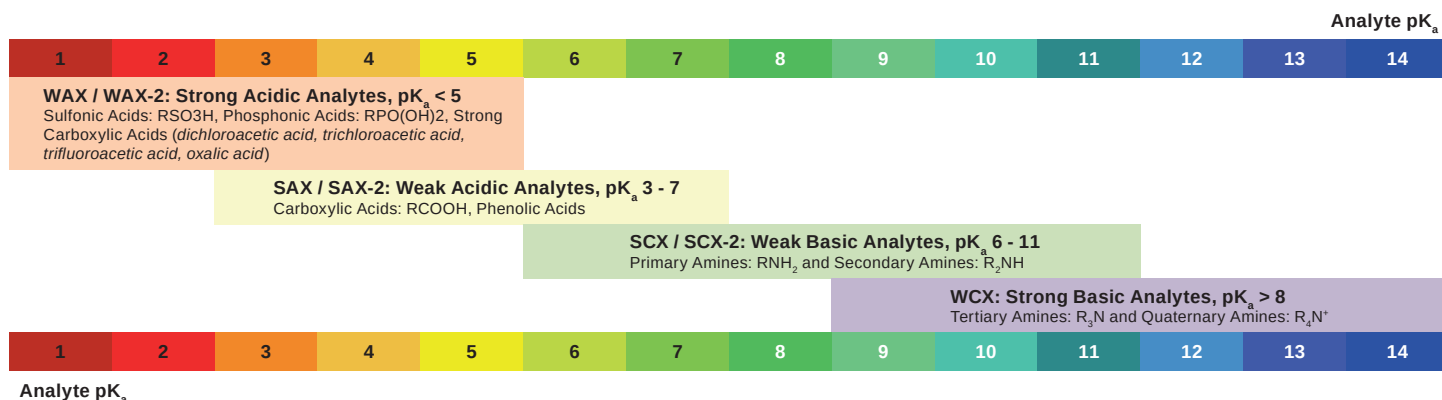
Normal Phases

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 (*amino, cyano and diol*) or alternative adsorbents (*Alumina and Florisil for example*).
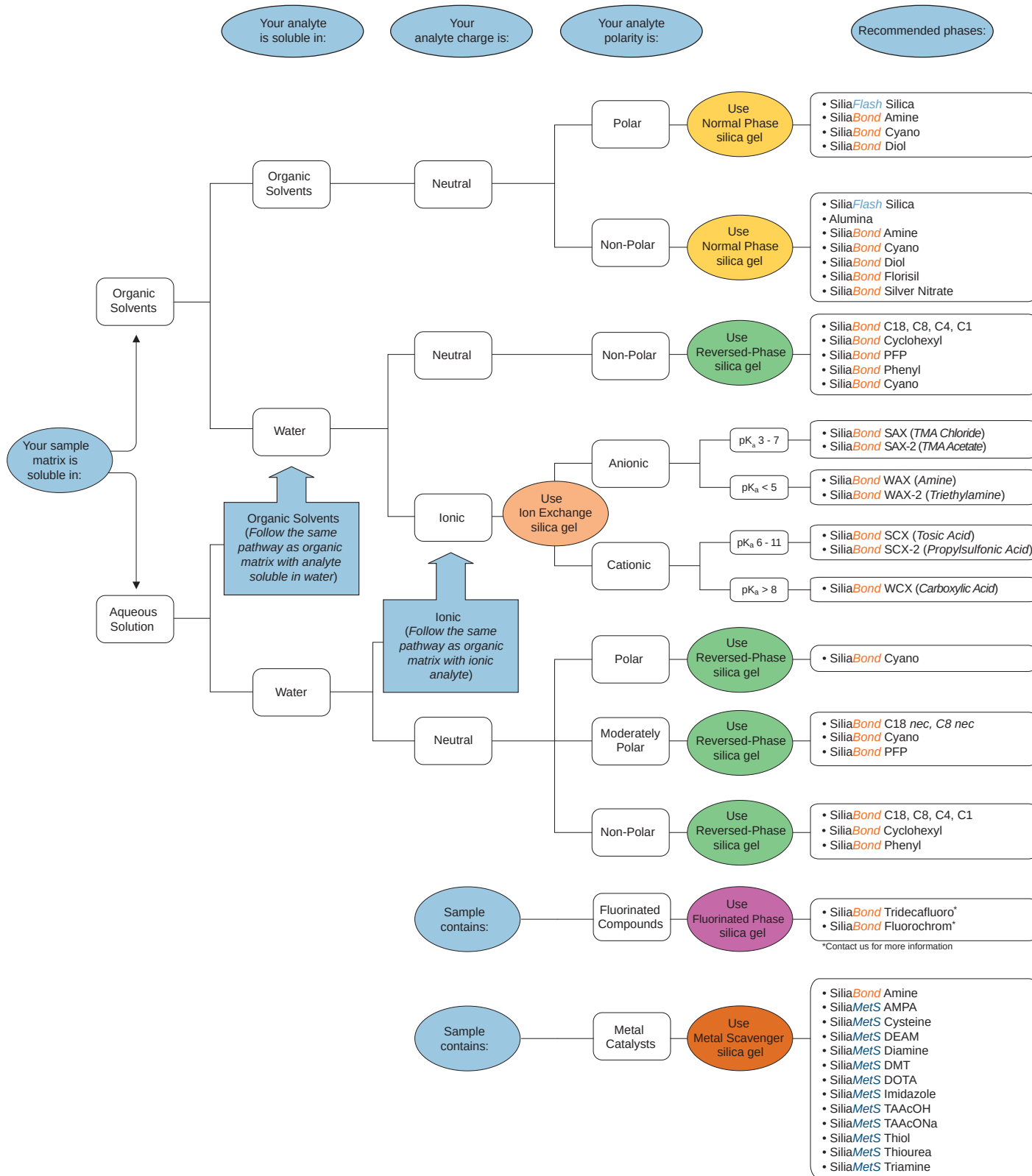
Ion Exchange Phases

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 & SAX*). And in the contrary if the stationary phase is negatively charged, cationic analytes only will retain (*these phases are called WCX & SCX*). Hence, pH of the mobile phase is of crucial importance and needs to be chosen carefully, so that both functions are charged:

Analyte pK_a

1	2	3	4	5	6	7	8	9	10	11	12	13	14

WAX / WAX-2: Strong Acidic Analytes, pK_a < 5
Sulfonic Acids: RSO3H, Phosphonic Acids: RPO(OH)2, Strong Carboxylic Acids (*dichloroacetic acid, trichloroacetic acid, trifluoroacetic acid, oxalic acid*)

SAX / SAX-2: Weak Acidic Analytes, pK_a 3 - 7
Carboxylic Acids: RCOOH, Phenolic Acids

SCX / SCX-2: Weak Basic Analytes, pK_a 6 - 11
Primary Amines: RNH_2 and Secondary Amines: R_2NH

WCX: Strong Basic Analytes, pK_a > 8
Tertiary Amines: R_3N and Quaternary Amines: R_4N^+

1	2	3	4	5	6	7	8	9	10	11	12	13	14

Analyte pK_a

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.

Your analyte is soluble in:

Your analyte charge is:

Your analyte polarity is:

Recommended phases:

Your sample matrix is soluble in:

Organic Solvents → **Organic Solvents** → **Neutral**

- Polar → Use Normal Phase silica gel
  - Silia*Flash* Silica
  - Silia*Bond* Amine
  - Silia*Bond* Cyano
  - Silia*Bond* Diol

- Non-Polar → Use Normal Phase silica gel
  - Silia*Flash* Silica
  - Alumina
  - Silia*Bond* Amine
  - Silia*Bond* Cyano
  - Silia*Bond* Diol
  - Silia*Bond* Florisil
  - Silia*Bond* Silver Nitrate

Water → **Neutral** → **Non-Polar** → Use Reversed-Phase silica gel
  - Silia*Bond* C18, C8, C4, C1
  - Silia*Bond* Cyclohexyl
  - Silia*Bond* PFP
  - Silia*Bond* Phenyl
  - Silia*Bond* Cyano

Water → **Ionic** → Use Ion Exchange silica gel

- Anionic
  - pK_a 3 - 7
    - Silia*Bond* SAX (*TMA Chloride*)
    - Silia*Bond* SAX-2 (*TMA Acetate*)
  - pK_a < 5
    - Silia*Bond* WAX (*Amine*)
    - Silia*Bond* WAX-2 (*Triethylamine*)

- Cationic
  - pK_a 6 - 11
    - Silia*Bond* SCX (*Tosic Acid*)
    - Silia*Bond* SCX-2 (*Propylsulfonic Acid*)
  - pK_a > 8
    - Silia*Bond* WCX (*Carboxylic Acid*)

Organic Solvents (*Follow the same pathway as organic matrix with analyte soluble in water*)

Ionic (*Follow the same pathway as organic matrix with ionic analyte*)

Aqueous Solution → **Water**

- Polar → Use Reversed-Phase silica gel
  - Silia*Bond* Cyano

- Moderately Polar → Use Reversed-Phase silica gel
  - Silia*Bond* C18 *nec, C8 nec*
  - Silia*Bond* Cyano
  - Silia*Bond* PFP
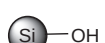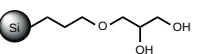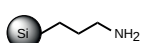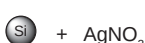
- Non-Polar → Use Reversed-Phase silica gel
  - Silia*Bond* C18, C8, C4, C1
  - Silia*Bond* Cyclohexyl
  - Silia*Bond* Phenyl

Sample contains: Fluorinated Compounds → Use Fluorinated Phase silica gel
  - Silia*Bond* Tridecafluoro[*]
  - Silia*Bond* Fluorochrom[*]

  *Contact us for more information

Sample contains: Metal Catalysts → Use Metal Scavenger silica gel
  - Silia*Bond* Amine
  - Silia*MetS* AMPA
  - Silia*MetS* Cysteine
  - Silia*MetS* DEAM
  - Silia*MetS* Diamine
  - Silia*MetS* DMT
  - Silia*MetS* DOTA
  - Silia*MetS* Imidazole
  - Silia*MetS* TAAcOH
  - Silia*MetS* TAAcONa
  - Silia*MetS* Thiol
  - Silia*MetS* Thiourea
  - Silia*MetS* Triamine

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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**
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
Cyclohexyl (*C6*) PN: R61530B		% C: ≥ 9.5 % Density: 0.662 g/mL	• Less retention compared to C18 and C8 • Additional steric interaction
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	$\text{Si}-C_1$	% 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
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	Si—OH	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	Si—OH	Pore size: 90 Å Particle size: 25 µm 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
Neutral Alumina PN: AUT-0054	Al_2O_3	Particle size: 75 - 150 µm Pore size: 70 Å	• For aromatic compounds, aliphatic amines & compounds containing electronegative functions
Florisil PN: AUT-0014	$SiMgO_3$	Particle size: ≤ 75 µm Pore size: 80 Å	• For separation of chlorinated pesticides, polychlorinated biphenyls (*PCBs*) & polysaccharides
Silver Nitrate (*AgNO$_3$*) PN: R23530B	Si + AgNO$_3$	Loading: 10 % w/w Density: 0.604 g/mL	• For separation of cis / trans isomers of unsaturated compounds (*alkenes, lipids, steroids and terpenes*)

Reversed-phases (rows C18 through Cyano); Normal Phases (rows Silica through Silver Nitrate).

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 can 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**	**NH₂**	**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	✓										
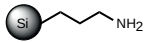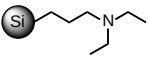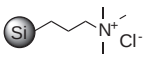
$AgNO_3$ is particularly useful to separate isomers that present unsaturated groups.
Neutral Alumina is used for the separation of aldehydes, ketones, quinines, esters, lactones and glucosides.
Florisil will help analyze pesticides, PCBs and PAHs.

SILICYCLE
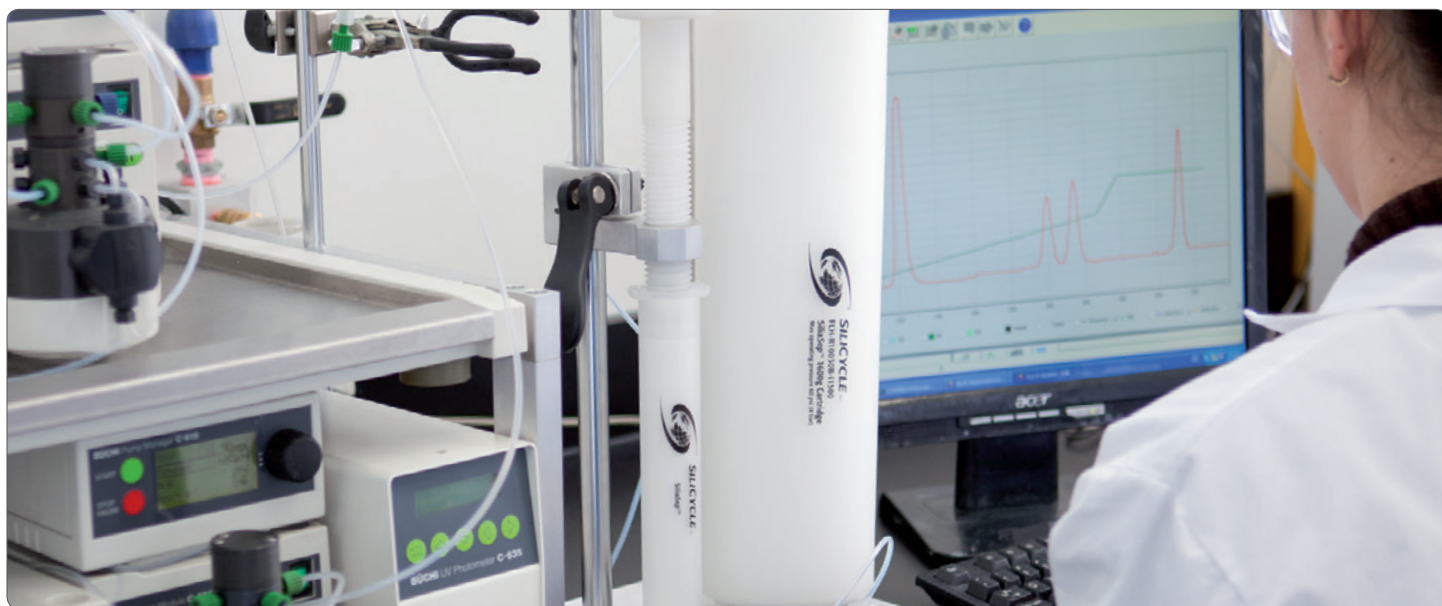
Ion Exchange Phases Portfolio

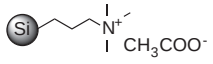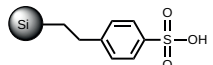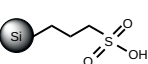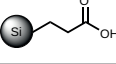|	Sorbent	Structure	Typical Characteristics	Typical Applications
Low Pressure Chromatography Ion Exchange Phases Characteristics				
Ion Exchange Phases	**Amine** (*NH₂, WAX*) PN: R52030B	Si—NH₂	Loading: ≥ 1.2 mmol/g Density: 0.700 g/mL	• Weak anion exchanger (*pK$_a$ of 9.8*), positively charged at 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	Si—N	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 (*TMA Chloride*) PN: R66530B	Si—N⁺ Cl⁻	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 (*TMA Acetate*) PN: R66430B	Si—N⁺ CH₃COO⁻	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	Si—(ring)—S(=O)₂—OH	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	Si—S(=O)₂—OH	Loading: ≥ 0.63 meq/g Density: 0.728 g/mL	
	WCX (*Carboxylic Acid*) PN: R70030B	Si—C(=O)—OH	Loading: ≥ 0.92 mmol/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 phases are available endcapped and non-endcapped.
Other phases can 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.

Lower selectivity
Easier to displace

Higher selectivity
Harder to displace

Solutions for Purification & Chromatography
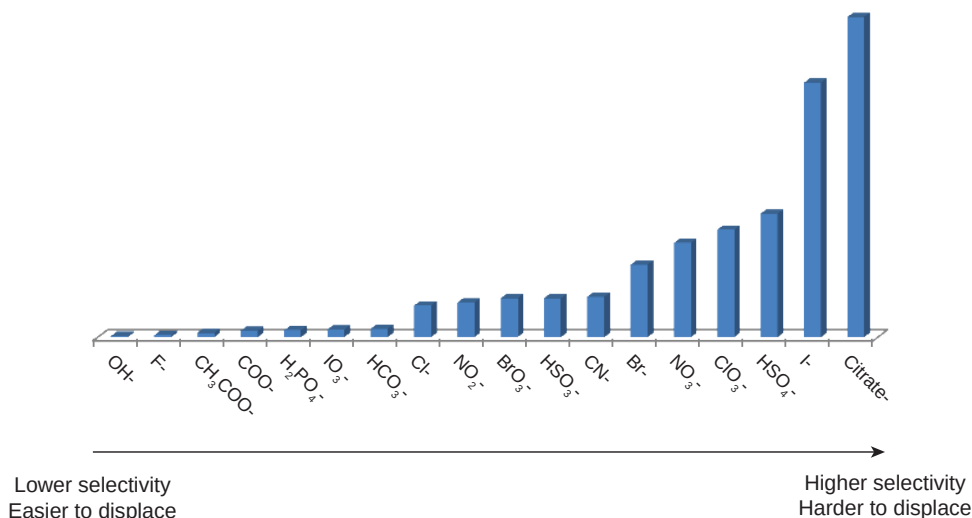
SiliaBond Bulk Ordering Information

To build your own product number, just add the Format to the Phase PN: **[Phase PN]**-**[Format Code]**
Example: 100 g of C18 silica gel, 40 - 63 µm, 60 Å: R33230B-100G.

SiliaBond phases are available on all irregular SiliaFlash silicas (R100-) and on all spherical SiliaSphere 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.

	SiliaBond Phases				
Phase	**Irregular Silica (R)**				**Spherical Silica (S)**
	60 Å, 40 - 63 µm **(30B)**	**60 Å, 60 - 200 µm** **(40B)**	**60 Å, 200 - 500 µm** **(70B)**	**300 Å, 40 - 63 µm** **(30M)**	**100 Å, 40 - 75 µm** **(30E)**
SiliaBond Silica (R100)	R10030B	R10040B	R10070B	R10030M	S10030E
SiliaBond Amine (R520)	R52030B	R52040B	R52070B	R52030M	S52030E
SiliaBond Diol nec (R350)	R35030B	R35040B	R35070B	R35030M	S35030E
SiliaBond Cyano (R380)	R38030B	R38040B	R38070B	R38030M	S38030E
SiliaBond C18 (R332)	R33230B	R33240B	R33270B	R33230M	S33230E
SiliaBond C8 (R308)	R30830B	R30840B	R30870B	R30830M	S30830E
SiliaBond Phenyl (R340)	R34030B	R34040B	R34070B	R34030M	S34030E
SiliaBond PFP (R675)	R67530B	R67540B	R67570B	R67530M	S67530E
SiliaBond SCX (R605)	R60530B	R60540B	R60570B	R60530M	S60530E
SiliaBond SCX-2 (R512)	R51230B	R51240B	R51270B	R51230M	S51230E
SiliaBond SAX nec (R665)	R66530B	R66540B	R66570B	R66530M	S66530E
SiliaBond SAX-2 nec (R664)	R66430B	R66440B	R66470B	R66430M	S66430E

SiliaBond 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**

SILICYCLE
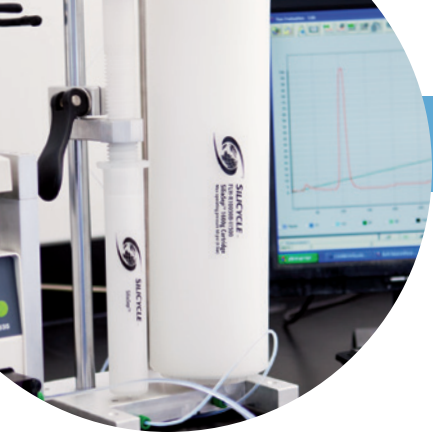
With a more tightly and homogeneous packed silica bed compared to conventional manual flash chromatography, the use of pre-packed flash cartridges improves purification efficiency by offering superior reproducibility and productivity.

With Silia*Sep*, benefit from the same quality that all our products are known for: selectivity, speed & reliability.

Silia*Sep*™ Flash Cartridges

Cartridge Design

Available in two silica gel grades:
- 25 µm (PREMIUM)
- 40 - 63 µm, with various chemistries

Effective packing technology for superior performance and separation

Standard male slip tip for compatibility with any flash system

Standard female luer-lock for compatibility with any flash system

SILICYCLE™
FLH-R10030B-IS120
SiliaSep™ 120g Cartridge
Max operating pressure 205 psi (13 bar)

Wide choice of cartridge sizes from grams to kilograms (*purification scale-up from mg to hundreds of grams*)

End fitting caps to ensure constant moisture percentage

Unique one-piece polypropylene cartridge for high pressure and solvent resistance

Secure screw cap with unique design to prevent leaks and ensure consistent sample loads

Features & 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 & safety
- Leak-free guaranteed by unique one-piece cartridge design
- Reproducible performance from lot-to-lot (*stringent quality control*)
- Excellent durability to withstand high pressures
- Universal luer fittings for compatibility with any flash system

Versatility
- Wide choice of cartridge sizes from 4 g to 41 kg
- Purification scale-up from milligrams to kilograms
- Variety of sorbents to meet any separation need

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

Cost effectiveness
- Excellent performance vs price ratio
- Readily available from stock inventory for many volumes

Portfolio

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

SiliaSep Flash Cartridges Adsorbents	
Adsorbent type	**Adsorbent**
Bare silica	• Standard Silia*Flash* 40 - 63 µm Irregular Silica • PREMIUM 25 µm Spherical Silica **NEW** (*learn more page 24*)
Bonded phases	• Silia*Bond* Chromatographic Phases (*C18, C8, Phenyl, PFP, etc.*) • Silia*MetS* Metal Scavengers (*Thiol, DMT, etc.*) • Silia*Bond* Organic Scavengers (*Amine, Tosic Acid, etc.*)

Formats

	Cartridge [Code]	Silica weight	Quantity	Dimensions (*Diam. x Length**)	Column volume	Recommended flow rate	Loading capacity	Max operating pressure
Discovery & R&D	**Silia*Sep* 4 g** [ISO04]	Bare: 4 g	20 / box	12 x 98 mm	4.9 mL	15 - 25 mL/min	Bare: 0.04 - 0.4 g Bonded: 0.02 - 0.2 g	225 psi / 16 bar
		Bonded: ≥ 5 g	2 / box					
	Silia*Sep* 12 g [ISO12]	Bare: 12 g	20 / box	21 x 117 mm	17 mL	20 - 40 mL/min	Bare: 0.12 - 1.2 g Bonded: 0.06 - 0.6 g	
		Bonded: ≥ 15 g	1 / box					
	Silia*Sep* 25 g [ISO25]	Bare: 25 g	15 / box	21 x 165 mm	31 mL	20 - 45 mL/min	Bare: 0.25 - 2.5 g Bonded: 0.125 - 1.25 g	
		Bonded: ≥ 30 g	1 / box					
	Silia*Sep* 40 g [ISO40]	Bare: 40 g	15 / box	27 x 169 mm	47 mL	25 - 50 mL/min	Bare: 0.4 - 4 g Bonded: 0.2 - 2 g	
		Bonded: ≥ 45 g	1 / box					
	Silia*Sep* 80 g [ISO80]	Bare: 80 g	12 / box	31 x 237 mm	123 mL	40 - 80 mL/min	Bare: 0.8 - 8 g Bonded: 0.4 - 4 g	
		Bonded: ≥ 90 g	1 / box					
	Silia*Sep* 120 g [IS120]	Bare: 120 g	10 / box	36 x 256 mm	190 mL	60 - 120 mL/min	Bare: 1.2 - 12 g Bonded: 0.6 - 6 g	205 psi / 13 bar
		Bonded: ≥ 130 g	1 / box					
	Silia*Sep* 220 g [IS220]	Bare: 220 g	4 / box	60 x 195 mm	306 mL	60 - 190 mL/min	Bare: 2.2 - 22 g Bonded: 1.1 - 11 g	160 psi / 11 bar
		Bonded: ≥ 230 g	1 / box					
	Silia*Sep* 330 g [IS330]	Bare: 330 g	4 / box	60 x 268 mm	441 mL	80 - 190 mL/min	Bare: 3.3 - 33 g Bonded: 1.65 - 16.5 g	
		Bonded: ≥ 360 g	1 / box					
Development & Process	**Silia*Sep* BT 75S** [75iS]	Bare: 200 g	2 / box	75 x 90 mm	300 mL	100 - 250 mL/min	Bare: 0.2 - 20 g Bonded: 0.1 - 10 g	90 psi / 6.5 bar (inside the compression module)
		Bonded: 200 g	1 / box					
	Silia*Sep* BT 75M [75iM]	Bare: 400 g	2 / box	75 x 170 mm	500 mL		Bare: 0.4 - 40 g Bonded: 0.2 - 20 g	
		Bonded: 400 g	1 / box					
	Silia*Sep* BT 75L [75iL]	Bare: 800 g	2 / box	75 x 350 mm	1 L		Bare: 0.8 - 80 g Bonded: 0.4 - 40 g	
		Bonded: 800 g	1 / box					
	Silia*Sep* XL 800 g** [IS750]	Bare: 800 g	2 / box	78 x 382 mm	1.5 L	200 - 300 mL/min	Bare: 8 - 80 g Bonded: 4 - 40 g	125 psi / 8 bar
		Bonded: ≥ 870 g	1 / box					
	Silia*Sep* XL 1,600 g** [I1500]	Bare: 1,600 g	2 / box	104 x 429 mm	2.9 L	300 - 450 mL/min	Bare: 16 - 160 g Bonded: 8 - 80 g	100 psi / 7 bar
		Bonded: ≥ 1,700 g	1 / box					
	Silia*Sep* XL 3,000*** [I3000]	Contact us for more information: info@silicycle.com						

* Cartridge length includes luer-lock and connection tip.

** For Silia*Sep* XL formats, you will need to use an XL Adapter to plug the cartridge on your system. Part number AUT-0127-1.

*** Compatible with the Torrent® system.

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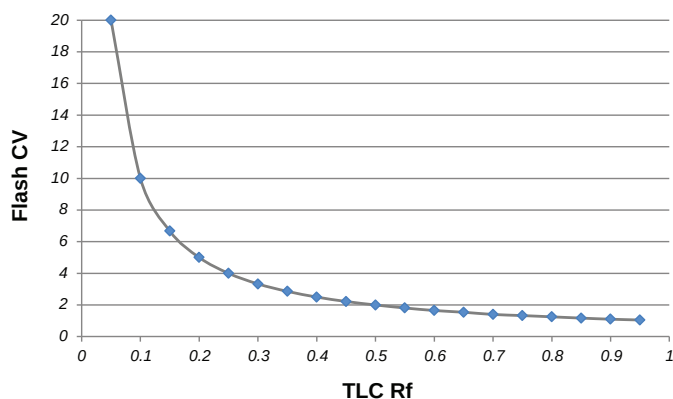
Rf vs CV	
TLC Rf	**Flash CV**
0.95	1.05
0.90	1.10
0.85	1.17
0.80	1.25
0.75	1.33
0.70	1.40
0.65	1.54
0.60	1.65
0.55	1.81
0.50	2.00
0.45	2.22
0.40	2.50
0.35	2.86
0.30	3.33
0.25	4.00
0.20	5.00
0.15	6.67
0.10	10.00
0.05	20.00

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

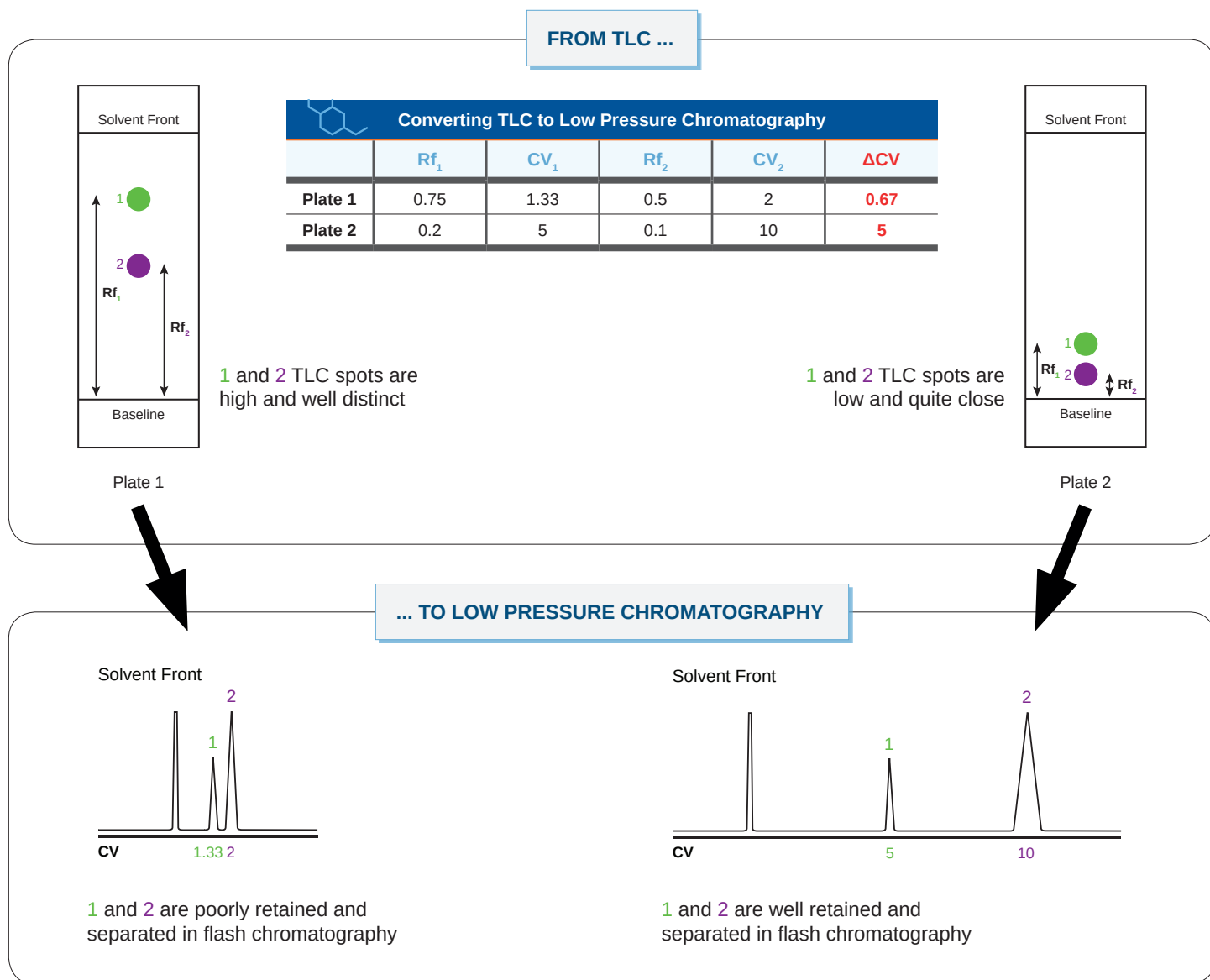| ΔCV values according to Rf_1 and Rf_2 values																		
0.05	0.00																	
0.10	10.00	0.00																
0.15	13.33	3.33	0.00															
0.20	15.00	5.00	1.67	0.00														
0.25	16.00	6.00	2.67	1.00	0.00													
0.30	16.67	6.67	3.34	1.67	0.67	0.00												
0.35	17.14	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
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**

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. Adjust the TLC solvent mixture (*solvent polarity and composition of the mixture*) to obtain the preferred Rfs.

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

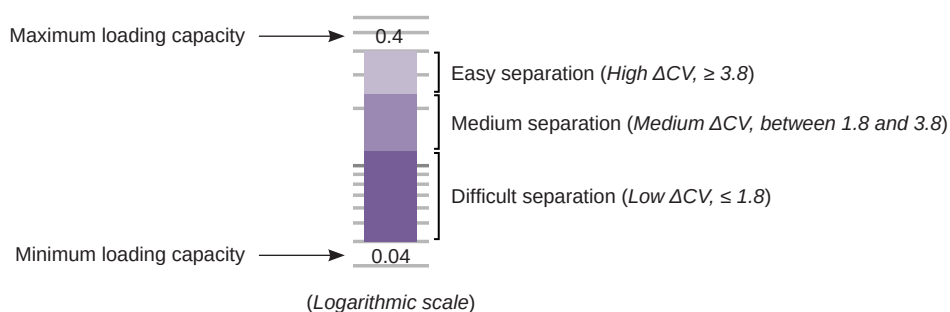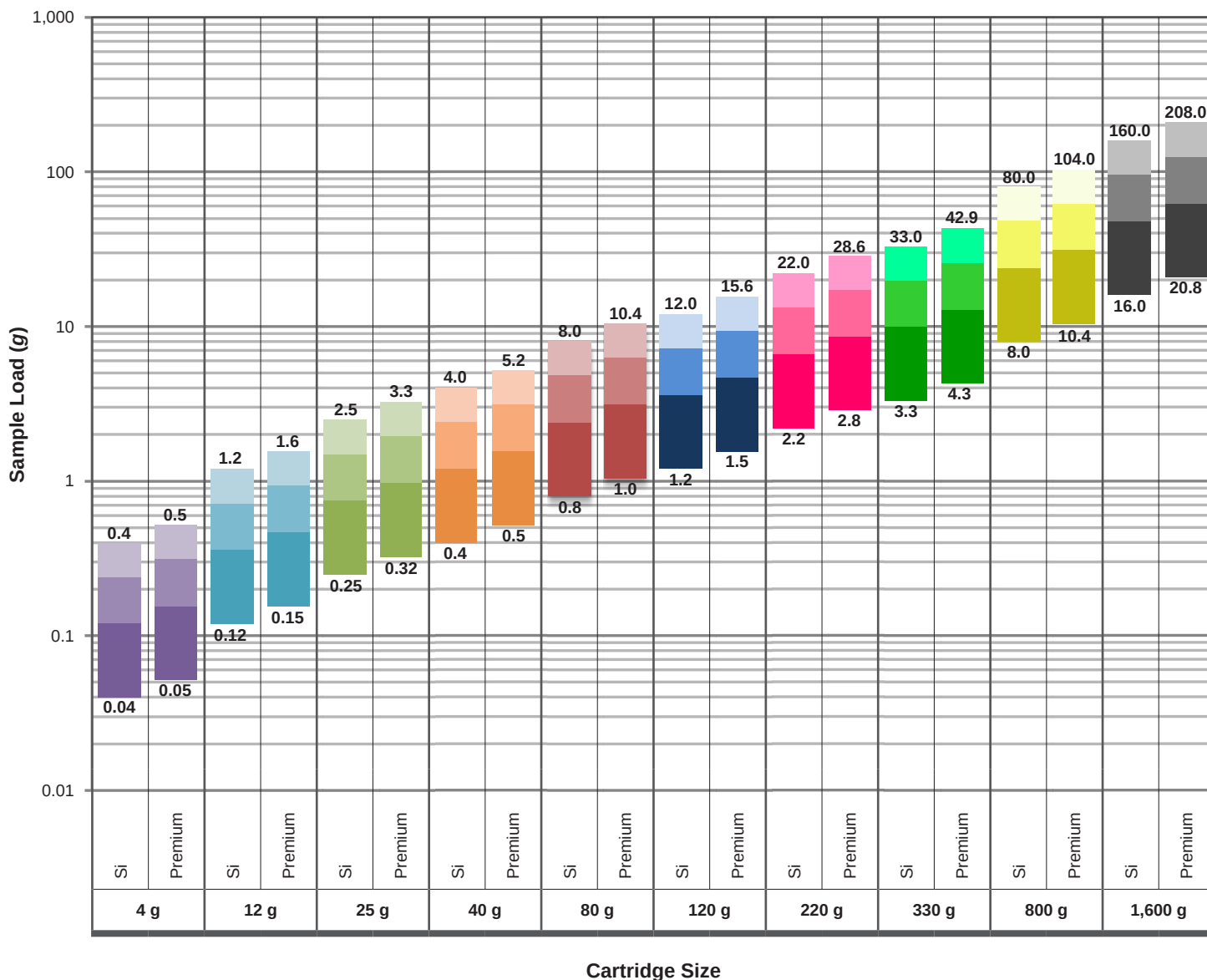We need to separate two analytes, 1 and 2. We will study two different TLC configurations.

FROM TLC ...

	Rf$_1$	CV$_1$	Rf$_2$	CV$_2$	ΔCV
Converting TLC to Low Pressure Chromatography					
Plate 1	0.75	1.33	0.5	2	0.67
Plate 2	0.2	5	0.1	10	5

Plate 1:
Solvent Front
1
2
Rf$_1$
Rf$_2$
Baseline

1 and 2 TLC spots are high and well distinct

Plate 2:
Solvent Front
1
2
Rf$_1$ Rf$_2$
Baseline

1 and 2 TLC spots are low and quite close

... TO LOW PRESSURE CHROMATOGRAPHY

Solvent Front
2
1
CV   1.33 2

1 and 2 are poorly retained and separated in flash chromatography

Solvent Front
2
1
CV   5   10

1 and 2 are well retained and separated in flash chromatography

In summary:

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

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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.

Sample Load (*g*) vs Cartridge Size (Logarithmic scale)

Cartridge Size	Si	Premium
4 g	0.04 – 0.4	0.05 – 0.5
12 g	0.12 – 1.2	0.15 – 1.6
25 g	0.25 – 2.5	0.32 – 3.3
40 g	0.4 – 4.0	0.5 – 5.2
80 g	0.8 – 8.0	1.0 – 10.4
120 g	1.2 – 12.0	1.5 – 15.6
220 g	2.2 – 22.0	2.8 – 28.6
330 g	3.3 – 33.0	4.3 – 42.9
800 g	8.0 – 80.0	10.4 – 104.0
1,600 g	16.0 – 160.0	20.8 – 208.0

Legend:

Maximum loading capacity → 0.4

- Easy separation (*High ΔCV, ≥ 3.8*)
- Medium separation (*Medium ΔCV, between 1.8 and 3.8*)
- Difficult separation (*Low ΔCV, ≤ 1.8*)

Minimum loading capacity → 0.04

(*Logarithmic scale*)

SILICYCLE Solutions for Purification & Chromatography

Loading capacity depends on the sample itself, the column dimensions and the column chemistry. 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.

Dimensions ID x Length	Silia*Sep* Format	Silia*Sep* Phase	Load (g) Difficult Separation ΔCV = 0.1 - 0.6	ΔCV = 0.7 - 1.2	ΔCV = 1.3 - 1.8	Medium Separation ΔCV = 1.9 - 2.4	ΔCV = 2.5 - 3.1	ΔCV = 3.2 - 3.8	Easy Separation ΔCV = 3.9 - 4.5	ΔCV = 4.6 - 5.2	ΔCV = 5.3 - 6.0	ΔCV > 6
12 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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 x 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
		Spherical Silica	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.

Note: There is no linearity between TLC and flash for bonded phases (*not the exact same silica*).
The loading capacities for bonded phases written above are just informative, they won't necessarily match the ΔCVs measured in TLC.

SILICYCLE

Silia*Sep* PREMIUM - Spherical Silica Flash Cartridges

NEW

Packed with our 25 µm spherical silica gels, Silia*Sep* offers a greater resolution and better separation.

Silia*Sep* PREMIUM Scalability

Silia*Sep* PREMIUM perfect linearity between all formats, from 4 g cartridges to XL 1,600 g cartridges, will allow you to easily scale-up your methods and transfer your preliminary tests on bigger cartridges size.

You will find below the purification of a solution of Aniline, Anisole and Benzoic Acid in DMSO on Silia*Sep* PREMIUM C18 25 g and 330 g. Chromatograms are almost identical!

Chromatographic Conditions

Mobile phase: gradient Water / Methanol (*90:10 to 0:100*)

Flow rate: 40 mL/min

Injection volume: 0.5 mL

Wavelength: 254 nm
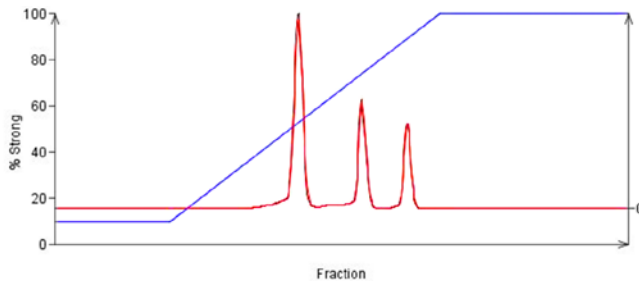
Silia*Sep* PREMIUM C18 25g
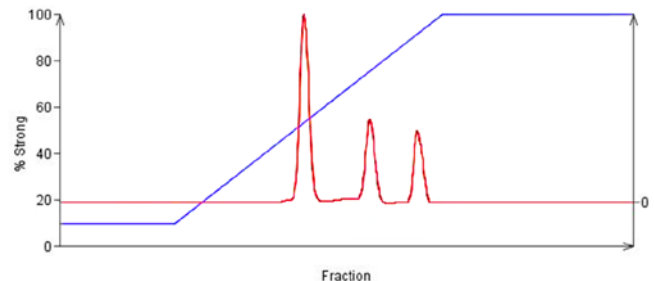
Silia*Sep* PREMIUM C18 330g

Silia*Sep* PREMIUM against the Competition

We compared the results on our Silia*Sep* PREMIUM against two established players in chromatography and purification. This study shows that Silia*Sep* PREMIUM cartridges perform very well and represent a valuable and reliable choice.
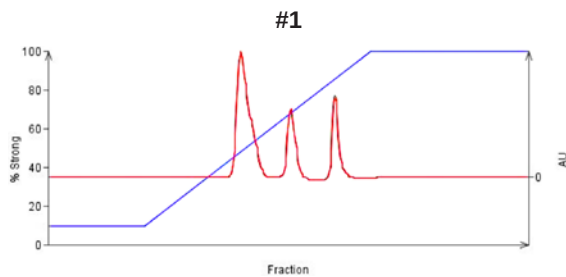
Chromatographic Conditions

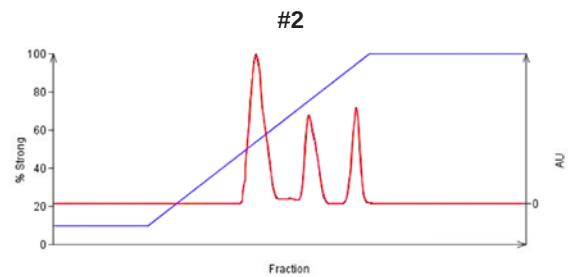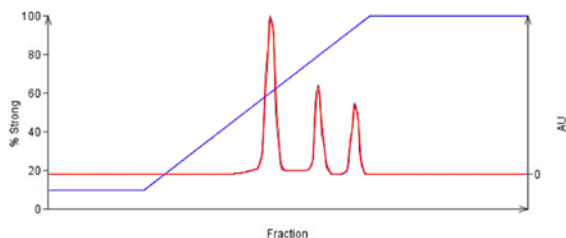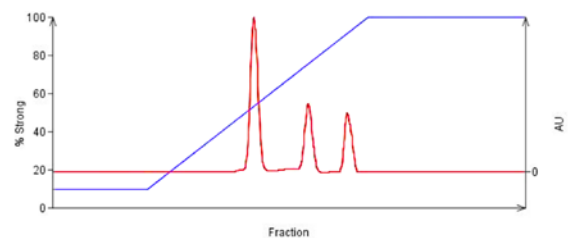**Mobile phase**: gradient Water / Methanol (*90:10 to 0:100*)

Flow rate: 40 mL/min

Injection volume: 0.5 mL

Wavelength: 254 nm

#1

Competitor I

#2

Competitor B

Silia*Sep* PREMIUM 12g

Silia*Sep* PREMIUM 40g

Silia*Sep* Cartridges Cleaning & 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 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 user responsibility. SiliCycle does not warranty any Flash cartridge for multiple injections and all process validation is the clients' responsibility.

Please see www.fda.gov/ohrms/dockets/dailys/04/oct04/101904/03n-0059-c00004-01-vol1.pdf, for the Guidance for Industry ChromPac, Manufacturing Chromatography Systems Post-Approval Changes: Chemistry, Manufacturing and Controls Document, submitted to Docket #03N-0059 Pharmaceutical cGMPs by bhr PDA for detailed discussion.

Guidelines for Flash cartridge use in cGMP environments	
Cartridge	**Recommended Use & Cleaning Procedure**
Bare Silica	Porous silica is used in adsorption chromatography processes, where the product and its impurities "bind" to the surface at different strengths. 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 or these impurities may elute in a subsequent run. Clearly, if the user wants to use the cartridge 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. The user must demonstrate each of the multiple injections gives the same elution profile and that the product purity is consistent in each of the sequential runs. Typically, users will set the process control points 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 user is considering this multiple lot strategy, the cleaning process will require a high level of data to support the decision.* • **Used for multiple batches of multiple API** SiliCycle is not aware of any user who has developed a validated process to run multiple different API's on a single silica cartridge. 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 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 full validated cleaning procedure must be implemented. If a user is considering this multiple lot strategy, 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 (*typically methanol, ethanol or acetonitrile*) in the elution mixture, often carried out in reverse flow mode. The cartridge must be re-equilibrated, in normal flow mode, with the initial solvent mix prior to the next injection. The cleaning step and re-equilibration step will each use a minimum of 3-column volumes each of solvent. SiliCycle recommends that Flash C18 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 user must set and define the upper and lower control limits 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 may be prohibitive.

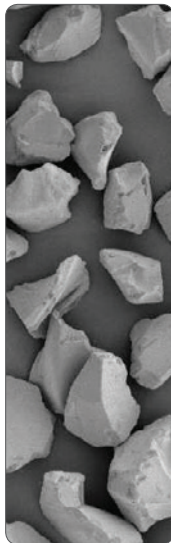SILICYCLE

Silia*Sep* Cartridges 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* C18 40 - 63 µm irregular grade, 4 g cartridge: FLH-R33230B-ISO04.
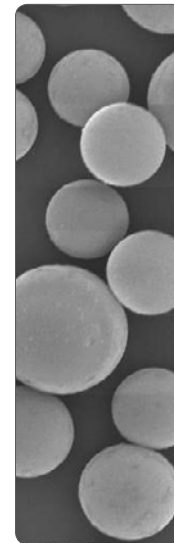
Silia*Sep* Phases

Silia*Sep* 40 - 63 µm Irregular Silica

Silia*Sep* Phases	
Phases	**Phase PN**
Bare Irregular Silica	**R10030B**
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**

FUNCTIONALIZED IRREGULAR SILICA

Silia*Sep* PREMIUM 25 µm Spherical Silica

Silia*Sep* Phases	
Phases	**Phase PN**
Bare Spherical Silica	**10095D-A**
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**

FUNCTIONALIZED SPHERICAL SILICA

Note: Other phases can be offered, like Metal Scavengers. Contact us for more information: info@silicycle.com.

Silia*Sep* Formats

Silia*Sep* Formats			
Formats	**Qty/Box***		**Format Code**
	Bare Silica	**Functionalized Silica**	
4 g	20	2	**ISO04**
12 g	20	1	**ISO12**
25 g	15	1	**ISO25**
40 g	15	1	**ISO40**
80 g	12	1	**ISO80**
120 g	10	1	**IS120**
220 g	4	1	**IS220**
330 g	4	1	**IS330**
XL 800 g	2	1	**IS750**
XL 1,600 g	2	1	**I1500**
XL 3,000	1	1	**I3000**

* Bigger box sizes available, contact us for more information: info@silicycle.com.

Notes:

• For bigger columns, please contact us.

• For Silia*Sep* XL formats, you will need to use an XL Adapter to plug the cartridge on your system. Part number: AUT-0127-1.

SiliaSep Solid-Load Cartridges Ordering Information

The use of solid-load technique (*also called dry-load*) will improve chromatography resolution, especially for compounds soluble only in strong solvents or in large volumes of solvents. SiliaSep solid-load luer-lock cartridges are designed to be used with SiliaSep flash cartridges for sample loading. To better suit your needs, two formats are available:

- **SiliaSep pre-packed solid-load** (*for liquid injection, various choices of media available: silica, amine, diol, cyano and C18*). 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.

- **SiliaSep empty solid-load** (*for silica-sample slurry, dry by evaporating the solvent for a more concentrated sample and to eliminate any solvent effect on the purification*). For a dry sample slurry, use a 1:1 ratio (*1 g of silica for 1 g of dry sample*) but for an oily sample prefer a 3:1 ratio (*3 g of silica for 1 g of oily sample*).

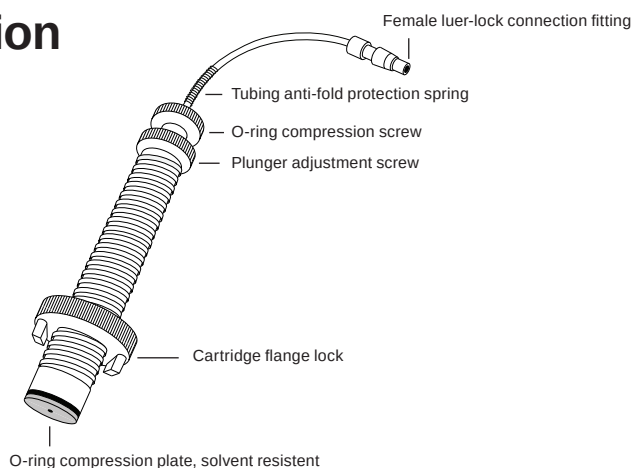| Product Number	Sorbent	Weight / Volume	Description	Qty / Box
SPL-R10030B-10U	Silica (*40 - 63 μm*)	2 g / 10 mL	SiliaSep Silica Pre-packed Solid-Load Cartridge, 2 g, 10 mL	20
SPL-R10030B-10X	Silica (*40 - 63 μm*)	5 g / 10 mL	SiliaSep Silica Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R10030B-60Y	Silica (*40 - 63 μm*)	10 g / 60 mL	SiliaSep Silica Pre-packed Solid-Load Cartridge, 10 g, 60 mL	16
SPL-R10030B-60K	Silica (*40 - 63 μm*)	25 g / 60 mL	SiliaSep Silica Pre-packed Solid-Load Cartridge, 25 g, 60 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-60K	Amine	25 g / 60 mL	SiliaSep Amine Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-R35030B-10X	Diol	5 g / 10 mL	SiliaSep Diol Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R35030B-60K	Diol	25 g / 60 mL	SiliaSep Diol Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-R38030B-10X	Cyano	5 g / 10 mL	SiliaSep Cyano Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R38030B-60K	Cyano	25 g / 60 mL	SiliaSep Cyano Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-R33230B-10X	C18 (*17 %*)	5 g / 10 mL	SiliaSep C18 (*17 %*) Pre-packed Solid-Load Cartridge, 5 g, 10 mL	20
SPL-R33230B-60K	C18 (*17 %*)	25 g / 60 mL	SiliaSep C18 (*17 %*) Pre-packed Solid-Load Cartridge, 25 g, 60 mL	16
SPL-0009-010	Empty	- / 10 mL	SiliaSep Empty Solid-Load Cartridge, 10 mL (*with 200 frits*)	100
AUT-0134	-	-	Frits for SiliaSep Empty Solid-Load Cartridge, 10 mL	100
SPL-0012-060	Empty	- / 60 mL	SiliaSep Empty Solid-Load Cartridge, 60 mL (*with 200 frits*)	100
AUT-0135	-	-	Frits for SiliaSep Empty Solid-Load Cartridge, 60 mL	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

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

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

SiliaSep Plungers Ordering Information

SiliaSep Plungers*	
Product Number	Description
AUT-0060-010	Plunger for 10 mL Solid-Load Cartridge (*16 mm*)
AUT-0060-060	Plunger for 60 mL Solid-Load Cartridge (*27 mm*)

Ask for a SiliaSep Plungers Operating Instructions Guide!

Female luer-lock connection fitting
Tubing anti-fold protection spring
O-ring compression screw
Plunger adjustment screw
Cartridge flange lock
O-ring compression plate, solvent resistent

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SiliaSep OT (*Open Top*) Flash Cartridges Ordering Information

SiliaSep 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: SiliaSep OT, C18, 15 g cartridge: FLH-R33230B-70i.

SiliaSep OT Phases	
Phases	**Phase PN**
Bare Silica	**R10030B**
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 OT Formats					
Formats	**Qty/Box**	**Dimensions** *(ID x length)*	**Volume**	**Format Code**	
				Bare Silica	**Functionalized Silica**
2 g	20	15.8 x 90 mm	12 mL	**15U**	**15U**
5 g	20	20.5 x 100 mm	25 mL	**25X**	**15U**
10 g	16	26.8 x 154 mm	70 mL	**70Y**	
15 g	16	26.8 x 154 mm	70 mL	**70i**	
20 g	16	26.8 x 154 mm	70 mL	**70Z**	
25 g	10	38.2 x 170 mm	150 mL	**95K**	
50 g	10	38.2 x 170 mm	150 mL	**95M**	
70 g	10	38.2 x 170 mm	150 mL	**95N**	
100 g	12	40.0 x 220 mm	276 mL	**276F**	

Notes:

- Other phases can be offered, contact us for more information: info@silicycle.com.

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

- Maximum operating pressure: 60 psi.

Silia*Sep* BT 75 Cartridges Ordering Information

These cartridges are designed to enhance your purifications when using Biotage™ Flash 75 development-scale purification systems. These cartridges offer a faster and safer solution compared to traditional glass columns.

Specifications

SiliaSep BT Specifications							
Cartridge	**Code**	**Silica Weight**	**Dimensions**	**Column Volume**	**Recommended Flow Rate**	**Loading Capacity**	**Max Operating Pressure**
Silia*Sep* BT 75S	75iS	200 g	75 x 90 mm	300 mL	100 - 250 mL/min	Bare: 0.2 - 20 g Bonded: 0.1 - 10 g	90 psi / 6.5 bar (inside compression module)
Silia*Sep* BT 75M	75iM	400 g	75 x 170 mm	500 mL		Bare: 0.4 - 40 g Bonded: 0.2 - 20 g	
Silia*Sep* BT 75L	75iL	800 g	75 x 350 mm	1 L		Bare: 0.8 - 80 g Bonded: 0.4 - 40 g	

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, Diol nec, 75iM cartridge: FLH-R35030B-75iM.

Silia*Sep* BT Phases	
Phases	**Phase PN**
Bare Irregular Silica	**R10030B**
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**

(Functionalized Irregular Silica applies to the phases from C18 through SAX-2 nec)

Silia*Sep* BT Formats				
Formats	**Silica Weight**	**Qty/Box**		**Format Code**
		Bare silica	**Functionalized Silica**	
75S	200 g	2	1	**75iS**
75M	400 g	2	1	**75iM**
75L	800 g	2	1	**75iL**

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

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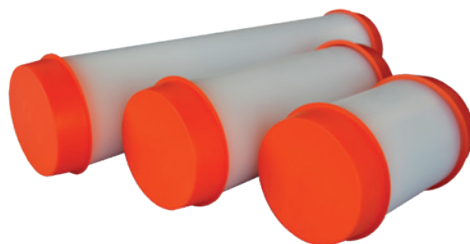
- Wide choice of sizes, sorbents & thicknesses available
- Excellent reproducibility between Silia*Plate* TLC plates and bulk silicas or Flash cartridges

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
- 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, high performance TLC *(also called HPTLC)* and preparative TLC *(PLC)*. The plate types are selected based on the analysis required and the available budget.

Differences Between Classical TLC, HPTLC and PLC			
Properties	**Classical TLC**	**HPTLC**	**Preparative (*PLC*)**
Applications	Quick, inexpensive, flexible and classical separations	Highly sophisticated separations, complex samples	Purification on a TLC plate
Analysis	Qualitative	Qualitative & Quantitative	Quantitative
Detection	UV - Stains	Instrumented analysis *(use of scanners for detection)*	UV
Distribution [*Mean Particle Size*]	5 - 20 µm [*10 - 14 µm*]	4 - 8 µm [*5 - 6 µm*]	5 - 40 µm [*22 - 25 µm*]
Layer Thickness	200 - 250 µm	150 - 200 µm	500 - 2,000 µm
Typical Sample Volume	1 - 5 µL	0.1 - 0.5 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 Low weight & consequent shipping costs High heating stability Low fragility 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 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
Total Weight	High	Low	Medium
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 *(TLC or HPLTC)*. The most common layer thicknesses are:

- 150 - 200 µm *(HPTLC plates)*
- 200 - 250 µm *(analytical TLC plates)*
- 500 - 2,000 µm *(preparative TLC plates)*

UV Indicator

Silia*Plate* TLC plates are mostly offered with an F_{254} UV indicator.

Some plates may be available with a F_{366} UV indicator, or without UV indicator. Contact us: info@silicycle.com.
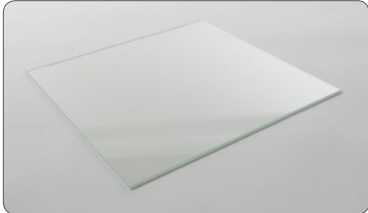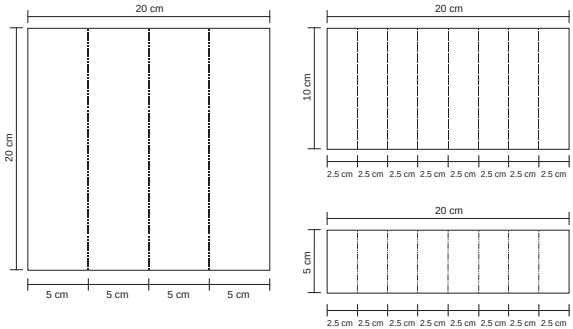
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Sorbents

Available Sorbents		
Classical Silica Gel	**Reversed-Phases**	**Special Phases**
A universal matrix for daily, fast, reliable analysis of the largest spectra of molecules	**The two most popular modes of separation employed in TLC are normal and reversed-phases.**	
The particle size distribution used for the silica is related to the nature of the plate.	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*).	
For standard TLC, silica gel with a mean particle size of 10 - 14 μm is used compared to HPTLC, where a smaller particle size is required.	When satisfactory separations cannot be achieved by unmodified silica, other functionalized matrices have been designed for specific applications:	
In both cases, pore diameter is always 60 Å.	C2, C8 and C18 phases are functionalization 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.

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.

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:	Also for your convenience, SiliCycle provides ready-to-use micro TLC plates in the following formats:	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 x 20 cm	2.5 x 10 cm	20 x 20 cm plates scored to four 5 x 20 cm plates *(or multiple of 5 cm width)*
10 x 20 cm	2.5 x 7.5 cm	10 x 20 cm plates scored to eight 2.5 x 10 cm plates *(or multiple of 2.5 cm width)*
5 x 20 cm	2.5 x 5 cm	5 x 20 cm plates scored to eight 2.5 x 5 cm plates *(or multiple of 2.5 cm width)*
5 x 10 cm		
10 x 10 cm		
Example:	Example:	

Binder

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

- **B**'s layer is polymeric: it has been added a small percentage of inorganic, hardening agent for a uniform and hard surface, smooth and dense, that will not crack, blister nor 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 quiclky choose the right plate for your specific application.

SiliaPlate 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 m²/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**	

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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**	**HPTLC**	**Preparative**
Available Backings			
Glass	Yes	Yes	Yes
Aluminum	Yes	No	No
Plastic	Yes	No	No
Available Adsorbents			
Bare silica	Yes	Yes	Yes
Functionalized Silica	No	Yes	Yes
Silica Specifications			
Mean Particle Size	10 - 14 µm	5 - 6 µm	22 - 25 µm
Mean Pore Diameter	60 Å	60 Å	60 Å
Type of Plate Available			
Scored Plate	Yes	No	Yes
Channeled Plate	Yes	No	No
Layer Thickness	Glass: 250 µm Flexible: 200 µm	Glass: 150 - 200 µm	Glass: • 500 µm • 1,000 µm Flexible: • 1,500 µm • 2,000 µm
Plate Size	• 2.5 x 5 cm • 2.5 x 7.5 cm • 2.5 x 10 cm • 5 x 10 cm • 5 x 20 cm • 10 x 20 cm • 20 x 20 cm	• 2.5 x 5 cm • 2.5 x 7.5 cm • 2.5 x 10 cm • 5 x 10 cm • 5 x 20 cm • 10 x 20 • 20 x 20 cm	• 20 x 20 cm

Solutions for Purification & Chromatography

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 x 7.5 cm	250 µm	F254	100
TLG-R10014B-424	Silica, Hard Layer	5 x 20 cm	250 µm	F254	100
TLG-R10014B-323	Silica, Hard Layer	20 x 20 cm	250 µm	F254	25
TLG-R10014B-323N	Silica, Hard Layer	20 x 20 cm	250 µm	None	25
TLGZ-R10011B-723	Silica with Preadsorbent Zone	10 x 20 cm	250 µm	F254	25
TLGZ-R10011B-323	Silica with Preadsorbent Zone	20 x 20 cm	250 µm	F254	25
TLG-R10014BK-417	Silica, optimized for $KMnO_4$ revelation	2.5 x 5 cm	250 µm	F254	200
TLG-R10014BK-124	Silica, optimized for $KMnO_4$ revelation	2.5 x 7.5 cm	250 µm	F254	100
TLG-R10014BK-527	Silica, optimized for $KMnO_4$ revelation	5 x 10 cm	250 µm	F254	200
TLG-R10014BK-424	Silica, optimized for $KMnO_4$ revelation	5 x 20 cm	250 µm	F254	100
TLG-R10014BK-725	Silica, optimized for $KMnO_4$ revelation	10 x 20 cm	250 µm	F254	50
TLG-R10014BK-323	Silica, optimized for $KMnO_4$ revelation	20 x 20 cm	250 µm	F254	25
TLG-R10014BK-323N	Silica, optimized for $KMnO_4$ revelation	20 x 20 cm	250 µm	None	25
TLG-R10014BKB-323	Silica, optimized for $KMnO_4$ revelation	20 x 20 cm	250 µm	F254 , F366	25
Channeled with preadsorbent zone					
TLGCZ-R10011B-423	Silica	5 x 20 cm	250 µm	F254	25
TLGCZ-R10011B-723	Silica	10 x 20 cm	250 µm	F254	25
TLGCZ-R10011B-323	Silica	20 x 20 cm	250 µm	F254	25
TLGCZ-R10011B-323N	Silica	20 x 20 cm	250 µm	None	25
Scored TLC plates					
TLGSR10011B-423	Silica	5 x 20 cm, scored to 2.5 x 5 cm	250 µm	F254	25
TLGSR10011B-424	Silica	5 x 20 cm, scored to 2.5 x 5 cm	250 µm	F254	100
TLGSR10011B-723	Silica	10 x 20 cm, scored to 2.5 x 10 cm	250 µm	F254	25
TLGSR10011B-323	Silica	20 x 20 cm, scored to 5 x 20 cm	250 µm	F254	25
Functionalized silica & other adsorbents					
TLG-AUT0014-423	Florisil	5 x 20 cm	250 µm	F254	25
TLG-AUT0014-723	Florisil	10 x 20 cm	250 µm	F254	25
TLG-AUT0014-323	Florisil	20 x 20 cm	250 µm	F254	25
TLG-AUT0337-323B	Basic Alumina	20 x 20 cm	250 µm	F254	25
TLG-AUT0337B-424N	Neutral Alumina	5 x 20 cm	250 µm	F254	100
TLG-AUT0337-323N	Neutral Alumina	20 x 20 cm	250 µm	F254	25
TLG-AUT0337-323NF	Neutral Alumina	20 x 20 cm	250 µm	None	25
TLG-AUT0337B-323N	Neutral Alumina	20 x 20 cm	250 µm	F254	25

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Glass-backed HPTLC Plates

	Glass-backed Analytical HPTLC Plates				
PN	**Sorbent**	**Plate Size**	**Thickness**	**UV indicator**	**Qty/box**
Silica					
HPTLG-R10011B-1010	Silica	10 x 10 cm	150 µm	F_{254}	25
HPTLG-R10011B-2020	Silica	20 x 20 cm	150 µm	F_{254}	25
HPTLG-R10014BK-1010	Silica, optimized for $KMnO_4$ revelation	10 x 10 cm	150 µm	F_{254}	25
HPTLG-R10014BK-1020	Silica, optimized for $KMnO_4$ revelation	10 x 20 cm	150 µm	F_{254}	25
HPTLG-R10014BK-2020	Silica, optimized for $KMnO_4$ revelation	20 x 20 cm	150 µm	F_{254}	25
HPTLGSR10011B-1010	Silica	10 x 10 cm, scored to 5 x 5 cm	150 µm	F_{254}	25
HPTLGSR10011B-1020	Silica	10 x 20 cm, scored to 2.5 x 10 cm	150 µm	F_{254}	25
HPTLGZ-R10011B-203	Silica with Preadsorbent Zone	10 x 10 cm	150 µm	F_{254}	25
HPTLGZ-R10011B-703	Silica with Preadsorbent Zone	10 x 20 cm	150 µm	F_{254}	25
Functionalized silica & other adsorbents					
TLG-R30314BK-213	C18 (*100 %*), optimized for $KMnO_4$ revelation	10 x 10 cm	150 µm	F_{254}	25
TLG-R30314BK-213N	C18 (*100 %*), optimized for $KMnO_4$ revelation	10 x 10 cm	150 µm	None	25
TLG-R30411B-213	C18 (*13 %*)	10 x 10 cm	150 µm	F_{254}	25
TLG-R30411B-303	C18 (*13 %*)	20 x 20 cm	150 µm	F_{254}	25
TLG-R30414B-313	C18 (*13 %*)	20 x 20 cm	200 µm	F_{254}	25
TLG-R30411B-323	C18 (*13 %*)	20 x 20 cm	250 µm	F_{254}	25
TLG-R30414BK-213	C18 (*13 %*), optimized for $KMnO_4$ revelation	10 x 10 cm	200 µm	F_{254}	25
TLG-R30414BK-313	C18 (*13 %*), optimized for $KMnO_4$ revelation	20 x 20 cm	200 µm	F_{254}	25
TLG-R31011B-203	C8	10 x 10 cm	150 µm	F_{254}	25
TLG-R31011B-303	C8	20 x 20 cm	150 µm	F_{254}	25
TLG-R32611B-203	C2	10 x 10 cm	150 µm	F_{254}	25
TLG-R32611B-303	C2	20 x 20 cm	150 µm	F_{254}	25
TLG-R32614BK-313	C2, optimized for $KMnO_4$ revelation	20 x 20 cm	200 µm	F_{254}	25
TLG-R32614BK-713	C2, optimized for $KMnO_4$ revelation	10 x 20 cm	200 µm	F_{254}	25
TLG-R35011B-713	Diol	10 x 20 cm	200 µm	F_{254}	25
TLG-R35014BK-213	Diol, optimized for $KMnO_4$ revelation	10 x 10 cm	200 µm	F_{254}	25
TLG-R35014BK-713	Diol, optimized for $KMnO_4$ revelation	10 x 20 cm	200 µm	F_{254}	25
TLG-R35014BK-313	Diol, optimized for $KMnO_4$ revelation	20 x 20 cm	200 µm	F_{254}	25
TLG-R38011B-203	Cyano (*CN*)	10 x 10 cm	150 µm	F_{254}	25
TLG-R38011B-723	Cyano (*CN*)	10 x 20 cm	150 µm	F_{254}	25
TLG-R38011B-303	Cyano (*CN*)	20 x 20 cm	150 µm	F_{254}	25
TLG-R38014BK-213	Cyano (*CN*), optimized for $KMnO_4$ revelation	10 x 10 cm	200 µm	F_{254}	25
TLG-R38014BK-713	Cyano (*CN*), optimized for $KMnO_4$ revelation	10 x 20 cm	200 µm	F_{254}	25
TLG-R38014BK-313	Cyano (*CN*), optimized for $KMnO_4$ revelation	20 x 20 cm	200 µm	F_{254}	25
TLG-R52011B-203	Amine (*NH_2*)	10 x 10 cm	150 µm	F_{254}	25
TLG-R52011B-723	Amine (*NH_2*)	10 x 20 cm	150 µm	F_{254}	25
TLG-R52011B-303	Amine (*NH_2*)	20 x 20 cm	150 µm	F_{254}	25
TLG-R52014BK-213	Amine (*NH_2*), optimized for $KMnO_4$ revelation	10 x 10 cm	200 µm	F_{254}	25
TLG-R52014BK-713	Amine (*NH_2*), optimized for $KMnO_4$ revelation	10 x 20 cm	200 µm	F_{254}	25
TLG-R52014BK-313	Amine (*NH_2*), optimized for $KMnO_4$ revelation	20 x 20 cm	200 µm	F_{254}	25
TLG-R23511B-423	$AgNO_3$ (*10 %*)	5 x 20 cm	250 µm	F_{254}	25
TLG-R23511B-303	$AgNO_3$ (*10 %*)	20 x 20 cm	250 µm	F_{254}	25
TLG-R23611B-423	$AgNO_3$ (*15 %*)	5 x 20 cm	250 µm	F_{254}	25
TLG-R23611B-323	$AgNO_3$ (*15 %*)	20 x 20 cm	250 µm	F_{254}	25
TLG-R23711B-423	$AgNO_3$ (*20 %*)	5 x 20 cm	250 µm	F_{254}	25
TLG-R23711B-323	$AgNO_3$ (*20 %*)	20 x 20 cm	250 µm	F_{254}	25
TLG-R23M11B-323	$AgNO_3$ (*5-10-15-20%, 5 TLC each*)	5 x 20 cm	250 µm	F_{254}	5 x 4
TLGSR1234511B-723	Trial Packing of Functionalized Silica	10 x 20 cm, scored to 2.5 x 10 cm	150 µm	F_{254}	25
TLG-AUT0308-203	RP Silanized	10 x 10 cm	150 µm	F_{254}	25

Solutions for Purification & Chromatography

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Glass-backed Preparative TLC Plates

PN	Sorbent	Plate Size	Thickness	UV indicator	Qty/box
Glass-backed Preparative TLC Plates					
Silica					
TLG-R10011B-333	Silica	20 x 20 cm	500 µm	F_{254}	25
TLG-R10011B-341	Silica	20 x 20 cm	1,000 µm	F_{254}	25
TLG-R10011B-363	Silica	20 x 20 cm	1,500 µm	F_{254}	25
TLG-R10011B-353	Silica	20 x 20 cm	2,000 µm	F_{254}	25
Functionalized silica & other adsorbents					
TLG-AUT0337-343N	Neutral Alumina	20 x 20 cm	1,000 µm	F_{254}	25
TLG-AUT0337-343NF	Neutral Alumina	20 x 20 cm	1,000 µm	None	25
TLG-AUT0337-443	Neutral Alumina	5 x 20 cm	1,000 µm	F_{254}	25
TLG-AUT0337-443F	Neutral Alumina	5 x 20 cm	1,000 µm	None	25
TLG-AUT0337B-341N	Neutral Alumina	20 x 20 cm	1,000 µm	None	15
TLG-R23511B-433	AgNO$_3$ (*10 %*)	5 x 20 cm	500 µm	F_{254}	25
TLG-R23511B-333	AgNO$_3$ (*10 %*)	20 x 20 cm	500 µm	F_{254}	25
TLG-R23611B-433	AgNO$_3$ (*15 %*)	5 x 20 cm	500 µm	F_{254}	25
TLG-R23611B-333	AgNO$_3$ (*15 %*)	20 x 20 cm	500 µm	F_{254}	25
TLG-R23711B-433	AgNO$_3$ (*20 %*)	5 x 20 cm	500 µm	F_{254}	25
TLG-R23711B-333	AgNO$_3$ (*20 %*)	20 x 20 cm	500 µm	F_{254}	25
TLG-R30411B-341	C18 (*13 %*)	20 x 20 cm	1,000 µm	F_{254}	15
TLG-R30414BK-341	C18 (*15 %*), optimized for KMnO$_4$ revelation	20 x 20 cm	1,000 µm	F_{254}	15
Scored preparative TLC plates					
TLGSR10011B-333	Silica	20 x 20 cm, scored to 5 x 20 cm	500 µm	F_{254}	25
TLGSR10011B-341	Silica	20 x 20 cm, scored to 5 x 20 cm	1,000 µm	F_{254}	25
TLGSR10011B-353	Silica	20 x 20 cm, scored to 5 x 20 cm	2,000 µm	F_{254}	25

SILICYCLE

Aluminum-backed TLC Plates

	Aluminum-backed Analytical TLC Plates				
PN	**Sorbent**	**Plate Size**	**Thickness**	**UV indicator**	**Qty/box**
Silica					
TLA-R10011B-005	Silica	4 x 8 cm	150 µm	F_{254}	50
TLA-R10011B-124	Silica	2.5 x 7.5 cm	200 µm	F_{254}	200
TLA-R10011B-323	Silica	20 x 20 cm	200 µm	F_{254}	25
TLA-R10011B-323N	Silica	20 x 20 cm	200 µm	None	25
TLA-R10011B-415	Silica	5 x 20 cm	200 µm	F_{254}	50
TLA-R10011B-515	Silica	5 x 10 cm	200 µm	F_{254}	50
TLA-R10011B-712	Silica	10 x 20 cm	200 µm	F_{254}	20
TLA-R10014BK-1112	Silica, optimized for $KMnO_4$ revelation	5 x 7.5 cm	200 µm	F_{254}	20
Functionalized silica & other adsorbents					
TLA-AUT0337-323N	Neutral Alumina	20 x 20 cm	200 µm	F254	25
TLA-AUT0337-323NF	Neutral Alumina	20 x 20 cm	200 µm	None	25
TLA-R30411B-005	Silica C18 (13 %)	4 x 8 cm	150 µm	F254	50
TLA-R30411B-303	Silica C18 (13 %)	20 x 20 cm	150 µm	F254	25
TLA-R30411B-405	Silica C18 (13 %)	5 x 20 cm	150 µm	F254	50
TLA-R30411B-505	Silica C18 (13 %)	5 x 10 cm	150 µm	F254	50
TLA-R30414BK-303	Silica C18 (13 %), opt. for KMnO4 revelation	20 x 20 cm	150 µm	F254	25
TLA-R52014BK-005	Amine (NH2), optimized for KMnO4 revelation	4 x 8 cm	150 µm	F254	50

	Aluminum-backed Analytical HPTLC Plates				
PN	**Sorbent**	**Plate Size**	**Thickness**	**UV indicator**	**Qty/box**
Silica					
HPTLA-R10011B-323	Silica	20 x 20 cm	150 µm	F_{254}	25
HPTLA-R10011B-323N	Silica	20 x 20 cm	150 µm	None	25

Solutions for Purification & Chromatography

Plastic-backed TLC Plates

Plastic-backed Analytical TLC Plates					
PN	Sorbent	Plate Size	Thickness	UV indicator	Qty/box
Silica					
TLP-R10011B-005N	Silica	4 x 8 cm	150 µm	None	50
TLP-R10011B-117	Silica	2.5 x 7.5 cm	200 µm	F254	200
TLP-R10011B-323	Silica	20 x 20 cm	200 µm	F254	25
TLP-R10011B-323N	Silica	20 x 20 cm	200 µm	None	25
TLP-R10014B-0115	Silica	5 x 6.7 cm	200 µm	F254	50
TLP-R10014BK-0115	Silica, optimized for KMnO4 revelation	5 x 6.7 cm	200 µm	F254	50
TLP-R10014BK-0116	Silica, optimized for KMnO4 revelation	3.3 x 6.6 cm	200 µm	F254	50
Other adsorbents					
TLP-R83014BK-303	Polyamide-6, optimized for KMnO4 revelation	20 x 20 cm	100 µm	F254	25
TLP-R83014BK-405	Polyamide-6, optimized for KMnO4 revelation	5 x 20 cm	100 µm	F254	50
TLP-R83014BK-405N	Polyamide-6, optimized for KMnO4 revelation	5 x 20 cm	100 µm	None	50
TLP-R83014BK-303N	Polyamide-6, optimized for KMnO4 revelation	20 x 20 cm	100 µm	None	25

SILICYCLE

TLC Accessories

PN	TLC Accessories		Qty/box
	Accessory		
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	TLC Plate (20 x 20 cm) Cutter		1
AUT-0183	Replacement Scriber for TLC Plate Cutter		1
AUT-1182	TLC Plate Pencil Glass Cutter		1

Solutions for Purification & Chromatography
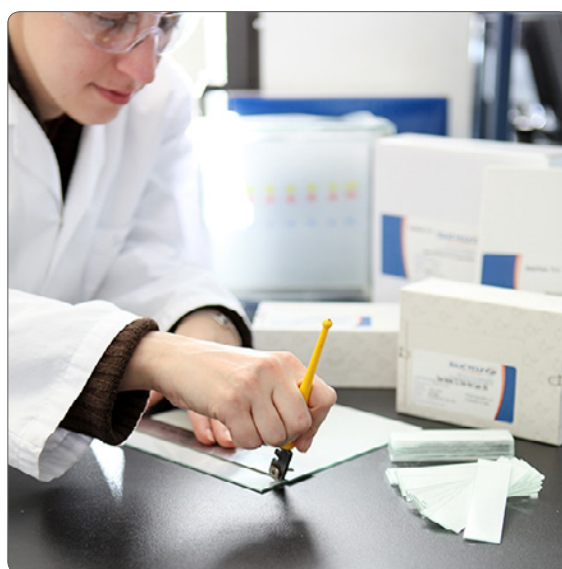
Thin Layer Chromatography Practical Guide

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$_3$N*) 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)* & 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.

SILICYCLE

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.

Solutions for Purification & Chromatography
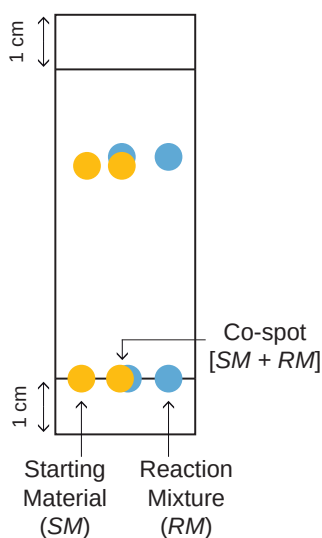
TLC Plate Development

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. See page 46.

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.

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

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   &   **ΔCV = 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.

SILICYCLE
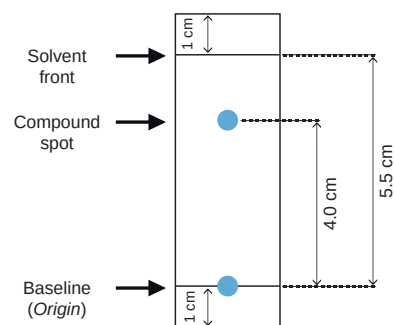
TLC Visualization Methods

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

N.B. Shaded lines refer to "Universal stains" ; "BG" stands for "background".

Stains for Thin Layer Chromatography			
Name	**Visualization of...**	**Stain Recipe**	**Comments**
p-Anisaldehyde #1	**Universal stain** *Good for nucleophiles and oxygenated compounds*	**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
Note: Tends to be insensitive to alkenes, alkynes and aromatic compounds unless other functional groups are present.			
p-Anisaldehyde #2	*Acronycine* *Cineoles* *Terpenes*	**Prepare stain as follows [*1:10:20:80*]** • p-anisaldehyde • perchloric acid • acetone • 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*	**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
Note: Highly sensitive stain; very low concentration of product may appear as a significant impurity.			
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
Chromic Acid	**Difficultly stainable compounds**	**Prepare stain as follows** • 2.5 g of potassium chromate • 100 mL of 20 % sulfuric acid in water	
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

Stains for Thin Layer Chromatography			
Name	**Visualization of...**	**Stain Recipe**	**Comments**
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***
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 & 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*	**Prepare stain as follows** • 0.1 % 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 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.*	**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

Note: Can be used for detection of alcohols, amines, sulfides and mercaptans groups when gently heated.

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

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.

SILICYCLE

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 (*see page 42*).

- 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.
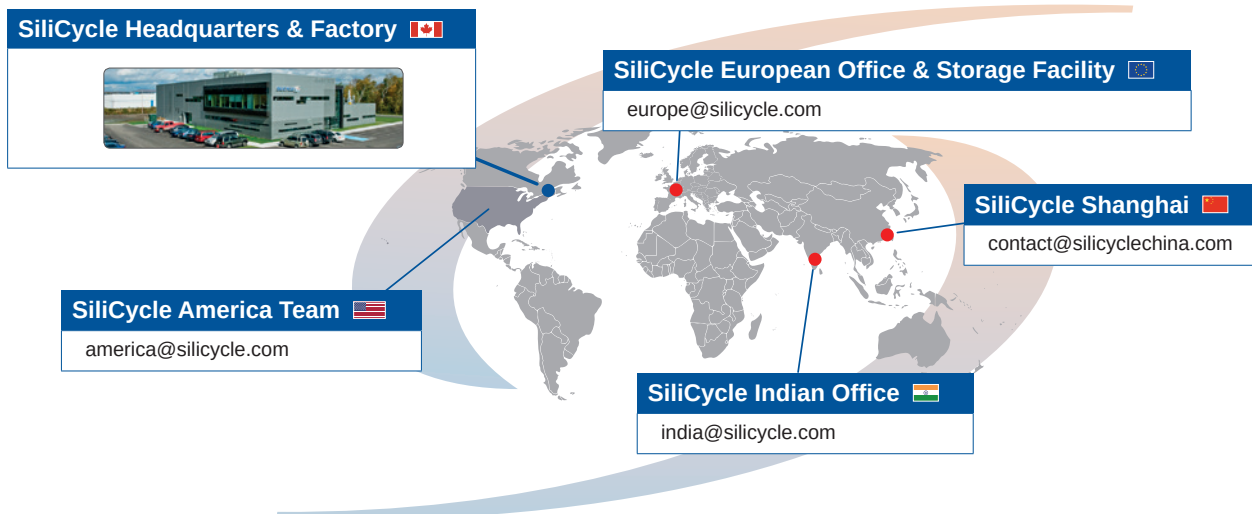
Contact us

📍 **SiliCycle Inc.**
2500, Parc-Technologique Blvd,
Quebec City (QC) G1P 4S6
CANADA 🇨🇦

✉ E-mail: **info@silicycle.com**

📞 Phone: **+1 418.874.0054**
Toll Free **+1 877.745.4292** (*North America only*)

🖨 Fax: **+1 418.874.0355**

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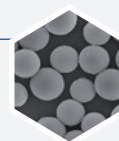**Silia*Plate*™** − TLC Plates

SAMPLE PREPARATION

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Silia*Prep*™ − Micro-SPE Tips
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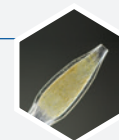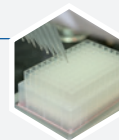
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SiliCycle Inc.
2500, Parc-Technologique Blvd,
Quebec City (Quebec) G1P 4S6
CANADA

Phone: **+1 418.874.0054**
Toll Free **+1 877.745.4292** (*North America only*)
Fax: **+1 418.874.0355**

Email: **info@silicycle.com**

Website: **www.silicycle.com**

Overseas Offices

SiliCycle Europe   europe@silicycle.com
SiliCycle India   india@silicycle.com
SiliCycle Shanghai   contact@silicyclechina.com

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