SOLUTIONS FOR PURIFICATION & CHROMATOGRAPHY







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

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Bulk Silicas for 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
- SiliaSphere[™] PC Spherical silicas

SiliCycle: Silica Expert

With pore diameters ranging from 30 to 1,000 Ångström (Å) and particle sizes up to 1,200 microns (μ m), 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 SiliaFlash & SiliaSphere 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 (<i>smaller dead volume</i>)			
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 SiliaFlash Irregular or SiliaSphere 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 SiliaSphere 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

SiliaSphere 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.





SiliaFlash & SiliaSphere PC Characteristics

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

Importance	e 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 preferential pathways (channeling)	No molecule sequestration due to fluid diffusion incide pares		
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





SiliCycle

Competitor

Effects of Homogeneous vs Uneven Packing

The connection between particle size distribution and column performance is very simple. When the distribution is broad, the packing is uneven. Some parts are composed of only large particles where the solvent will flow fast and meet little resistance, and there are sections composed of small particles where the solvent flows slowly and meets great resistance.

As a result, the solvent will take the path of least resistance through the column and flow around the pockets of small particles instead of straight through the column.

This uneven flow greatly affects the separation because the compounds will have different retention times depending on their flow path. As they exit the column, the compounds will give broad and poorly separated peaks.

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



Preferential

path chosen

Narrow Particle Size Distribution



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 (<i>mg/kg</i>)							
Metals		SiliCycle F60 R10030B	Manufacturer A	Manufacturer B			
Aluminum	AI	33	262	280			
Barium	Ва	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			





SiliaFlash 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.







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: SiliaFlash F60 (R10030B) VS SiliaFlash P60 (R12030B)

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

Two	o Different Grades of 40 - 63 μ	m, 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 removedLower price



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

60 - 200 µm, 60 Å Gels: SiliaFlash G60 (R10040B) VS SiliaFlash GE60 (R10140B)

-		
Τως	Different Grades of 60 - 200 µ	um, 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

Each grade offers its own particle size distribution profile.

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





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

		Typical Metal Content of Most Popular Irregular Silicas					
Product		F60	P60	Acid Washed*	G60	GE60	
Product Number		R10030B	R12030B	R10530B	R10040B	R10140B	
Particle Size	ticle Size		40 - 63 µm			00 μm	
Pore Diameter			60 Å		60	Å	
Metal (<i>mg/kg</i>)							
Aluminum	Al	< 200	< 1,000	< 70	< 350	< 900	
Antimony	Sb		< 0.2		< 0	0.2	
Arsenic	Ar		< 1		<	1	
Barium	Ва	< 40	< 40	< 5	< 4	40	
Beryllium	Ве		< 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	Со		< 0.1		< (0.1	
Copper	Cu		< 1		<	1	
Iron	Fe	< 75	< 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	Мо		< 0.1		< 0.1		
Nickel	Ni		< 1		< 1		
Potassium	К	< 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	Те		< 0.1		< (0.1	
Thallium	TI		< 0.1		< (0.1	
Tin	Sn	< 0.4 < 0.4 < 0.2			< ().4	
Titanium	Ti	< 200	< 250	< 90	< 2	50	
Uranium	U		< 0.1		< 0.1		
Vanadium	V		< 1		<	1	
Zinc	Zn		< 1		<	1	

* Acid washed SiliaFlash 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.



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.





A Particle Size for Each Application

		Most Popular Particle Size Applications				
Particle Size Irregular Particles	Distribution Spherical Particles	Applications				
Particles for Preparative	TLC Plates					
From 0 to 20 μm	-	 Contains neither binder (<i>organic or inorganic</i>) nor UV indicator (<i>F</i>₂₅₄) Can also be used in flash chromatography if higher resolution is required (<i>higher back-pressure</i>) 				
Particles for Difficult Sep	arations					
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 				
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				





SiliaFlash & SiliaSphere PC Ordering Information

This is only an overview of gels we can provide. Please contact us if you are looking for a different product: <u>info@silicycle.com</u>. Available formats: from 1 kg to 25 kg, even up to multi-ton scale!

Silia <i>Flash</i> Irregular Silica Gels Portfolio				SiliaSpher	e PC Spherica	l Silica Gels	Portfolio	
Product Number	Partic	le Size	Pore Diameter (Å)	Product Number	Partic	e Size	Pore Diameter (Å)	
r roduct Number	μm	mesh	Tore Diameter (A)	Troduct Number	μm	mesh	Tore Diameter (A)	
R10137L	75 - 150	100 - 200	30	S10095W-A	25	*	50	
R10130A	40 - 63	230 - 400		S10030B-A	50	300		
R10150A	60 - 120	325 - 625]	S10027B-A	60	250]	
R10140A	60 - 200	70 - 230	40	S10034B-A	75	200]	
R10160A	120 - 200	70 - 125	40	S10040B-A	100	150	60	
R10170A	200 - 500	35 - 70		S10063B-A	150	100		
R10180A	500 - 1,000	18 - 35		S10066B-A	200	70		
R10117B	15 - 40	*		S10068B-A	300	50		
R10023B	20 - 45	*]	S10020C	20 - 45	*		
R10030B (<i>F60</i>)				S10040C	75 - 200	70 - 200	70	
R12030B (P60)	40 - 63	230 - 400		S10030C	40 - 75	200 - 400		
R10530B (Acid-Washed)				S10070C	200 - 500	35 - 70		
R10150B	60 - 120	325 - 625		S10095D-A	25	*	90	
R10040B (G60)	60 200	70 220	60	S10020E	20 - 45	*		
R10140B (GE60)	00-200	70-230		S10030E	40 - 75	200 - 400		
R10137B	75 - 150	100 - 200		S10040E	75 - 200	70 - 200	100	
R10157B	105 - 175	86 - 140		S10065E	150 - 250	60 - 100		
R10160B	120 - 200	70 - 125		S10070E	200 - 500	35 - 70		
R10165B	150 - 250	60 - 100]	S10030G-A	50	300		
R10170B	200 - 500	35 - 70		S10034G-A	70	200	120	
R10180B	500 - 1,000	18 - 35		S10040G-A	100	150	120	
R10130D	40 - 63	230 - 400		S10063G-A	150	100		
R10140D	60 - 200	70 - 230		S10020M	20 - 45	*	_	
R10157D	105 - 175	86 - 140		S10030M	40 - 75	200 - 400	300	
R10170D	200 - 500	35 - 70	50	S10040M	75 - 200	70 - 200	300	
R10180D	500 - 1,000	18 - 35]	S10070M	200 - 500	35 - 70		
R10181D	800 - 1,200	16 - 22		S10020P	20 - 45	*		
R10130H	40 - 63	230 - 400		S10030P	40 - 75	200 - 400	500	
R10150H	60 - 120	325 - 625		S10040P	75 - 200	70 - 200		
R10140H	75 - 250	60 - 200]	S10070P	200 - 500	35 - 70		
R10157H	105 - 175	86 - 140		S10020S	20 - 45	*		
R10160H	120 - 200	70 - 125	150	S10030S	40 - 75	200 - 400	800	
R10170H	200 - 500	35 - 70		S10040S	75 - 200	70 - 200	000	
R10072H	250 - 500	35 - 60]	S10070S	200 - 500	35 - 70		
R10180H	500 - 1,000	18 - 35]	S10020T	20 - 45	*		
R10181H	800 - 1,200	16 - 22		S10030T	40 - 75	200 - 400	1 000	
R10130M	40 - 63	230 - 400		S10040T	75 - 200	70 - 200	1,000	
R10140M	60 - 200	70 - 230	300	S10070T	200 - 500	35 - 70		
B10170M	200 - 500	35 - 70	1					

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

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

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





Chromatographic Phases

Thanks to its high mechanical resistance, silica is the most widely used media in chromatography. With SiliaBond 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!

SiliaBond Phases for Low Pressure Chromatography

For all our listed SiliaBond sorbents, particle size is 40 - 63 µm and pore diameter is 60 Å. But we can graft any irregular SiliaFlash or spherical SiliaSphere PC silica gel, with the function of your choice. Contact us for more information.

All functionalized SiliaBond sorbents are available in bulk but also pre-packed in SiliaSep flash cartridges and SiliaPrep 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:





Sorbent Selection Chart

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





Reversed & Normal Phases Portfolio

Available formats: from 5 g to 25 kg.

Low Pressure Chromatography Reversed & Normal Phases Characteristics					
	Sorbent	Structure	Typical Characteristics	Typical Applications	
	C18 PN: R33230B	S	% 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	S	% 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	G	% C: ≥ 9.5 % Density: 0.662 g/mL	 Less retention compared to C18 and C8 Additional steric interaction 	
l-phases	Pentafluorophenyl (<i>PFP</i>) PN: R67530B		% C: ≥ 9.0 % Density: 0.761 g/mL	 For an alternative selectivity, with aromatic ring interactions For purification of conjugated compounds (<i>isomers</i>) 	
eversed	Phenyl (<i>PHE</i>) PN: R34030B	G -{>	% C: ≥ 8.0 % Density: 0.637 g/mL	 Moderately non-polar sorbent Alternative selectivity for aromatic compounds, compared to other reversed-phases 	
œ	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	S - C1	% 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 (<i>CN</i>) PN: R38030B	S N	% 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 (<i>Si</i>) PN: R10030B	SI-ОН	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 	
Ises	Diol nec PN: R35030B	Ся сторон	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 	
mal Pha	Amine (<i>NH₂, WAX</i>) PN: R52030B	SI NH2	Loading: ≥ 1.2 mmol/g Density: 0.700 g/mL	For purification of compounds with basic properties, or for monosaccharides separation	
Nor	Neutral Alumina PN: AUT-0054	Al ₂ O 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 (<i>PCBs</i>) & polysaccharides	
	Silver Nitrate (AgNO ₃) PN: R23530B	(s) + 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)	

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.

Ya.	Typical Applications Using Reversed and Normal Phases											
Analytes	Examples 0		C8	C6	PFP	PHE	C 4	C1	CN	NH ₂	Si	Diol
Biomolecules	Peptides, proteins	\checkmark	\checkmark	\checkmark			\checkmark	\checkmark	H	-		\checkmark
Nucleotides	Deoxyribonucleotides, ribonucleotides	\checkmark								\checkmark		
Lipids	Phospholipids		\checkmark	\checkmark			\checkmark	\checkmark		\checkmark		
Carbohydrates	Sugars								\checkmark	\checkmark		\checkmark
Glycosides	Glucosides, fructosides								\checkmark	\checkmark		\checkmark
Oligosaccharides	Malto-oligosaccharides									\checkmark		\checkmark
Pesticides	Organophosphates	\checkmark	\checkmark									
PCBs	Dichlorobiphenyl, trichlorobiphenyl	\checkmark			\checkmark	\checkmark						
PAHs	Anthracene, pyrene	\checkmark	\checkmark		\checkmark	\checkmark						
Drugs	Basic drugs, metabolites	\checkmark	\checkmark	\checkmark					\checkmark	\checkmark	\checkmark	
Alkaloids	Cocaine, morphine, nicotine, quinine	\checkmark	\checkmark						\checkmark		\checkmark	
Analgesics	Aspirin, acetaminophen, ibuprofen	\checkmark	\checkmark		\checkmark				\checkmark			
Cyclosporine	-	\checkmark							\checkmark			
Conjugated Compounds	Phenols, chloroanilines, steroids, caffeine	\checkmark										
Natural Compounds	Tannins, aflatoxins, flavonoids, carotenoids	\checkmark										
Fat-Soluble Vitamins	Vitamins A, D, E and K	\checkmark	\checkmark									
Water-Soluble Vitamins	Vitamins B and C									\checkmark	\checkmark	
Heterocyclic Compounds	Dioxins, furans	\checkmark										

AgNO₃ is particularly useful to separate isomers that present unsaturated groups.

Neutral Alumina is used for the separation of aldehydes, ketones, quinines, esters, lactones and glucosides. Florisil will help analyze pesticides, PCBs and PAHs.







Ion Exchange Phases Portfolio

Low Pressure Chromatography Ion Exchange Phases Characteristics								
	Sorbent	Structure	Typical Characteristics	Typical Applications				
	Amine (<i>NH</i> ₂ , <i>WAX</i>) PN: R52030B	SI NH2	Loading: ≥ 1.2 mmol/g Density: 0.700 g/mL	 Weak anion exchanger (<i>pK_a</i> of 9.8), positively charged at pH below 7.8 For very strong anions (<i>such as sulfonic acids</i>), that may be too strongly retained on SAX phases 				
	WAX-2 (<i>Triethylamine</i>) PN: R76530B	S ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	Loading: ≥ 1.04 mmol/g Density: 0.761 g/mL	 Weak anion exchanger (<i>pK_a</i> of 10.5), positively charged at pH below 8.5 For catch & release of compounds bearing a permanent negative charge (<i>like salts of sulfonic acids</i>) 				
ge Phases	SAX (<i>TMA Chloride</i>) PN: R66530B	SI ~~~ N ⁺ ⊂I-	Loading: ≥ 0.90 meq/g Density: 0.700 g/mL	 Strong anion exchanger, permanently positively charged (<i>pH independant</i>) For weak anions (<i>such as carboxylic acids</i>) that may not bind strongly enough on WAX phases For analysis of acidic drugs / analgesics, biomolecules & water-soluble vitamins 				
n Exchang	SAX-2 (<i>TMA Acetate</i>) PN: R66430B	SI ~~ I ⊂ H ₃ COO-	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) 				
<u>o</u>	SCX (<i>Tosic Acid</i>) PN: R60530B		Loading: ≥ 0.54 meq/g Density: 0.698 g/mL	 Strong cation exchangers (pK_a < 1), permanently negatively charged (pH independant) 				
-	SCX-2 (<i>Propylsulfonic Acid</i>) PN: R51230B	S CH	Loading: ≥ 0.63 meq/g Density: 0.728 g/mL	 For catch and release purification of weak cations (basic drugs / analgesics, biomolecules & water-soluble vitamins) 				
	WCX (Carboxylic Acid) PN: R70030B	Сястон	Loading: ≥ 0.92 mmol/g Density: 0.687 g/mL	 Weak cation exchanger (<i>pK_a of 4.8</i>), 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.





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 μ m, 60 Å: R33230B-100G.

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

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

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







Flash Cartridges

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 SiliaSep, benefit from the same quality that all our products are known for: selectivity, speed & reliability.

SiliaSep[™] Flash Cartridges

Cartridge Design



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 SiliaSep flash cartridges to accomodate your chemistry.

	SiliaSep Flash Cartridges Adsorbents		
Adsorbent type	Adsorbent	-	MACTELE Martine and Martine an
Bare silica	 Standard Silia<i>Flash</i> 40 - 63 μm Irregular Silica PREMIUM 25 μm Spherical Silica NEW (<i>learn more page 24</i>) 	1.1	
Bonded phases	 SiliaBond Chromatographic Phases (C18, C8, Phenyl, PFP, etc.) SiliaMetS Metal Scavengers (Thiol, DMT, etc.) SiliaBond Organic Scavengers (Amine, Tosic Acid, etc.) 		91

Formats

Ý			Silia	Sep Flash Cartridge	es Portfolio	D			
	Cartridge [Code]	Silica weight	Quantity	Dimensions (Diam. x Length*)	Column volume	Recommended flow rate	Loading capacity	Max operating pressure	
	Silia Son 4 a [ISO04]	Bare: 4 g	20 / box	12 v 09 mm	4.0 ml	15 25 ml /min	Bare: 0.04 - 0.4 g		
	Sindsep 4 g [13004]	Bonded: ≥ 5 g	2 / box	12 X 90 11111	4.9 IIIL	15 - 25 IIIL/IIIII	Bonded: 0.02 - 0.2 g		
	Silia San 12 a [ISO12]	Bare: 12 g	20 / box	21 v 117 mm	17 ml	20 - 40 ml /min	Bare: 0.12 - 1.2 g		
	Sindsep 12 9 [13012]	Bonded: \geq 15 g	1 / box	21 X 117 11111	17 1112	20 - 40 me/min	Bonded: 0.06 - 0.6 g		
_		Bare: 25 g	15 / box	21 v 165 mm	21 ml	20 45 ml /min	Bare: 0.25 - 2.5 g	225 pci / 16 bor	
ÅD	Sindsep 25 y [13025]	Bonded: ≥ 30 g	1 / box	21 X 105 11111	STIIL	20 - 43 ME/MIN	Bonded: 0.125 - 1.25 g	225 psi / 10 bai	
Ř		Bare: 40 g	15 / box	27 v 160 mm	47 ml	25 50 ml /min	Bare: 0.4 - 4 g		
2	SindSep 40 g [13040]	Bonded: ≥ 45 g	1 / box	27 X 109 mm	47 IIIL	23 - 30 me/mm	Bonded: 0.2 - 2 g		
Discover		Bare: 80 g	12 / box	21 v 227 mm	122 ml	40 - 80 ml /min	Bare: 0.8 - 8 g		
		Bonded: ≥ 90 g	1 / box	51 X 257 11111	123 1112	40 - 00 mE/mm	Bonded: 0.4 - 4 g		
	SiliaSep 120 g [IS120]	Bare: 120 g	10 / box	36 v 256 mm	190 ml	60 - 120 ml /min	Bare: 1.2 - 12 g	205 psi / 12 bar	
		Bonded: ≥ 130 g	1 / box	50 X 250 mm	190 IIIC	00 - 120 me/mm	Bonded: 0.6 - 6 g	203 0517 15 081	
	Silia San 220 a [IS220]	Bare: 220 g	4 / box	60 x 195 mm	306 ml	60 - 190 ml /min	Bare: 2.2 - 22 g		
	SindSep 220 g [13220]	Bonded: ≥ 230 g	1 / box	00 X 133 mm	500 IIIL	00 - 130 mL/mm	Bonded: 1.1 - 11 g	160 nsi / 11 har	
	Silia San 330 a [IS330]	Bare: 330 g	4 / box	60 x 268 mm	441 ml	80 - 190 ml /min	Bare: 3.3 - 33 g	100 p317 11 bai	
	Sindsep 330 g [13330]	Bonded: ≥ 360 g	1 / box	00 x 200 mm	441 1112	00 - 130 mL/mm	Bonded: 1.65 - 16.5 g		
	Silia Sen BT 75S [75iS]	Bare: 200 g	2 / box	75 x 90 mm	300 ml		Bare: 0.2 - 20 g		
		Bonded: 200 g	1 / box	75 × 50 mm	500 IIIL		Bonded: 0.1 - 10 g	90 nsi / 6 5 har	
ess	SiliaSen BT 75M [75iM]	Bare: 400 g	2 / box	75 x 170 mm	500 ml	100 - 250 ml /min	Bare: 0.4 - 40 g	(inside the	
Ö		Bonded: 400 g	1 / box	75 X 170 mm	500 IIIE	100 200 me/mm	Bonded: 0.2 - 20 g	compression module)	
Р.	Silia Sen BT 751 [75il]	Bare: 800 g	2 / box	75 x 350 mm	11		Bare: 0.8 - 80 g	moduley	
nt &		Bonded: 800 g	1 / box	13 X 000 mm			Bonded: 0.4 - 40 g		
neı	SiliaSen XI 800 a** [IS750]	Bare: 800 g	2 / box	78 x 382 mm	151	200 - 300 ml /min	Bare: 8 - 80 g	125 nsi / 8 har	
Ido		Bonded: ≥ 870 g	1 / box	10 x 302 mm	1.5 L	200 000 me/mm	Bonded: 4 - 40 g	125 her o ngi	
vel	SiliaSen XI 1 600 a* [11500]	Bare: 1,600 g	2 / box	104 x 429 mm	291	300 - 450 ml /min	Bare: 16 - 160 g	100 nsi / 7 har	
De		Bonded: ≥ 1,700 g	1 / box	104 / 420 mm	2.5 L		Bonded: 8 - 80 g	100 00177 000	
	SiliaSep XL 3,000*** [I3000]	Contact us for more information: info@silicycle.com							

* Cartridge length includes luer-lock and connection tip.

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



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 v	s 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 Δ CV is a measure of two analytes separation and resolution: Δ CV = CV₂ - CV₁ = (1 / Rf₂) - (1 / Rf₁)

V	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.



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



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.



Cartridge Size





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 SiliaSep flash cartridges. For easily separated compounds ($\Delta CV > 6$) we suggest to load up to 5 % on irregular bonded phases, up to 10 % on bare irregular silica and up to 15 % on bare spherical silica.

	Low Pressure Chroma							ırt				
Dimensions ID x Length	SiliaSep Format	Silia <i>Sep</i> Phase	Diffic ΔCV = 0.1 - 0.6	cult Separa ΔCV = 0.7 - 1.2	ation ΔCV = 1.3 - 1.8	Medi ΔCV = 1.9 - 2.4	Loa um Separ ΔCV = 2.5 - 3.1	d (g) ation ΔCV = 3.2 - 3.8	ΔCV = 3.9 - 4.5	Easy Se ΔCV = 4.6 - 5.2	paration ΔCV = 5.3 - 6.0	ΔCV > 6
		Irregular Silica	0.040	0.080	0.120	0.160	0.200	0.240	0.280	0.320	0.360	0.400
12 x 98 mm	4 a	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
		Irregular Silica	0.120	0.240	0.360	0.480	0.600	0.720	0.840	0.960	1.080	1.200
21 x 117 mm	12 g	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
		Irregular Silica	0.250	0.500	0.750	1.000	1.250	1.500	1.750	2.000	2.250	2.500
21 x 165 mm	25 g	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
		Irregular Silica	0.400	0.800	1.200	1.600	2.000	2.400	2.800	3.200	3.600	4.000
27 x 169 mm	40 g	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
	80 g	Irregular Silica	0.800	1.600	2.400	3.200	4.000	4.800	5.600	6.400	7.200	8.000
31 x 237 mm		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
	120 g	Irregular Silica	1.200	2.400	3.600	4.800	6.000	7.200	8.400	9.600	10.800	12.000
36 x 256 mm		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
		Irregular Silica	2.200	4.400	6.600	8.800	11.000	13.200	15.400	17.600	19.800	22.000
60 x 195 mm	220 g	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
		Irregular Silica	3.300	6.600	9.900	13.200	16.500	19.800	23.100	26.400	29.700	33.000
60 x 268 mm	330 g	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
		Irregular Silica	8.000	16.000	24.000	32.000	40.000	48.000	56.000	64.000	72.000	80.000
78 x 382 mm	800 g	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
		Irregular Silica	16.000	32.000	48.000	64.000	80.000	96.000	112.000	128.000	144.000	160.000
104 x 429 mm	1,600 g	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
			Diffic	cult Separa	ation	Medi	um Separ	ation		Easy Se	paration	

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 Δ CVs measured in TLC.



SiliaSep PREMIUM - Spherical Silica Flash Cartridges



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

SiliaSep 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 SiliaSep 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



SiliaSep PREMIUM C18 25g



SiliaSep PREMIUM against the Competition

We compared the results on our SiliaSep PREMIUM against two established players in chromatography and purification. This study shows that SiliaSep 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







SiliaSep 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 <u>www.fda.gov/ohrms/dockets/dailys/04/oct04/101904/03n-0059-c00004-01-vol1.pdf</u>, 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
	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.
Bare Silica	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.
	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.*
C18	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 (<i>typically methanol, ethanol or acetonitrile</i>) 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.



SiliaSep Cartridges Ordering Information

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

SiliaSep Phases

SiliaSep 40 - 63 µm Irregular Silica

	SiliaSep P		
Ph	ases	Phase PN	
	Bare Irregular Silica	R10030B	A C
	C18	R33230B	
LICA	C8	R30830B	
JLAR SIL	Phenyl	R34030B	
	PFP	R67530B	
RG	Amine	R52030B	
O IRF	Diol nec	R35030B	YAN
-IZEI	Cyano	R38030B	4/2
NAL	SCX	R60530B	X
CTIC	SCX-2	R51230B	
FUN	SAX nec	R66530B	
_	SAX-2 nec	R66430B	

	SiliaSep P	hases	
Ph	ases	Phase PN	
	Bare Spherical Silica	10095D-A	
	C18	03295D-A	2
LICA	C8	30895D-A	
- SI	Phenyl	34095D-A	
ICAL	PFP	67595D-A	
НШК	Amine	52095D-A	
O SP	Diol nec	35095D-A	
IZEI.	Cyano	38095D-A	-
CTIONAL	SCX	60595D-A	
	SCX-2	51295D-A	
NU N	SAX nec	66595D-A	
	SAX-2 nec	66495D-A	



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

SiliaSep Formats

	SiliaS		
Formata	Qty/	Format Code	
Formats	Bare Silica	Functionalized Silica	Format Code
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	11500
XL 3,000	1	1	13000



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

Notes:

• For bigger columns, please contact us.

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



SiliaSep PREMIUM 25 µm Spherical Silica

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. Silia*Sep* solid-load luer-lock cartridges are designed to be used with Silia*Sep* 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).

		SiliaSep S	olid-Load Cartridges	
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!



O-ring compression plate, solvent resistent



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	SiliaSep OT Formats					
Phases	Phase PN	Formats	Oty/Box	Dimensions	Volume	Format Code	
Bare Silica	R10030B	T Official	QUIDOX	(ID x length)	Volume	Bare Silica	Functionalized Silica
C18	P33230B	- 2 g	20	15.8 x 90 mm	12 mL	15U	15U
C10	D20020D	- 5 g	20	20.5 x 100 mm	25 mL	25X	15U
	R30630B	- 10 g	16	26.8 x 154 mm	70 mL		70Y
Phenyl	R34030B	- 15 g	16	26.8 x 154 mm	70 mL		70i
PFP	R67530B	- <u>20 a</u>	16	26.8 x 154 mm	70 ml		707
Amine	R52030B	- 25 g	10	20.0 × 134 mm	150 ml) CL
Diol nec	R35030B	25 y	10	38.2 X 170 mm	150 ML		70K
Cyano	R38030B	50 g	10	38.2 x 170 mm	150 mL		95M
SCX	R60530B	70 g	10	38.2 x 170 mm	150 mL	9	95N
SCX-2	R51230B	100 g	12	40.0 x 220 mm	276 mL	2	76F

Notes:

SAX nec

SAX-2 nec

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

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

R66530B R66430B

Maximum operating pressure: 60 psi. ٠





SiliaSep 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 <i>Sep</i> BT 75S	75iS	200 g	75 x 90 mm	300 mL		Bare: 0.2 - 20 g Bonded: 0.1 - 10 g	
Silia <mark>Sep</mark> BT 75M	75iM	400 g	75 x 170 mm	500 mL	100 - 250 mL/min	Bare: 0.4 - 40 g Bonded: 0.2 - 20 g	90 psi / 6.5 bar (inside compression module)
Silia <i>Sep</i> 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.

	SiliaSep BT Phases					
Pha	ises	Phase PN				
	Bare Irregular Silica	R10030B				
	C18	R33230B				
	C8	R30830B				
ar Silica	Phenyl	R34030B				
	PFP	R67530B				
egul	Amine	R52030B				
nl be	Diol nec	R35030B				
aliz(Cyano	R38030B				
Iction	SCX	R60530B				
Fur	SCX-2	R51230B				
	SAX nec	R66530B				
	SAX-2 nec	R66430B				

	SiliaSep BT Formats					
Formats	Silica Weight	Qty/	Format Code			
Formats	Shica Weight	Bare silica	Functionalized Silica	Format Coue		
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.







Thin Layer Chromatography (TLC)

- Wide choice of sizes, sorbents & thicknesses available
- Excellent reproducibility between SiliaPlate 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.

SiliaPlate 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.

SiliaPlate 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 [<i>10 - 14 μm</i>]	4 - 8 μm [5 - <i>6 μm</i>]	5 - 40 μm [<i>22 - 25 μm</i>]			
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

SiliaPlate TLC plates are mostly offered with an $\rm F_{_{254}}$ UV indicator.

Some plates may be available with a F₃₆₆ UV indicator, or without UV indicator. Contact us: info@silicycle.com.



Sorbents

Available Sorbents				
Classical Silica Gel	Reversed-Phases Special Phases			
A universal matrix for daily, fast, reliable analysis of the largest spectra of molecules The particle size distribution used for the silica is related to the nature of the plate. For standard TLC, silica gel with a mean particle size of 10 - 14 μm is used compared to HPTLC, where a smaller particle size is required	 The two most popular modes of separation employed in TLC are normal and reversed-phases. In normal phase separation, the mobile phase is less polar than the stationary phase. Inversely, in reversed mode, the mobile phase (<i>usually a mixture of water and organic solvent</i>) is more polar than the stationary phase (<i>C18</i>). 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 (<i>CN</i>) 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	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 (<i>commonly called Alumina</i>) is the second most commonly used matrix, and shows similar selectivity to that of silica, with 3 different pH ranges (<i>basic, neutral, acidic</i>). 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
SiliaPlate TLC plates are available in the following standard sizes depending on the coating used: 20 x 20 cm 10 x 20 cm 5 x 20 cm 5 x 20 cm 10 x 10 cm Example:	Also for your convenience, SiliCycle provides ready-to-use micro TLC plates in the following formats: 2.5 x 10 cm 2.5 x 7.5 cm 2.5 x 5 cm Example:	An interesting compromise between standard and micro plate sizes is our Scored Silia <i>Plate (glass backing)</i> . Three different formats are available and possible cut combinations are shown in the image below. 20 x 20 cm plates scored to four 5 x 20 cm plates (or multiple of 5 cm width) 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 plates scored to eight 2.5 x 5 cm plates (or multiple of 2.5 cm width) 5 x 20 cm plates scored to eight 2.5 x 5 cm plates (or multiple of 2.5 cm width)



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.

• **<u>BK</u>'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 KMnO₄, 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	В	ВК		
Example	TLG-R10014 <mark>B</mark> -323	TLG-R10014 <mark>BK</mark> -323		
UV Fluorescence (F_{254})	Higher brightness Less background noise from layer	Yes		
	Stable in almost a	Il organic solvents		
Binder Sensitivity	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	≈ 25	0 μm		
Stain Compatibility				
KMnO ₄	Compatible	Highly compatible		
САМ	Compatible			
p-Anisaldehyde	Compatible	Highly compatible		
Ninhydrin	Highly compatible			
Vanilin	Highly compatible			



SiliaPlate 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: <u>info@silicycle.com</u>.

Various combinations are possible with SiliaPlate TLC plates and are summarized in the table below.

SiliaPlate 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		



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 & ot	her 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	



Glass-backed HPTLC Plates

Glass-backed Analytical HPTLC Plates					
PN	Sorbent	Plate Size	Thickness	UV indicator	Qty/box
Silica	A-		î	Ť	1
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 KMnO4 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 & ot	her adsorbents		A.		
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



Glass-backed Preparative TLC Plates

	Glass-backed Preparative TLC Plates					
PN	Sorbent	Plate Size	Thickness	UV indicator	Qty/box	
Silica						
TLG-R10011B-333	Silica	20 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 p	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	

SiliaPlate TLC	Glass Backed TL	C Extra Hard Layer, 6	wi _5	-
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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 & ot	her 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	



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Plastic-backed TLC Plates

Plastic-backed Analytical TLC Plates					
PN	Sorbent	Plate Size	Thickness	UV indicator	Qty/box
Silica					I
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



TLC Accessories

TLC Accessories				
PN	Accessory	Qty/box		
AUT-0161	Rectangular TLC Developing Chamber	1		
AUT-0161B	Replacement Lid for Rectangular Developing Chamber	1		
AUT-0162	TLC Adsorbent Scraper	1		
AUT-0163	TLC Spotting Capillaries	300		
AUT-0182	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		





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 (*Et₃N*) to the solvent mixture (0.1 2.0 % but typical quantity is 0.1 %) or 1 10 % ammonia (*NH₃*) 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.





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 micro pipette*). In both cases, a spotting guide can be used to facilitate sample application.

Co-spotting

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







TLC Plate Development

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

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

TLC Plate Visualization

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

Non-destructive methods

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

Destructive methods

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

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

Prediction of Column Volumes (CV)

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

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

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

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





TLC Visualization Methods

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

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 a	lkenes, alkynes and aromatic compo	unds unless other functional groups are present.	Γ	
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 ap	pear 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 CoCl₂ 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 ColorsSpots: BlueBG: White	



Stains for Thin Layer Chromatography				
Name	Visualization of	Stain Recipe	Comments	
2,4-Dinitrophenyl-hydrazine	Aldehydes	Prepare stain as follows	Visualization Colors	
(DNP)	Ketones	12 g of 2,4-dinitrophenylhydrazine	Spots: Yellow to red	
		60 mL of conc. sulfuric acid	BG: Light orange	
		80 mL of water	DO NOT HEAT dipped plate	
		• 200 mL of 95 % ethanol		
Dragendorff Reagent	Nitrogenous Compounds	Prepare stain as follows:	Visualization Colors	
	Alkaloids, amines,	Solution A	Spots: Orange to red	
	organics bases, etc. Phenols	• 1.7 g of bismuth nitrate	BG: Yellow	
		80 mL of water	Appropriate Storage	
		20 mL of acetic acid	Aluminum wrapped	
		Solution B	Stain Shelf-Life	
		40 g of potassium iodide	One or two weeks	
		• 100 mL of water	Solutions A and B are long term storable	
		Tip: mix 5 mL of each solution A and B to a solution of 20 mL of acetic acid in 70 mL of water.	DO NOT HEAT dipped plate	
Ferric Chloride	Phenols	Prepare stain as follows	Visualization Colors	
(FeCl ₃)		2 g of ferric chloride	Spots: Red	
		• 102 mL of 0.5 N hydrochloric acid	BG: Yellow	
lodine	Unsaturated & Aromatic	Prepare stain as follows	Visualization Colors	
	compounds	Iodine crystals in an amber bottle	Spots: Dark brown	
			BG: Light brown	
Note: lodine stain can be remove	ed by heating.		•	
Morin Hydrate	Universal stain	Prepare stain as follows	Visualization Colors	
(Hydroxy Flavone)	Fluorescently active	0.1 % of morin hydrate in methanol	Spots: Various colors	

Morini Hydrate	Oniversal stain	Frepare stain as follows	Visualization Colors
(Hydroxy Flavone)	Fluorescently active	0.1 % of morin hydrate in methanol	Spots: Various colors
			BG: White
Ninhydrin	Amino acids	Prepare stain as follows	Visualization Colors
(Indanetrione Hydrate)	Amino sugars	• 1.5 g of ninhydrin	Spots: Various colors
	Amines	3 mL acetic acid	BG: White
		• 100 mL of n-butanol	
Phosphomolybdic Acid	Universal stain	Prepare stain as follows	Visualization Colors
(PMA)	Very effective against	• 10 % of PMA solution in ethanol	Spots: Dark green to black
	diluted sample	• or 10 g of PMA in 100 mL of ethanol	BG: Light green
Potassium Permanganate	Olefins	Prepare stain as follows	Visualization Colors
(KMnO₄)	Readily oxidized groups	• 1.5 g of potassium permanganate	Spots: Yellow to light brown
	Alcohols, aldehydes,	• 10 g of potassium carbonate	BG: Purple to pink
	alkenes, alkynes, etc.	• 1.25 mL of 10 % sodium hydroxide	Stain Shelf-Life
		200 mL of water	Three months
Note: Can be used for detection	of alcohols, amines, sulfides and me	rcaptans groups when gently heated.	•

Vanillin	Universal stain	Prepare stain as follows	Visualization Colors
	Very effective for same	• 15 g of vanillin	Spots: Various colors
	polarity products (Rf)	• 250 mL of 95 % ethanol	• BG: Light tan
		• 2.5 mL of conc. sulfuric acid	

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.



SiliaPlate 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 UVactive.
- 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.







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