SOLUTIONS FOR SCAVENGING OF METAL AND ORGANIC IMPURITIES









SiliCycle Scavenging Solutions

- Broadest portfolio of scavengers with associated applications
- Great variety of formats for all purifications scales: from laboratory to plant scale
- Excellent scavenging efficiency for a wide range of metal and organic impurities
- Green and environmentally friendly technology
- Compatible with various experimental conditions, solvents, pH, and temperatures

SiliaMetS and SiliaBond Silica-Based Scavengers

Chemists have been searching for techniques and tools to separate, isolate and purify chemical substances from one another to improve the quality of the synthesized molecules. SiliCycle scavenging technologies enable powerful purification processes to help reach new purity standards.

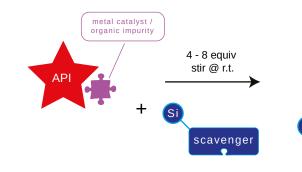
Silica-based scavengers have been proven to be the most effective method for removing metal or organic impurities without contaminating drug candidates. A silica matrix offers many advantages over polymers:

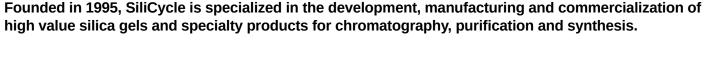
- Reduced purification steps
- No swelling
- · More general solvent compatibility



What makes Silia*MetS* metal scavengers and Silia*Bond* organic scavengers so easy to use is their heterogeneous nature. When used in bulk in a contaminated solution, all you need to do is filter off the scavenger with the bound impurity and dispose of it.

When used in SPE or flash cartridges for batch reactions, the filtration process - with adapted experimental conditions - will leave the contaminant in the cartridge, rendering the solution free of impurities.





Scavenging Solutions

Scavenging Solutions Overview	3
Different Formats for Different Applications	4
Quality and Regulatory Documentation, Stability and Leaching Studies and Manufacturing Capability	8
Metal and Organic Scavenging Screening Services	9
Silia <i>MetS®</i> Metal Scavengers Portfolio	. 10
Silia <i>MetS[®]</i> Metal Scavengers Selection Table	. 12
Silia <mark>Bond®</mark> Organic Scavengers Portfolio	. 14
SiliaBond® Organic Scavengers and Potential Genotoxic Impurities (PGI) Selection Tables	. 16
Typical Experimental Procedures	. 18
Experimental Procedure Optimization in Bulk	. 20
Experimental Procedure Optimization in E-PAK	. 23
Ordering Information	. 24
Resource Center	. 27

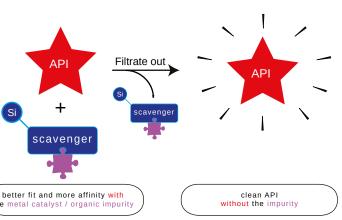


- · Higher mechanical and thermal stabilities
- · Easily scalable applications
- · Availability in different product formats, such as bulk, E-PAK flow cartridges, SPE or flash cartridges, etc.

Functional Group

Typical structure of our functionalized silicas with various organic groups

Linker





Analysis

R&D Services



Different Formats for Different Applications

Please refer to our Ordering Information Section to learn more about all formats available and the corresponding part numbers.

Scavengers as Bulk Silica

All our scavengers can be used in bulk directly in your reaction flask or reactor and are available from 5 g to 25 kg formats, up to multi-ton scale.

- All our scavengers have, by default, the same silica backbone: our SiliaFlash R10030B.
 - Particle Size: 40 63 μm
 - Pore Size: 60 Å
- All our Silia Flash silica gels of various particle sizes and pore sizes are available as silica backbones upon request.

Scavengers in SiliaPrep SPE and SiliaSep OT Cartridges

All our scavengers are available in pre-packed cartridges.

- SiliaPrep SPE cartridges From 3 mL / 200 mg to 12 mL / 2 g
- SiliaSep OT (Open Top) flash cartridges From 25 mL / 5 g to 150 mL / 70 g

Scavengers in SiliaSep Flash Cartridges

All our scavengers are available pre-packed in flash cartridges.

 SiliaSep flash cartridges From 4 g to 1.6 kg bed weight

Packings can also be tailored to your available equipment and scales.



Scavengers in SiliaChrom Guard Cartridges

SiliaChrom HPLC Guard Cartridges are designed to effectively protect both analytical and preparative HPLC columns. The usage of this shorter column is highly recommended to prolong column lifetime and does not alter chromatography. All metals can be prejudicial and very damaging to your column and detector, complicating purification steps, often making them longer, more laborious and less effective.

Crude reaction mixtures can now directly be injected without further metal removal, which will save precious time for the chemist. Another great benefit is that there is much less risk of corroding the equipment by injecting dirty samples.



SiliaChrom Guard Cartridges are available in lengths of 10 - 20 mm and three internal diameters (ID: 4.0, 10 and 21.2 mm). The Guard Column internal diameter should be the same as the HPLC column or one size smaller. Never use a guard column with a larger ID than the HPLC column (risk of efficiency loss).

SiliaChron	o Guard Cartridges
Guard Cartridge Name	Favorite Metal Guard Cartridge
Scavenger Packing #	K346
Effective Scavenger for:	Pd, Ag, Hg, Ir, Ni, Os, Pt, Rh, Ru
Can also Remove:	Cd, Co, Cu, Fe, Pb, Sc, Sn, Zn



Scavengers in E-PAK Flow Cartridges

E-PAK is a family of radial flow adsorption cartridges developed specifically for pharmaceutical processings.

Created with proprietary technology, E-PAK cartridges provide rapid adsorption kinetics at flow rates and processing capacities suitable for laboratory, pilot and commercial operations. They are designed for use with both organic and aqueous solvents, and incorporate design features useful for the production of active pharmaceutical ingredients (API).

Features and Benefits

- Proven cartridge design ensures rapid, simple and reliable technology
- High adsorption capacity and flow rate
- Fixed-bed design ensuring safer handling, clean-up and disposal
- · Large adsorbent capacity in small area footprint increases product recovery and reduces solvent requirements

Sorbents for E-PAK Cartridges

E-PAK cartridges are available in a range of sorbents to accommodate the broad range of processing requirements. Other adsorbents are available under request.

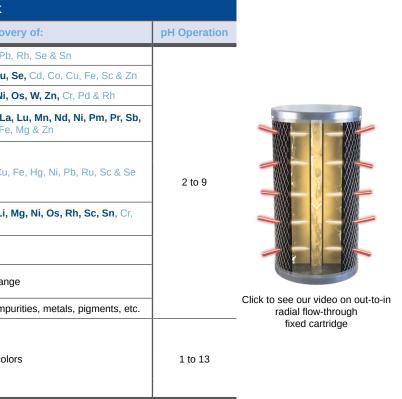
	Sorbents for E-PAK				
Active Adsorbents (typical loading)	For Removal and/or Recov				
SiliaMetS Thiol (1.2 mmol/g)	Pd, Ag, Hg, Os, Ru, Cu, Ir, Pl				
SiliaMetS DMT (0.5 mmol/g)	Pd, As, Ir, Ni, Os, Pt, Rh, Ru				
SiliaMetS Imidazole (0.96 mmol/g)	Cd, Co, Cu, Fe, Ir, Li, Mg, Ni				
SiliaMetS AMPA (0.8 mmol/g)	Al, Ce, Dy, Er, Eu, Gd, Ho, La Sn, Tb, Tm, V, Yb, Co, Cu, Fe				
SiliaBond Amine (1.2 mmol/g)					
SiliaMetS Diamine (1.28 mmol/g)	Pd, Cr, Pt, W, Zn, Cd, Co, Cu				
SiliaMetS Triamine (1.11 mmol/g)					
SiliaMetS TAAcONa (0.41 mmol/g)	Ca, Cd, Cs, Cu, Fe, Ir, La, Li Pd, Ru, Se & Zn				
SiliaBond Cyano (1.38 mmol/g) and Florisil	Various organic molecucles				
SiliaBond Propylsulfonic Acid SCX-2 (0.63 mmol/g)	Amines & anilines, ion exchar				
Silia <i>Flash</i> Bare Silica	Very vast range of organic im				
Activated Carbon SiliaCarb CA					
Activated Carbon SiliaCarb HA					
Activated Carbon SiliaCarb VA	Precious metal catalysts & co				
Activated Carbon SiliaCarb VW	<u> </u>				

Best scavenger for the removal of a particular metal is indicated in Navy Blue while good scavenger is indicated in Pale Blue.

E-PAK cartridges are manufactured using a proprietary technology and chemically stable materials in most common organic solvents. They have been tested and found satisfactory for use with the following commonly used solvents:

- 2-Butanone
- Dichloromethane
- Ethanol
- Ethyl acetate
- Heptane





- Methanol
- MTBE
- N-ethyl-2-pyrolidone
- Tetrahydrofuran (at room temperature)
- Toluene (at room temperature)



Scavenging



Various Ways of Using E-PAK

Depending on the application and how you prefer to work, E-PAK cartridges can be used in different ways as shown below on all housing scales (lab, pilot and commercial scale). Click on the picture to access the video on E-PAK methods.

For a single pass usage, we suggest to run at very low flow rate compared to recirculation process, which can be run at higher flow rate.



SiliaMetS E-PAK Portfolio

Lab Scale

Lab scale cartridges are designed to facilitate small samples evaluation. Testing with loose media can be done with samples as small as a few milliliters and is normally done before cartridge testing to identify the formula with the highest capacity to remove contaminants with the highest recovery.

\mathcal{L}		Lab Scale Cartridges		
Cartridge Size	Typical	Pressure Drop	Media We	ight <i>(g)</i>
Diameter × Height (cm)	Flow Rate Range (mL/min)	with w/1 cP Fluid (psig)	Silia <i>Flash</i> , Silia MetS and Silia <mark>Bond</mark>	SiliaCarb
5×1	1 - 20	≤ 5	8	5
5 × 10	10 - 200	≤ 5	75	50
5 × 25	25 - 500	≤ 5	200	125

Note: Faster flow rates can be used for the lab scale cartridges depending on the application or the scavenging difficulty (1 cm up to 100 mL/min, 10 cm up to 500 mL/min and 25 cm up to 1 L/min).

Pilot and Commercial Scale

E-PAK pilot scale cartridges provide rapid processing for volumes from 10 to hundreds of liters, and can establish the parameters upon moving to larger scales, since E-PAK achieve true linear scalability.

E-PAK commercial scale cartridges provide rapid processing for manufacturing operations needing to process batch sizes of > 10,000 liters or can be adapted for continuous operation using a duplex design.

Ya.	Pilot and Commercial Scale Cartridges										
Cartridge Size Diameter × Height (cm)	Typical Flow Rate Range (<i>L/min</i>)	Pressure Drop with w/1 cps Fluid (psig)	Media Wei Silia <i>Flash</i> , Silia <i>MetS</i> and Silia <i>Bond</i>	ght (<i>kg</i>) SiliaCarb							
Pilot Scale	î.										
16.5 × 12.5	0.10 - 2.5	≤ 10	0.87	0.55							
16.5 × 25	0.25 - 5	≤ 10	1.75	1.10							
Commercial Scale											
16.5 × 50	0.50 - 10	≤ 10	3.50	2.10							
16.5 × 100	1 - 20	≤ 10	7.00	4.10							

Both pilot and commercial cartridges are provided with a Code 8 (closed top and open bottom end caps-bottom with double 2-222 Teflon® encapsulated Viton® o-ring) cartridge sealing configuration. To meet commercial processing requirements, E-PAK cartridges can be operated in parallel for increased capacity.

6

Housings

Lab Scale

- Various housing length available (for 1 cm, 10 cm, and 25 cm cartridges)
- Made in stainless steel 316L or Hastelloy C276
- Pressure rating for housing 150 psi (10 bar)
- Easy housing conversion for all lengths by changing the bowl
- · Operated with standard pump, low pressure and peristatic

Pilot and Commercial Scale

- · Various housings available for simultaneous operation of 1 to 12 cartridges (for 12.5 cm, 25 cm, 50 cm and 100 cm)
- Made in stainless steel 316L or Hastelloy C276
- Pressure rating for housing 150 psi (10 bar)
- Can be operated in parallel to process batch sizes of ≥ 1,000 L
- ASME and PED 2014/68/EU, CE compliant

Scale-Up Calculation

Although there are always exceptions, scale-up projections based on a linear extrapolation of adsorbent mass have proven to be quite accurate when test conditions including contact time, temperature, solvent type and contaminant and compound levels are held constant. The following table shows the scale-up/relative change in mass between lab, pilot and commercial size E-PAK cartridges available with activated carbons and scavengers.

	Scale-Up Calculation for Various Adsorbents												
Cartridge Sizes (cm)	5 x 1	5 x 10	5 x 25	16.5 x 12.5	16.5 x 25	16.5 x 50	16.5 x 100						
Typical Scale-up Factor	-	10	25	110	220	440	875						
Mass of Activated Carbon (g)	5	50	125	550	1,100	2,100	4,100						
Mass of Silica (g)	8	75	200	875	1,750	3,500	7,000						
# mmol SiliaMetS AMPA (0.8 mmol/g)	6.4	60	160	700	1,400	2,800	3,600						
# mmol SiliaMetS Diamine (1.28 mmol/g)	10.2	96	256	1,120	2,240	4,480	8,960						
# mmol SiliaMetS DMT (0.50 mmol/g)	4.0	38	100	438	875	1,750	3,500						
# mmol SiliaMetS Imidazole (0.96 mmol/g)	7.7	72	192	840	1,680	3,360	6,720						
# mmol SiliaMetS TAAcONa (0.41 mmol/g)	3.3	31	82	359	717	1,435	2,870						
# mmol SiliaMetS Triamine (1.11 mmol/g)	8.9	83	222	971	1,942	3,885	7,770						
# mmol SiliaMetS Thiol (1.20 mmol/g)	9.6	90	240	1,050	2,100	4,200	8,400						
# mmol SiliaBond Amine (1.20 mmol/g)	9.6	90	240	1,050	2,100	4,200	8,400						
# mmol SiliaBond Cyano (1.38 mmol/g)	11.0	103	276	1,207	2,415	4,830	9,660						
# mmol SiliaBond SCX-2 (0.63 mmol/g)	5.0	47	126	551	1,102	2,205	4,410						
Bed Volume (<i>cm</i> ³)	18.8	188	470	2,375	4,750	9,500	19,000						
Recommended Flow Rate (mL/min)	7.5	75	190	950	1,900	3,800	7,600						
Typical Flow Rate Range (mL/min)	1 - 20	10 - 200	25 - 500	100 - 2,500	250 - 5,000	500 - 10,000	1,000 - 20,000						
Approximated Tank Volume (mL)	50	200	450	2,500	5,000	11,600	23,300						
Minimum System Flushing (mL)	150	600	1.35	7,500	15,000	35,000	70,000						

Measurement Methodology:

- Scale-up Factor: mass of silica / 8 g (smallest size)
- Mass of Activated Carbon (g) / Mass of Silica (g): amount of SiliaCarb / SiliaMetS / SiliaBond in the cartridge
- # mmol SiliaMetS / SiliaBond XXX (X.X mmol/g): silica mass x typical loading of SiliaMetS / SiliaBond
- Bed Volume (cm³): total volume of the cartridge without the volume of the hole
- Recommended Flow Rate (mL/min): for residence times of 2.5 minutes
- Typical Flow Rate Range (mL/min): for residence times from 1 to 20 minutes. Faster flow rate can be used for some applications.
- needed for conditioning can vary depending on experimental conditions. It is recommended to do a minimum of 3 system flushing before use.













Click to watch our video: from lab to commercial scale E-PAK housings

Minimum System Flushing (mL): corresponds to 3 tank volumes measured experimentally with the cartridges inside the housing. The volume of solvents



Quality and Regulatory Documentation

The SiliCycle scavengers have been used for decades in pilot plants and production units under GMP conditions by the pharmaceutical industry as well as CMOs and CROs. They have ran their own analysis proving that silica-based scavengers can safely be used, both in reactors and cartridges to purify their compounds.

SiliCycle is committed to high quality standards and all products are manufactured in an ISO 9001:2015 compliant facility and subjected to stringent quality control.

For any inquiries, please contact: scavenger@silicycle.com

All products are shipped with the following information:

- · Certificate of Analysis
- Safety Data Sheet (SDS)
- · Technical information

- Other statements available under request
- BSE / TSE declaration (non animal-derived)
- GMO-Free Certificate

· Melamine-Free Certificate, etc.



Our Regulatory Support Files (RSF) are documents that include both proprietary and non-proprietary information on performance, chemical / thermal / mechanical stability, extractable and leachable compounds, SOPs, scale-up procedures, batch history, analytical methods and more. RSF documentation can be obtained through a Non-Disclosure Agreement (NDA).

SiliCycle can also work with you to provide customized regulatory documents, including specific analytical tests in line with your needs.

Stability and Leaching Studies

Since our metal and organic scavengers are being used by many pharmaceutical companies, each SiliaMetS and SiliaBond manufactured by SiliCycle is submitted to an extensive washing procedure to ensure the product exhibits extremely low levels of extractables and leachables.

Manufacturing Capability

SiliaMetS and SiliaBond are manufactured at our headquarter in Quebec City that can answer production needs.

Our state of the art facilities include (but are not limited to):

- 1,000 L to 10,000 L reactors (total capacity of 38,000 L)
- Stainless steel and hastelloy nutsche filters (3 m²)
- Bulk solvent tank farm (60,000 L capacity)





Metal and Organic Scavenging Screening Services

Scavenging screening services provide solutions to guickly develop the most efficient metal scavenging process providing both time and cost savings. Confidentiality is assured and we will be working with you, in function of your needs.

Screening and Optimization Services

Batch: Lab and Process Development

Step 1: Screening

- Different SiliaCarb activated carbons and SiliaMetS metal scavengers will be tested on the crude product at two different temperatures.
- · Influence of contact time will also be evaluated.

Step 2: Optimization

- · After this initial screening, we will select the most efficient carbon and scavenger to optimize the ratio vs the API.
- Pairwise combinations of metal scavengers could also be tested, if satisfactory results are not obtained. Scavenging yields as well as recoveries will be evaluated.

Step 3: Scale-up Confirmation and Optimization

- A new amount of crude product will then be treated with SiliaCarb (if relevant), using the best obtained conditions to get enough product to work on the next step.
- · Finally, we will test the best conditions on the API solubilized in two other solvent systems (depending on API solubility and customer's requirement). Scavenging yields as well as recoveries will be evaluated.
- Best conditions will be transposed on larger scale (to be determined based on the remaining crude product).

Includes	BATCH Scavenger Screning	BATCH TO E-PAK Scavenger Screening
SiliaCarb and SiliaMetS screening in bulk		\checkmark
Optimization in different solvents		\checkmark
Metal concentration tracked by ICP-MS or ICP-OES		\checkmark
Scale-up confirmation and optimization*		 Image: A start of the start of
Transfer of the optimized conditions to E-PAK radial flow cartridge		\checkmark
Evaluation of contact time in E-PAK radial flow cartridge		>
Scalability based on customer final equipment can be performed if enough product is available		 Image: Second sec
Complete report provided		\checkmark
Expected screening service delay after reception of customer's sample Typical crude quantity needed*	3 - 4 weeks 50 to 200 g of crude	4 - 6 weeks 100 to 200 g of crude





SiliCycle's facility

Chromatography

Analysis

R&D Services

Batch to E-PAK: Including E-PAK Process Development

Includes all points performed with "batch" service plus the following:

- SiliaCarb and SiliaMetS best conditions will be then transfered to E-PAK radial flow cartridges.
- Contact time's effect can be evaluated as well as parameter's optimization on E-PAK for both SiliaCarb and SiliaMetS reaction.
- Scalability study based on customer final equipment can be performed if enough crude is available.

→ Main objective is to accelerate research, optimize production costs, while remaining fully in compliance with new regulations and environmental challenges

➔ Scavenging strategies imply lower volumes of solvent, less person-days and higher yields by minimizing loss of product via classical purification processes



SiliaMetS Metal Scavengers Portfolio

	\black		SiliaMetS Metal Scavengers Technical Inf	formation	
	Scavengers	Structure	Brief Description	Metals Removed ¹	Typical Characteristics ^{2, 3}
pgl-S	SiliaMetS Thiol PN: R51030B Loading: ≥ 1.20 mmol/g Endcapping: Yes	Э	Silia <i>MetS</i> Thiol is our most versatile and robust metal scavenger for a variety of metals under a wide range of conditions.	Ag, Au, Hg, Os, Pd & Ru Cu, Ir, Pb, Rh & Sn	Color: White Density: 0.682 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years
PGI-S	SiliaMetS DMT PN: R79030B Loading: ≥ 0.50 mmol/g Endcapping: Yes	SI SI SI SI SI SI SI SI SI SI SI SI SI S	Silia <i>MetS</i> DMT is the silica-bound equivalent of 2,4,6-trimercaptotriazine (<i>trithiocyanuric acid</i> , <i>TMT</i>). It is a versatile metal scavenger for a variety of metals and the preferred metal scavenger for ruthenium catalysts and hindered Pd complexes (<i>i.e.</i> $Pd(dppf)Cl_2$).	Au, Ir, Ni, Os, Pd, Pt, Re, Rh & Ru Cd, Co, Cu, Fe, Sc & Zn	Color: Light brown Density: 0.732 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years
	SiliaBond Amine PN: R52030B Loading: ≥ 1.20 mmol/g Endcapping: Yes	G NH2		Cd, Cr, Pd, Pt, Rh, Re & Ru Co, Cu, Fe, Hg, Pb, W & Zn	Color: Off-white Density: 0.700 g/mL Solvent Compatibility: 2 Prolonged Storage: 2 Shelf Life: 2 Years
	SiliaMetS Diamine PN: R49030B Loading: ≥ 1.28 mmol/g Endcapping: Yes		Also known for their electrophile scavenging efficiencies and their base reagent qualities, Silia <i>MetS</i> Amine, Diamine and Triamine have also proven to be very useful for the scavenging of the following metals: Pd, Pt, Cr, W and Zn.	Cr, Pd, Pt, W & Zn Cd, Co, Cu, Fe, Hg, Ni, Pb, Ru, V & Sc	Color: Off-white Density: 0.728 g/mL Solvent Compatibility: 2 Prolonged Storage: 2 Shelf Life: 2 Years
pgl-5	SiliaMetS Triamine PN: R48030B Loading: ≥ 1.11 mmol/g Endcapping: Yes	() 		Cr, Pd, Pt, W & Zn Ag, Cd, Co, Cu, Fe, Hg, Ni, Os, Pb, Rh, Ru & Sc	Color: Off-white Density: 0.736 g/mL Solvent Compatibility: 2 Prolonged Storage: 2 Shelf Life: 2 Years
	SiliaMetS AMPA PN: R85130B Loading: ≥ 0.80 mmol/g Endcapping: Yes	С он он он он он он он он	Silia <i>MetS</i> AMPA is an aminomethyl-alkylphosphonic acid ligand known for its excellent metal-bonding properties. It is particularly efficient to remove AI, Sb, Ni, La, and also very effective for Co, Cu, Fe, Mg and Zn scavenging from reaction intermediates or final APIs.	Al, Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Mg, Mn, Nd, Ni, Pm, Pr, Sb, Sm, Tb, Tm, V, Yb & Zr Co, Cu, Fe, Mg & Zn	Color: Yellow Density: 0.707 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 1 Year
	SiliaMetS Cysteine PN: R80530B Loading: ≥ 0.30 mmol/g Endcapping: Yes	SH CNa	Silia <i>MetS</i> Cysteine is the silica-bound equivalent of the amino acid cysteine. It is a versatile scavenger for a variety of metals and the preferred metal scavenger for tin residues. By attaching the molecule to the backbone via the amino group, the thiol group remains free and accessible for higher metal scavenging efficiency.	Au, Cd, Fe, Ir, Os, Ru, Sc & Sn Ca, Cr, Cs, Cu, La, Mg, Pd, Pt, Rh & Zn	Color: Orange Density: 0.665 g/mL Solvent Compatibility: 1 (although we recommend organic solvent when possible) Prolonged Storage: 1 Shelf Life: 1 Year
pgl-5	SiliaMetS DEAM PN: R54430B Loading: ≥ 0.85 mmol/g Endcapping: Yes	Сон сон	Silia <i>MetS</i> DEAM is a versatile scavenger designed to remove trace metal of Ti, Zn, Fe and Ag as well as boronic acids from reaction intermediates or final APIs.	Ag, Au, Fe, Sn, Ti, Zn & Zr	Color: Off-white Density: 0.691 g/mL Solvent Compatibility: 1 Prolonged Storage: 2 Shelf Life: 2 Years

	\mathcal{L}		SiliaMetS Metal Scavengers Technical Info	ormation		ng
	Scavengers	Structure	Brief Description	Metals Removed ¹	Typical Characteristics ^{2,3}	engi
pGI-5	SiliaMetS DOTA PN: R91030B Loading: ≥ 0.38 mmol/g Endcapping: Yes	Contraction of the second seco	SiliaMetS DOTA is a silica-supported tetracarboxylic acid and its various conjugate bases. DOTA molecule is a well-adopted complexing agent. Linked to various metals, so formed-complexes are used as contrast agents in cancer treatments or other medical applications.	Ca, Cu, Gd, La, Ni & Zn Co, Fe, Mg, Pd, Pt & Rh	Color: Light yellow Density: 0.681 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 1 Year	Scavenging
	SiliaMetS Imidazole PN: R79230B Loading: ≥ 0.96 mmol/g Endcapping: Yes		Silia <i>MetS</i> Imidazole is a versatile metal scavenger for a variety of metals including Cd, Co, Cu, Fe, Ni, Os, Pd and Rh.	Cd, Co, Cu, Fe, Ir, Li, Mg, Ni, Os, W & Zn Cr, Pd & Rh	Color: Off-white Density: 0.681 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years	lesis
GI-5	SiliaMetS TAAcOH PN: R69030B Loading: ≥ 0.41 mmol/g Endcapping: No		Silia <i>MetS</i> TAAcOH & TAAcONa are supported versions of EDTA in their acid and sodium salt forms. These two products are effective metal scavengers for Ca, Mg, Li, Ir, Cs, Os, Sn, Pd, Ni	Au, Ca, Co, Ir, Li, Mg, Ni, Os, Ru & Sc Cr, Cs, Fe, Pd, Rh & Sn	Color: Off-white Density: 0.635 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years	Synthesis
	SiliaMetS TAAcONa PN: R69230B Loading: ≥ 0.41 mmol/g Endcapping: No	The second secon	and Cu. SiliaMetS TAAcOH is effective for metals in low or zero oxidation states, compared to SiliaMetS TAAcONa which is useful for metals in higher oxidation states (≥ 2).	Ca, Cd, Cs, Cu, Fe, Ir, La, Li, Mg, Ni, Os, Rh, Sc & Sn Cr, Pd, Ru & Zn	Color: Off-white Density: 0.712 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years	graphy
	SiliaMetS Thiourea PN: R69530B Loading: ≥ 1.07 mmol/g Endcapping: Yes	an ^h r	Silia <i>MetS</i> Thiourea is a versatile metal scavenger for all forms of palladium and is widely used in the pharmaceutical industry. Once complexed with a transition metal, it has been reported to be an effective catalyst.	Au, Pd & Ru Ag, Cu, Fe, Os, Rh, Sc & Sn	Color: Off-white Density: 0.767 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years	Chromatography
GI-5	SiliaBond Tosic Acid PN: R60530B Capacity: ≥ 0.54 meq/g Endcapping: Yes	аланананананананананананананананананана	Silia <i>Bond</i> Tosic Acid is in a class of strong acids used in different fields of synthetic organic chemistry. The aromatic ring makes it slightly more acidic than other supported sulfonic acids.	Fe, Rh & Sn Ag, Cu, Ni, Pd, Pt, Ru & Zn	Color: Off-white Density: 0.698 g/mL Solvent Compatibility: 2 Prolonged Storage: 1 Shelf Life: 2 Years	Sample Preparation
	¹ Scavenging Efficie		particular matal is indicated in Naur Plus			Sample Pr

¹ Scavenging Efficiency:

- Best scavenger for the removal of a particular metal is indicated in Navy Blue
- Good scavenger indicated in Pale Blue

² Solvent Compatibility:

- 1- All solvents, aqueous and organic
- 2- All organic solvents

³ Prolonged Storage:

- 1- Keep dry
- 2- Keep cool (< $8^{\circ}C$) and dry
- 3- Keep cool (< $8^{\circ}C$), dry and under inert atmosphere



Potentially Genotoxic Impurities (*PGI*) Scavenger See pages 16 - 17 or contact us for more information





SiliaMetS Metal Scavengers Selection Table

This table will help in selecting the most efficient scavenger for a specific metal and application. Since some parameters may affect the efficiency of the scavenging, we recommend performing a preliminary screening experiment using the Silia*MetS* Metal Scavenger Kit.

When selecting a metal scavenger, every parameter must be considered: metal catalyst, solvent, residual reagents, by-products, structure of the API (*or molecule of interest*) and temperature.

Best scavenger: Good scavenger:

		SiliaMetS I	Metal Scaveng <u>er</u>	s Selection Table							SiliaMetS Metal S	Scavenger <u>s Sele</u>	ction Table		
	Silia <i>MetS</i>	SiliaMetS	Silia Bond	SiliaMetS	SiliaMetS	SiliaMetS	Silia <i>MetS</i>	Silia <i>MetS</i>	Silia <i>MetS</i>	SiliaMetS	SiliaMetS	Silia <i>MetS</i>	Silia <i>MetS</i>	SiliaBond	
Scavenger	Thiol	DMT	Amine	AMPA	Cysteine	DEAM	Diamine	DOTA	Imidazole	TAAcOH	TAAcONa	Thiourea	Triamine	Tosic Acid	Scavenger
	(Si-Thiol)	(Si-DMT)	(Si-WAX)	(Si-AMPA)	(Si-CYS)	(Si-DEAM)	(Si-DIA)	(Si-DOTA)	(Si-IMI)	(Si-TAAcOH)	(Si-TAAcONa)	(Si-THU)	(Si-TRI)	(Si-SCX)	-
PN	R51030B	R79030B	R52030B	R85130B	R80530B	R54430B	R49030B	R91030B	R79230B	R69030B	R69230B	R69530B	R48030B	R60530B	PN
Loading (mmol/g)	≥ 1.20	≥ 0.50	≥ 1.20	≥ 0.80	≥ 0.30	≥ 0.85	≥ 1.28	≥ 0.38	≥ 0.96	≥ 0.41	≥ 0.41	≥ 1.07	≥ 1.11	≥ 0.54 meq/g	Loading (<i>mmol/g</i>)
Typical Density (g/mL)	0.682	0.732	0.700	0.707	0.665	0.691	0.728	0.681	0.681	0.635	0.712	0.767	0.736	0.698	Typical Density (g/mL)
Ag															Ag
Al				-											Al
Ar As															Ar
As															As Au
Ca	-														Ca
Cd								-							Cd
Ce			-												Ce
Co								•							Co
Cr															Cr
Cs															Cs
Cu															Cu
Fe								•							Fe
Gd															Gd
Hg															Hg
ъ Ir															lr o
be La															La
Li															Li
м мg															Mg e
Mn Is															Mn Q
Ni Meta															gM nM wetars to be iN
Os													•		Os
Pb															Pb
Pd								•							Pd
Pt								•							Pt
Re															Re
Rh								•					•		Rh
Ru															Ru
Sb															Sb
Sc							•	_					•		Sc
Se	•							•							Se
Sn Ti										•		•			Sn
Ti															Ti V
w				-											w v
Zn														•	Zn
Zn							-								Zn
21															21





SiliaBond Organic Scavengers Portfolio

	SiliaBo	ond Organic Scavengers Tecl	hnical Information			SiliaBo	ond Organic Scavengers Tec	hnical Information		bu
Scavengers	Structure	Nature	Molecules Removed	Typical Characteristics ^{1, 2}	Scavengers	Structure	Nature	Molecules Removed	Typical Characteristics ^{1, 2}	avenging
Silia^{Bond} Amine PN: R52030B	S NH2	Scavenger for Electrophiles (Covalent Bonding)	(Covalent Bonding) Isocyanates, Ketones & Color: Off-white Sulfonyl Chlorides Density: 0.700 g/mL		SiliaBond Piperazine PN: R60030B		Scavenger for Electrophiles (Covalent Bonding)	Acyl Chlorides, Aldehydes, Anhydrides, Chloroformates, Isocyanates, Ketones & Sulfonyl Chlorides	Color: Off-white Density: 0.671 g/mL Solvent Compatibility: 1	SC
Loading: ≥ 1.20 mmol/g Endcapping: Yes		Scavenger for Acids (<i>Ionic Bonding</i>) Catch & Release	Acids & Acidic Phenols	Solvent Compatibility: 2 Prolonged Storage: 2 Shelf Life: 2 Years	Loading: ≥ 0.83 mmol/g Endcapping: Yes	, NH	Scavenger for Acids (<i>Ionic Bonding</i>) Catch & Release	Acids & Acidic Phenols	Solvent Compatibility: 1 Prolonged Storage: 2 Shelf Life: 2 Years	sis
SiliaBond Carbamate PN: R50130B Loading: ≥ 1.16 mmol/g Endcapping: Yes	Solution to the second	Scavenger for Nucleophiles (Covalent Bonding)	Alcohols, Alkoxides, Amines, Anilines, Hydrazines, Organometallics, Thiols & Thiolates	Color: Off-white Density: 0.741 g/mL Solvent Compatibility: 3 Prolonged Storage: 2 Shelf Life: 2 Years	SiliaBond Propylsulfonic Acid PN: R51230B Loading: ≥ 0.63 mmol/g Endcapping: Yes	а совется в совется в совется в совется в со	Scavenger for Bases (Ionic Bonding)		Color: Off-white Density: 0.728 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years	Synthe
SiliaBond Carbonate PN: R66030B Loading: ≥ 0.46 mmol/g Endcapping: Yes	(CO ₂ ²) ₀₅	Scavenger for Acids (<i>Ionic Bonding</i>) Catch & Release	Acids, Acidic Phenols & Boronic Acids	Color: Off-white Density: 0.608 g/mL Solvent Compatibility: 3 Prolonged Storage: 1 Shelf Life: 1 Years	SiliaBond Tosic Acid PN: R60530B Loading: ≥ 0.54 meq/g Endcapping: Yes	я страна стран	Catch & Release	Amines & Anilines	Color: Off-white Density: 0.698 g/mL Solvent Compatibility: 2 Prolonged Storage: 1 Shelf Life: 2 Years	aphy
SiliaBond Carboxylic Acid PN: R70030B Loading: ≥ 0.92 mmol/g Endcapping: Yes	Элдон	Scavenger for Bases (<i>Ionic Bonding</i>) Catch & Release	Primary / Secondary Amines & Anilines	Color: Off-white Density: 0.687 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years	SiliaBond TMA Acetate PN: R66430B Loading: ≥ 0.71 mmol/g Endcapping: No	а С _{днсоо} .	Scavenger for Acids (<i>Ionic</i> Bonding) Catch & Release	Carboxylic Acids	Color: Off-white Density: 0.665 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years	togr
SiliaMetS DEAM PN: R54430B Loading: ≥ 0.85 mmol/g Endcapping: Yes	С	Scavenger for Electrophiles & Lewis Acids (Covalent & Ionic Bonding) Catch & Release	Boronic Acids	Color: Off-white Density: 0.691 g/mL Solvent Compatibility: 1 Prolonged Storage: 2 Shelf Life: 2 Years	SiliaBond Tosyl Chloride PN: R44030B Loading: ≥ 0.63 mmol/g Endcapping: Yes		Scavenger for Nucleophiles (Covalent Bonding)	Alcohols, Alkoxides, Amines, Anilines, Hydrazines, Organometallics, Thiols & Thiolates	Color: Off-white Density: 0.761 g/mL Solvent Compatibility: 4 Prolonged Storage: 3 Shelf Life: 6 months	n
SiliaMetS Diamine PN: R49030B		Scavenger for Electrophiles (Covalent Bonding)	Acyl Chlorides, Aldehydes, Anhydrides, Chloroformates, Isocyanates, Ketones & Sulfonyl Chlorides	Color: Off-white Density: 0.728 g/mL Solvent Compatibility: 2 Prolonged Storage: 2	SiliaMetS Triamine PN: R48030B	G NH2 NH2	Scavenger for Electrophiles (Covalent Bonding)	Acyl Chlorides, Aldehydes, Anhydrides, Chloroformates, Isocyanates, Ketones & Sulfonyl Chlorides	Color: Off-white Density: 0.736 g/mL	e Prepar
Loading: ≥ 1.28 mmol/g Endcapping: Yes		Scavenger for Acids (<i>Ionic Bonding</i>) Catch & Release	Acids & Acidic phenols	Solvent Compatibility: 2 Prolonged Storage: 2 Shelf Life: 2 Years	Loading: ≥ 1.11 mmol/g Endcapping: Yes	NH₂	Scavenger for Acids (<i>Ionic</i> Bonding) Catch & Release	Acids & Acidic Phenols	- Solvent Compatibility: 2 Prolonged Storage: 2 Shelf Life: 2 Years	Sam
SiliaBond Diol PN: R35030B Loading: ≥ 0.97 mmol/g Endcapping: No	С С С С С С С С С С С С С С С С С С С	Scavenger for Electrophiles & Lewis Acids (Covalent & Ionic Bonding) Catch & Release	Boronic Acids	Color: Off-white Density: 0.687 g/mL Solvent Compatibility: 2 Prolonged Storage: 1 Shelf Life: 2 Years	¹ <u>Solvent Compatibility:</u> 1- All solvents, aqueous and 2- All organic solvents	organic		·	·	Analysis
SiliaBond DMAP PN: R75630B Loading: ≥ 0.53 mmol/g Endcapping: Yes	G~~	Scavenger for Electrophiles (Covalent Bonding)	Acyl Chlorides & Sulfonyl Chlorides	Color: Light brown to brown Density: 0.674 g/mL Solvent Compatibility: 1 Prolonged Storage: 3 Shelf Life: 1 Years	 3- Anhydrous aprotic solvent: 4- Anhydrous aprotic solvent: 5- Polar solvents (<i>DMF, MeO</i>) 	s, unstable in DMF				A
SiliaBond Guanidine PN: R68230B Loading: ≥ 0.80 mmol/g Endcapping: Yes		Scavenger for Acids (<i>Ionic Bonding</i>) Catch & Release	Acids, Acidic Phenols & Boronic Acids	Color: Light yellow Density: 0.732 g/mL Solvent Compatibility: 1 Prolonged Storage: 1 Shelf Life: 2 Years	² <u>Prolonged Storage:</u> 1- Keep dry 2- Keep cool (< 8°C) and dry 3- Keep cool (< 8°C), dry and		here			2D Services



Potentially Genotoxic Impurities (*PGI*) Scavenger See pages 16-17 or contact us for more information



pGI-



SiliaBond Organic Scavengers Selection Table

Silia*Bond* Organic Scavengers can help you purify your API. Functional group is bound to silica, that will specifically react with a given product. Use the double-entry chart below to choose the best match for the impurity you are dealing with.

Electrophile scavenger (covalent bonding):

Nucleophile scavenger (covalent bonding): 🔻

Ya.		Silia	Bond Organic Sc	avengers Selection	on Table			SiliaBond Organic Scavengers Selection Table							
Organic Scavenger	Silia <mark>Bond</mark> Amine (Si-WAX)	Silia <mark>Bond</mark> Carbamate (Si-CAR)	Silia <mark>Bond</mark> Carbonate (<i>Si-CO₃</i>)	Silia <mark>Bond</mark> Carboxylic Acid (Si-WCX)	Silia <i>MetS</i> DEAM (S <i>i-DEAM</i>)	Silia <i>MetS</i> Diamine (Si-DIA)	Silia Bond Diol nec (Si-Diol)	Silia <mark>Bond</mark> DMAP (Si-DMAP)	Silia <mark>Bond</mark> Guanidine (Si-GUA)	Silia <mark>Bond</mark> Piperazine (Si-PPZ)	Silia <mark>Bond</mark> Propylsulfonic Acid (<i>Si-SCX-2</i>)	Silia <mark>Bond</mark> Tosic Acid (Si-SCX)	Silia <mark>Bond</mark> TMA Acetate nec (Si-SAX ₂)	Silia <mark>Bond</mark> Tosyl Chloride nec (Si-TsCl)	Silia <i>MetS</i> Triamine (Si-TRI)
Product Number	R52030B	R50130B	R66030B	R70030B	R54430B	R49030B	R35030B	R75630B	R68230B	R60030B	R51230B	R60530B	R66430B	R44030B	R48030B
Loading	≥ 1.20 mmol/g	≥ 1.16 mmol/g	≥ 0.46 mmol/g	≥ 0.92 mmol/g	≥ 0.85 mmol/g	≥ 1.28 mmol/g	≥ 0.97 mmol/g	≥ 0.53 mmol/g	≥ 0.80 mmol/g	≥ 0.83 mmol/g	≥ 0.63 meq/g	≥ 0.54 meq/g	≥ 0.71 mmol/g	≥ 0.63 mmol/g	≥ 1.11 mmol/g
Typical Density	0.700 g/mL	0.741 g/mL	0.608 g/mL	0.687 g/mL	0.691 g/mL	0.728 g/mL	0.687 g/mL	0.674 g/mL	0.732 g/mL	0.671 g/mL	0.728 g/mL	0.698 g/mL	0.665 g/mL	0.761 g/mL	0.736 g/mL
Solvent Compatibility	All organic solvents	Anhydrous aprotic solvents	Anhydrous aprotic solvents	All aqueous & organic solvents	All aqueous & organic solvents	All organic solvents	All organic solvents	All aqueous & organic solvents	All aqueous & organic solvents	All aqueous & organic solvents	All aqueous & organic solvents	All organic solvents	All aqueous & organic solvents	Anhydrous aprotic solvents, unstable in DMF	

Ionic bonding:

Functionalities to be scavenged

Acids & Acidic Phenols										
Acyl & Sulfonyl Chlorides	•				•	•	•			•
Alcohols & Alkoxides									▼	
Aldehydes, Anhydrides, Chloroformates, Isocyanates & Ketones	•				•		•			•
Amines & Anilines									▼	
Boronic Acids				•		••				
Carboxylic Acids										
Hydrazines										
Organometallics, Thiols & Thiolates		•							▼	

Potential Genotoxic Impurities (PGI) Scavengers Selection Table



Eleven compounds containing structurally alerting functional groups were studied and scavenged by at least one of our grafted silica.

Best scavenger: Good scavenger:

	Scavenging Affinity for Various Potentially Genotoxic Impurities						Scavenging Affinity for Various Potentially Genotoxic Impurities					
Alerting Functional Groups	Acetamide	Pyridine N-oxide	Aniline	Phenyl- hydroxylamine	Benzaldehyde	Octaldehyde	Methylmethane sulfonate	Propiolactone	Benzyl bromide	Allyl bromide	1,2-epoxyoctane	Alerting Functional
	H ₂ N H	С, ⁰	NH ₂	Н. ОН	ОН	O H	0,0 -5.0-		Br	Br		Groups
Silia <i>MetS</i> DMT												Silia <i>MetS</i> DMT
SiliaMetS Thiourea												SiliaMetS Thiourea
SiliaMetS Triamine							•					SiliaMetS Triamine
SiliaMetS TAAcOH												SiliaMetS TAAcOH
SiliaMetS TAAcONa							•					SiliaMetS TAAcONa
SiliaBond Tosic Acid												SiliaBond Tosic Acid
SiliaBond Tosyl Chloride												SiliaBond Tosyl Chloride



Catch and release:



Typical Experimental Procedures

In Batch Mode Reactor (Bulk)

YL.	Typical Experimental Procedures	
STEP	For initial screening, start with 4 - 8 molar equivalents* of Silia <i>MetS /</i> Silia <i>Bond</i> in respect to the residual metal or organic impurity concentration.	
1	Dissolve the crude product to be treated in a suitable solvent (<i>or directly use the crude reaction mixture</i>) and prepare vials containing the same solution volume. Directly add your chosen Silia <i>MetS</i> / Silia <i>Bond</i> to these vials. Note : no pre-wetting / pre-activation is required.	SLICYCLE C Stgr-by-step experimental procedure using scawergers
2	For initial tests, stir the solution for at least one hour at room temperature.	
	Scavenging progress can be followed by normal analytical techniques.	
	The scavenging progress can also be estimated by looking at the color of the solution, as depicted herein:	Watch the step-by-step experimental procedure vide
3		
	When the scavenging is almost complete, the solution is less colored and Silia <i>MetS /</i> Silia <i>Bond</i> becomes colored.	T
	In some occasional cases, if all the samples are still colored, try one or all of the following: let them react for a longer period of time; add more equivalents of the Silia <i>MetS /</i> Silia <i>Bond</i> ; increase the temperature of the reaction.	SILICYCLE CO What is the best way to calculate the amount of scavengers needed?
	Keep in mind that colors can come from other factors unrelated to the impurities.	
4	At the end of the scavenging, filter off the scavenger using a fritted funnel or filtration device.	5 0
5	Wash the Silia <i>MetS /</i> Silia <i>Bond</i> with additional solvent for total recovery of the API (or compound of interest) and concentrate the solution under vacuum.	Take a look at our FAQ abou how to calculate the amoun
6	Analyze the residual metal or organic impurity concentration of each vial to identify the most efficient scavenger.	of scavenger needed
7	Direct scale-up is now possible. Otherwise, scavenging optimization can be examined.	

* Please see page 20 for calculation exemple. Please keep in mind that the above procedures are standard and introductory, but optimization of conditions is key to optimal scavenging efficiency.

In SPE or Flash Cartridges

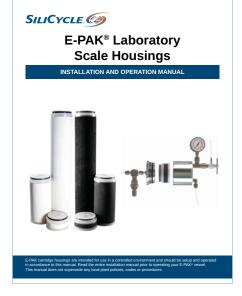
Fixed-bed formats are a great alternative for metal or organic removal and are directly scalable. We suggest initial screening investigations to be done using SiliaPrep 2 g / 6 mL SPE cartridges.

	Ú.	Typical Experimental Procedures						
Ş	STEP	Description						
	1	Condition the cartridge with 1 column volume using the same solvent as the solution to be treated.	1. Conditioning	2. Equilibration	3. Loading	4. Washing	5. Elution	
	0	Add the solution containing the API and the metal or organic impurity to the top of the cartridge and let it pass through the cartridge under gravity.			Analytes:			
	2	Note : if needed, a slight positive pressure on the top of the cartridge or a light vacuum at the bottom can be applied to speed up the flow rate.					e en imp	pu
	3	For most Silia <i>MetS</i> metal scavengers, a dark colored band will be observed on the top of the silica bed.			Non-retai	ed Eluted	Elu	
		Note : if the residual solution is still colored, multiple passes through the same cartridge can be done.	~		impurities	impuritie	as ana	aly
	4	Once the scavenging is completed, wash the cartridge using at least 1 column volume of solvent to ensure total API recovery.	-					

In E-PAK Cartridges

	Typical Experimental Procedures
STEP	Description
1	Ensuring that the selected E-PAK cartridge has been installed, it system with at least 3 tank volumes using a compatible solvent. carbon fines and other particulates from the system that could p This is best accomplished using solvent delivered at ≥ 10 mL/mi
2	Use bleed valve to remove trapped air from the housing then clo 3 tank volumes of solvent through the system. The solvent shou Typical tank volume per cartridge size can be found in the table
3	Once the system flushing is completed, set the pump to a low flo
4	Fully open the air bleed valve on top of the head of the housing.
	Turn the pump on and watch for test fluid exiting from the bleed
5	Note : Over the course of the test run, additional gas produced d action can accumulate in the housing. Periodically this gas shou valve to prevent interference with even flow distribution through
6	When the bleed valve begins discharging liquid, indicating that a system, close the bleed valve.
7	Observe the pressure gauge after closing the bleed valve. Press pressure increases rapidly, immediately shut the pump off and d and inspect for blockage. If no blockages are identified, consider viscosity or high solids can create pressure difficulties.
	Note: we recommend that users pre-filter test solutions containing solids.
8	Begin sample loading on the cartridge and collection at intervals a circulate the solution for the number of desired runs.
0	Note : during sample collection, periodically monitor flow and pre- pump setting as necessary to maintain target range.
9	Once the process is completed, flush and collect the system with compatible solvent.
10	When no more liquid is collected from the outlet valve, turn off th

E-PAK Housings Installation and Operation Manuals To get your copies of our Installation and Operation Manuals contact us at scavengers@silicycle.com





Retained impurities

Eluted analytes

Scavenging

Analysis

R&D Services

it is recommended to flush the . This will remove loose silica/ plug the back pressure control valve. nin.

lose the valve and pump at least uld be collected and discarded. e page 7.

flow rate.

l valve.

due to degassing from the pump uld be removed through the bleed the adsorbent cartridge.

all air has been bled from the

ssure should increase slowly. If disassemble the cartridge housing er feed material characteristics; high

ing a large amount of insoluble

according to desired data needs or

essure, adjusting metering valve and

ith 2 or 3 tank volumes of fresh clean

the pump.





See how easy it is working with E-PAK

SILICYCLE (

Experimental Procedure Optimization in Bulk

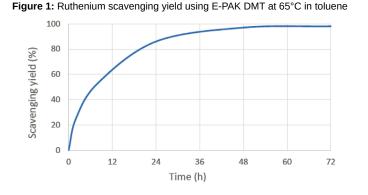
Following a screening test whereby the most performant scavenger has been selected, optimization steps can then be undertaken. There are nine main parameters that can be adjusted either independently one at a time or simultaneously, to improve overall scavenging efficiency. By experience, some are known to influence scavenging more than others and have therefore been grouped in three main steps.

1. First Step

1.1 Reaction Time

Increasing the contact time of the scavenging process can lead to improved efficiency. In fact, scavenging should be pursued until a plateau is reached.

See the example on the right and you can take a look at the Application Note Appn EP008-0 where the scavenging was done using the E-PAK technology, but nevertheless the same concept is applicable to the bulk media.



1.2 Amount of Scavenger

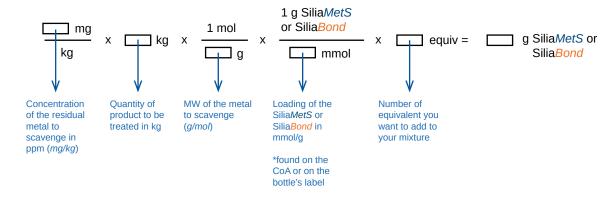
The amount of scavenger can play a role and should be fine tuned once the initial screening is completed.

Two different methods are suggested to calculate the amount of scavenger required: the molar equivalent method and the weight / weight method. The first makes it possible to calculate the exact number of equivalents to add, however it also requires having previously determined the level of metal contamination. On the other hand, the weight / weight method will allow to skip the pre-analysis step at the expense of risking an overestimate of the quantity really needed.

Molar Equivalent Method

For initial screening experiments, we suggest using 4 - 8 molar equivalents of scavengers relative to the concentration of the residual impurity. Once the optimized number of equivalents is selected, redo the calculation for the complete batch to purify.

Here is how to calculate the amount of scavenger to use:

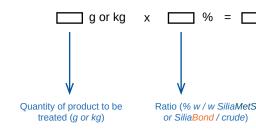


For example, if you need to scavenge 800 g of material containing 500 ppm (mg/kg) of Pd (MW of Pd: 106.42 g/mol) with SiliaMetS Thiol with a loading of 1.2 mmol/g and you want to add 4 molar equivalents:



The Weight / Weight Method

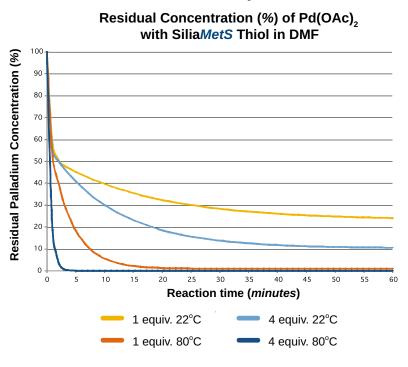
For initial screening experiments we suggest starting your tests with 20 % w / w ratio of scavenger vs crude. Once the optimized number of equivalents is selected, redo the calculation for the complete batch to purify.



For example, if you need to scavenge 800 g of material containing an unknown quantity of Pd.

1.3 Temperature

Generally, it is recommended to perform initial screenings at room temperature and this usually results in scavenging being completed within few hours. When shorter scavenging times are required, higher scavenging rates can be achieved by increasing the temperature as showed in Figure 2. Increasing temperature not only improves the kinetic of the reaction, it can also improve the scavenging efficiency. The SiliaMetS and SiliaBond products can be safely used without degradation at temperatures up to 150°C.



 \Box g or kg x \Box % = \Box g or kg of Silia*MetS* or Silia*Bond*

800 g x 20 % = 160 g of SiliaMetS or SiliaBond

Figure 2: Residual concentration (%) of Pd(OAc), with SiliaMetS Thiol in DMF



2. Second Step

2.1 Combination of Scavengers

A strategy that may be worthwhile is to mix multiple scavengers together, as some have shown to exhibit a synergistic effect that can produce a higher scavenging yield than either one independently. For example, in the <u>Application Note Appn_EP008-0</u>, a combination of two scavengers allowed to greatly improve the Ruthenium scavenging while using both separately gave lower results.

2.2 Subsequent Treatments

In some cases (*equilibrium process or the presence of multiple species*), multiple treatments in a row are preferable over a single larger treatment. It should be noted that for optimal results, filtration between each treatment must be carried out. An example of this method is shared in the Case Study CS_SM010-0. For their experiment, the researchers performed two subsequent treatments with Silia*MetS* Thiol in order to reduce the Palladium concentration to the desired amount instead of doing one larger batch treatment.

2.3 Pre-treatment with Activated Carbon

For large quantities of metal removal, a good strategy could be to pre-treat the solution with Silia*Carb* activated carbon prior to the scavenger treatment. This is an approach sometimes used by SiliCycle's researchers.

For example, in the <u>Application Note Appn_EP002-0</u>, a pre-treatment with activated carbon greatly reduced the Palladium concentration prior to the metal scavenger treatment.

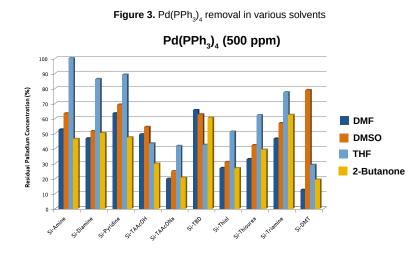
3. Third Step

Finally, if the two first sets of parameters do not provide a satisfying level of impurities removal, a final third set can be attempted. While these tend to be less effective than the previous ones described above, their effect on scavenging has been helpful in some cases. Hence why they are still recommended.

3.1 Solvent

A wide range of organic and aqueous solvents commonly used in laboratory and in process, such as DMF, DMSO, THF, 2-butanone, alcohols, ethers, chlorinated solvent, etc. can safely be used with metal scavengers. Exceptions are amine scavengers (*i.e. Si-Amine, Si-Diamine, and Si-Triamine*) which should not be used under aqueous conditions. Moreover, some are solely compatible with aprotic solvents (*Si-Carbonate, Si-Carbamate, and Si-Tosyl Chloride (also unstable in DMF)*).

As demonstrated in the Figure 3, the nature of the solvent can sometimes influence scavenging efficiency. If scavenging or reaction kinetics are too slow, changing solvent or adding a co-solvent should be considered.



3.2 Mixing Rate

SiliCycle's scavengers are mechanically stable and offer excellent scavenging efficiency in batch processes agitated by overhead stirrers as well as orbital shaking under low to moderate agitation rates. If required, mixing rates can be increased to get better scavenging results. With faster stirring, scavenger dispersion in solution is improved. Since silica-based products have an excellent mechanical stability, no worries grinding nor crushing the Silia*MetS* and Silia*Bond* products by applying a more vigorous stirring.

3.3 pH of the Aqueous Solution

When the scavenging is carried out in aqueous conditions, our recommended pH range is between 2 and 10. Depending on the nature of the scavenging agent, the pH can modify the functional groups present on the scavengers by charging them, thereby possibly affecting scavenging (*e.g.: amine groups in acidic conditions*). The various species in solution could also be affected by the pH variation modifying electronic effects, such as H-bonding for example, which could also have an influence on the scavenging efficiency.

SILICYCLE

Experimental Procedure Optimization in E-PAK

Following the screening tests described for Silia*MetS* where the best Silia*Carb* and / or Silia*MetS* have been identified, the optimization of the experimental conditions can start. There are 5 parameters that can be optimized to improve the scavenging efficiency with E-PAK.

1. First Step

1.1 Amount of Scavenger

Both the molar equivalent (*page 20*) and the weight / weight method (*page 21*) are suitable to calculate the amount of scavenger needed for the reaction. Once this has been determined, the corresponding E-PAK cartridge needs to be selected using the table below.

Δ	E-PAK Cartridge Selection	
Weight of Silia <i>Flash</i> , Silia <i>MetS</i> and Silia <mark>Bond</mark> (g)	Weight of Silia <i>Carb</i> (g)	Cartridge Size Required (cm)
0 to 8	0 to 5	5 x 1
> 8 to 75	> 5 to 50	5 x 10
> 75 to 200	> 50 to 125	5 x 25
> 200 to 875	> 125 to 550	16.5 x 12.5
> 875 to 1,750	> 550 to 1,100	16.5 x 25
> 1,750 to 3,500	> 1,100 to 2,100	16.5 x 50
> 3,500 to 7,000	> 2,100 to 4,100	16.5 x 100

1.2 Residence Time

The residence time or contact time refers to the time the solution spend in the E-PAK cartridge. We suggest to start your experiment with a residence time of 2.5 minutes as it was experimentally determined that this gave the best results. However, depending on the conditions and the type of impurities the results can vary. The residence time is correlated with the flow rate of the experiment, a higher flow rate will give a shorter residence time, while a lower flow rate will give a longer residence time.

2. Second Step

2.1 Selecting the Method

When using E-PAK, four methods are possible to maximize your scavenging yield (see *figure on the right*).

- **Single Pass**: consists on passing the solution through the E-PAK housing once, yielding the desired purity.
- Recirculation: which is the most common, consists on recirculating the solution until getting the desired scavenging yield.
- Multiple Sequential Passes: the solution is passing through like a single pass several times.
- Series Pass: consists on having 2 or more housings connected in series where you can recirculate the solution. In this case you can have different adsorbants or the same one.

2.2 Pre-treatment with Activated Carbon

Similar to the Silia*MetS*, if a large amount of contaminant needs to be removed, a pre-treatment with the Silia*Carb* activated carbon E-PAK could reduce it significantly, allowing the next scavenging step to get to the desired purity level (*for more infomation, take a look at this document <u>Application Note Appn EP002-0</u>).*

2.3 Temperature

Keeping in mind that the housings do not have any heating element and that the E-PAK cartridges are made with some polymers, it is possible to increase the temperature up to a maximum of 80°C to see if any significant increase in scavenging yield is noticed. When using the lab scale housing, the heat dissipation can be negligeable therefore getting your solution in the housing at the right temperature could be done. However when using pilot and commercial scale housings, an alternative will have to be found to minimize the heat dissipation (*heating up the pipes, etc.*).





Scavenging



Ordering Information

Batch Reactor Mode (Bulk)

All scavengers particle size and pore size are respectively 40 - 63 µm and 60 Å and are available in the following sizes: 5 g, 10 g, 25 g, 50 g, 100 g, 250 g, 500 g, 1 kg, 5 kg, 10 kg, 25 kg, etc. (Up to multi-ton scale!)

To build your own product number, just add the Format to the Phase PN: [Phase PN]-[Format Code]

Example: 100 g of Thiol silica gel, 40 - 63 µm, 60 Å: R51030B-100G

SiliaMetS Metal Scavengers					
Scavenger Name	Scavenger PN				
SiliaMetS AMPA	R85130B				
SiliaBond Amine (WAX)	R52030B				
SiliaMetS Cysteine	R80530B				
SiliaMetS DEAM	R54430B				
SiliaMetS DOTA	R91030B				
SiliaMetS DMT	R79030B				
SiliaMetS Diamine	R49030B				
SiliaMetS Imidazole	R79230B				
SiliaMetS TAAcOH nec	R69030B				
SiliaMetS TAAcONa nec	R69230B				
SiliaMetS Thiol	R51030B				
SiliaMetS Thiourea	R69530B				
SiliaBond Tosic Acid (SCX)	R60530B				
SiliaMetS Triamine	R48030B				

All activated carbons are available in 100 g, 1 kg, and 20 kg packagings.

SiliaCarb Activated Carbon				
Carbon Name	Carbon PN			
SiliaCarb CA	C-CA			
SiliaCarb HA	C-HA			
SiliaCarb VA	C-VA			
SiliaCarb VW	C-VW			

SiliaBond Organic Scavengers					
Scavenger Name	Scavenger PN				
Silia <mark>Bond</mark> Amine (WAX)	R52030B				
SiliaBond Carbonate	R66030B				
SiliaBond Carboxylic Acid (WCX)	R70030B				
SiliaMetS DEAM	R54430B				
SiliaBond DMAP	R75630B				
SiliaMetS Diamine	R49030B				
Silia <mark>Bond</mark> Diol	R35030B				
SiliaBond Guanidine	R68230B				
SiliaBond Carbamate	R50130B				
SiliaBond Piperazine	R60030B				
SiliaBond PropyIsulfonic Acid (SCX-2)	R51230B				
SiliaBond TMA Acetate nec	R66430B				
SiliaBond Tosic Acid (SCX)	R60530B				
SiliaBond Tosyl Chloride	R44030B				
SiliaMetS Triamine	R48030B				

Bulk Formats							
Quantity	Code	Quantity	Code				
5 g	5G	500 g	500G				
10 g	10G	1 kg	1KG				
25 g	25G	5 kg	5KG				
50 g	50G	10 kg	10KG				
100 g	100G	20 kg	20KG				
250 g	250G	25 kg	25KG				

Available Kit

Because all matrices are unique, and that small differences can influence scavenging efficiency, we recommend first trying one of our scavenger kits for screening purposes, especially if you are new to this technology. Steric hindrance of the catalyst, electronic effects, solubility in solvents, all are factors that can influence the removal of your impurity.

How to order: simply note the kit number which starts with "K", add a dash mark and your choice of format. Example: K30730B-10G to obtain 10 g of each one of the scavengers listed in the kit.

SiliaMetS Metal Scavenger Kit						
Kit Name	Kit Number	Composition				
SiliaMetS Universal Metal Scavenger Kit	K30730B	Cysteine, DMT, Imidazole, TAAcOH, TAAcONa, Thiol, Thiourea & Triamine				

These kits are available in 5 g, 10 g, 25 g, 50 g, and 100 g formats.

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E-PAK Cartridges

E-PA							
Туре	Labora	tory Scale - 5 cm d	iameter				
туре	1 cm	10 cm	25 cm				
SiliaFlash Iri	SiliaFlash Irregular Silica Gel						
40 - 63 µm	LS-R10030B-51	LS-R10030B-510	LS-R10030B-525				
60 - 200 µm	LS-R10040B-51	LS-R10040B-510	LS-R10040B-525				
SiliaMetS Me	etal Scavengers						
Thiol	LS-R51030B-51	LS-R51030B-510	LS-R51030B-525				
DMT	LS-R79030B-51	LS-R79030B-510	LS-R79030B-525				
Imidazole	LS-R79230B-51	LS-R79230B-510	LS-R79230B-525				
AMPA	LS-R85130B-51	LS-R85130B-510	LS-R85130B-525				
Amine	LS-R52030B-51	LS-R52030B-510	LS-R52030B-525				
Diamine	LS-R49030B-51	LS-R49030B-510	LS-R49030B-525				
Triamine	LS-R48030B-51	LS-R48030B-510	LS-R48030B-525				
TAAcONa	LS-R69230B-51	LS-R69230B-510	LS-R69230B-525				
Cyano	LS-R38030B-51	LS-R38030B-510	LS-R38030B-525				
SCX-2	LS-R51230B-51	LS-R51230B-510	LS-R51230B-525				
SiliaCarb Ac	tivated Carbon	L					
CA	LS-CCA-51	LS-CCA-510	LS-CCA-525				
НА	LS-CHA-51	LS-CHA-510	LS-CHA-525				
VA	LS-CVA-51	LS-CVA-510	LS-CVA-525				
vw	LS-CVW-51	LS-CVW-510	LS-CVW-525				

Cyano	LS-R38030B-51	LS-R380	030B-510	LS-R38030B-525	PS-R380308	3-1612	PS-R38030	B-1625	CS-R38030	B-1650	CS-R38030B-162	100
SCX-2	LS-R51230B-51	LS-R512	230B-510	LS-R51230B-525	PS-R512308	3-1612	PS-R51230	PS-R51230B-1625		B-1650	CS-R51230B-162	100
SiliaCarb Act	ivated Carbon				<u>a</u>		1				1	
СА	LS-CCA-51	LS-CO	CA-510	LS-CCA-525	PS-CCA-1	L612	PS-CCA-	1625	CS-CCA-	1650	CS-CCA-1610	5
НА	LS-CHA-51	LS-Cł	HA-510	LS-CHA-525	PS-CHA-1	L612	PS-CHA-	1625	CS-CHA-	1650	CS-CHA-1610	2
VA	LS-CVA-51	LS-C	VA-510	LS-CVA-525	PS-CVA-1	.612	PS-CVA-	1625	CS-CVA-	1650	CS-CVA-16100)
VW	LS-CVW-51	LS-C\	/W-510	LS-CVW-525	PS-CVW-:	1612	PS-CVW-	1625	CS-CVW-	1650	CS-CVW-1610	0
Lab Scale	IOUSINGS	aborator	ry Scale E-	РАК			Bowls for L	aborato	ry Scale E-	PAK Ho	ousing	
Housing Size)	:	316L	Hastelloy	Boy	vl Leng	gth	316	L	Hast	elloy	
5 x 1 cm		LS	HK-1-S	LSHK-1-H	1 cr	n		LS-BOV	VL1-S	LS-BO	WL1-H	
5 x 10 cm		LSI	HK-10-S	LSHK-10-H	5 cr	cm LS-BOWL10-S LS-BOWL10-H		WL10-H				
5 x 25 cm		LSI	HK-25-S	LSHK-25-H	25 0	cm	m LS-BOWL25		/L25-S	LS-BO	WL25-H	
5 x 1 cm + 5 x	10 cm	LSF	IK-110-S	LSHK-110-H	_							
5 x 1 cm + 5 x	25 cm	LSF	IK-125-S	LSHK-125-H	_							
5 x 1 cm + 5 x	10 cm + 5 x 25 cm	LSH	<-11025-S	LSHK-11025-H	-							
Pilot Scale	<u>)</u> *				Commer	cial S	Scale*					_
	Housings for Pilot	Scale Fi	ixed-Bed E	-PAK	Т С Т	lousing	s for Com	nercial	Scale Fixed	-Bed E	-PAK	
Housing Size	e 316L		Haste	lloy	Housing T	уре			316L		Hastelloy	
16.5 x 12.5 cm	PSHSR-1 PSHSR-2		PSHSR PSHSR		Single Rour Single Rour			CSH	SR-50100-S	C	SHSR-50100-H	1

K Cartridges

12.5 cm

Pilot Scale - 16.5 cm diameter

PS-R10030B-1612 PS-R10030B-1625

PS-R10040B-1612 PS-R10040B-1625

PS-R51030B-1612 PS-R51030B-1625

PS-R79030B-1612 PS-R79030B-1625

PS-R79230B-1612 PS-R79230B-1625

PS-R85130B-1612 PS-R85130B-1625

PS-R52030B-1612 PS-R52030B-1625 PS-R49030B-1612 PS-R49030B-1625

PS-R48030B-1612 PS-R48030B-1625

PS-R69230B-1612 PS-R69230B-1625

25 cm

Housings for Pilot Scale Fixed-Bed E-PAK					
Housing Size	316L	Hastelloy			
16.5 x 12.5 cm	PSHSR-12-S	PSHSR-12-H			
16.5 x 25 cm	PSHSR-25-S	PSHSR-25-H			

* For PED certified housing, simply add "-PED" at the end of the product number.

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ommercial Scale - 16.5 cm diameter

CS-R10030B-1650 CS-R10030B-16100

CS-R10040B-1650 CS-R10040B-16100

CS-R51030B-1650 CS-R51030B-16100

CS-R79030B-1650 CS-R79030B-16100

CS-R79230B-1650 CS-R79230B-16100

CS-R85130B-1650 CS-R85130B-16100 CS-R52030B-1650 CS-R52030B-16100

CS-R49030B-1650 CS-R49030B-16100

CS-R48030B-1650 CS-R48030B-16100

CS-R69230B-1650 CS-R69230B-16100

100 cm

50 cm

R&D Services

Housings for Commercial Scale Fixed-Bed E-PAK							
Housing Type	316L	Hastelloy					
Single Round, 16.5 x 50 cm Single Round, 16.5 x 100 cm	CSHSR-50100-S	CSHSR-50100-H					
3-Round, 16.5 x 50 cm 3-Round, 16.5 x 100 cm	CSH3R-50100-S	CSH3R-50100-H					
7-Round, 16.5 x 50 cm 7-Round, 16.5 x 100 cm	CSH7R-50100-S	CSH7R-50100-H					
12-Round, 16.5 x 50 cm 12-Round, 16.5 x 100 cm	CSH12R-50100-S	CSH12R-50100-H					



SiliaPrep SPE Cartridges and SiliaSep Flash Cartridges

To build your SPE or Flash Cartridge Product Number, simply start with the Prefix SPE or FLH, followed by the Scavenger Number you wish your cartridge to be packed with (see page 24), followed by the Format code.

SILICYCLE -FLIS & R100368-03300 Silia Sept "330g Cardividge Man sprong property and profile

SILICYCLE-Pice Stations 10:00 Stationy "Diglications

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Examples: • SiliaPrep Thiourea, 6 mL, 500 mg = SPE-R69530B-06P

- SiliaSep Open-Top TAAcONa, 70 mL, 10 g = FLH-R69230B-70Y
- SiliaSep TAAcONa, 4 g = FLH-R69230B-ISO04

SiliaPrep SPE and SiliaSep OT Cartridges						
Formats available	Prefix	Code	Units / Box			
3 mL / 200 mg	SPE	03G	50			
3 mL / 500 mg	SPE	03P	50			
6 mL / 500 mg	SPE	06P	50			
6 mL / 1 g	SPE	06S	50			
6 mL / 2 g	SPE	06U	50			
12 mL / 2 g	SPE	12U	20			
25 mL / 5 g	FLH	20X	20			
70 mL / 10 g	FLH	70Y	16			
70 mL / 15 g	FLH	70i	16			
70 mL / 20 g	FLH	70Z	16			
150 mL / 25 g	FLH	95K	10			
150 mL / 50 g	FLH	95M	10			
150 mL / 70 g	FLH	95N	10			

	SiliaSep Flash Cartridges					
	Formats available	Prefix	Code	Units / Box		
	4 g	FLH	ISO04	2		
	12 g	FLH	ISO12	1		
	25 g	FLH	ISO25	1		
-	40 g	FLH	ISO40	1		
•	80 g	FLH	ISO80	1		
-	120 g	FLH	IS120	1		
-	220 g	FLH	IS220	1		
-	330 g	FLH	IS330	1		
-	800 g	FLH	IS750	1		
	1,600 g	FLH	I1500	1		

Resource Center

Download our Posters

SULCYCLE CO Choosing your Metal & Organic Sca SiliCycle's Most Popular Fu



Choosing your metal & organic scavenging solution

SiliCycle's most popular functionalized silicas

Take a Look at some of our Multimedia Contents





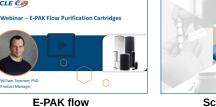
Introduction to metal and organic Scavengers

SILICYCLE 🍘

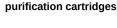
Metal scavenging using bulk SiliaMetS functionalized silica

SILICYCLE CO

Scale-up of impurity scavenging with E-PAK



Scale-up impurity scavenging with E-PAK





SILICYCLE (

Flash separation of dye mixture with SiliaSep Premium

chromatography work?

SILICYCLE (20)

How does flash



extraction (SPE)

size distribution – D50, D90 and D10 Understanding particle size The 5 steps of a solid phase

distribution - D50, D90 and D10

SILICYCLE

/hat is the sample ma loading capacity of



for functionalized silica?

What is the sample mass loading capacity of preparative TLC plates?

SiliaChrom Guard Cartridges are available in lengths of 10, 20 and 30 mm and four internal diameters (ID: 4.0, 10, 21.2 and 30 mm). The Guard Cartridges internal diameter should be the same as the HPLC column or one size smaller.

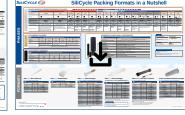
SiliaChrom Guard Cartridges

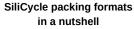


Never use a guard cartridge with a larger ID than that of the HPLC column (risk of efficiency loss).

SiliaChrom Guard Cartridges							
Formats Available (internal diameter x length in mm)	5 μm PN	10 µm PN	Units / Box	Guard Holder PN			
4.0 x 10	HPLG-K34605E-A-N010	HPLG-K34607E-A-N010	4/box	HPH-N010			
4.0 x 20	HPLG-K34605E-A-N020	HPLG-K34607E-A-N020	4/box	HPH-N020			
10 x 10	HPLG-K34605E-A-Q010	HPLG-K34607E-A-Q010	2/box	HPH-Q010			
21.2 x 10	HPLG-K34605E-A-T010	HPLG-K34607E-A-T010	1/box	HPH-T010			
30 x 10	HPLG-K34605E-A-V010	HPLG-K34607E-A-V010	1/box	HPH-V010			
30 x 20	HPLG-K34605E-A-V020	HPLG-K34607E-A-V020	1/box	HPH-V020			
30 x 30	HPLG-K34605E-A-V030	HPLG-K34607E-A-V030	1/box	HPH-V030			









Functionalized silicas and reference information



How to calculate the amount of scavenger needed



E-PAK cartridge housings, from lab to commercial scale



What are the parameters that influence scavenging efficiency?



See how easy it is working with E-PAK



Understanding Column Volume



What is the relationship between retention factor and column volume





Get a Copy of our E-Books



A collection of various case studies and application notes using scavengers



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

Synthesis





We Redefine Purity High Value Silica-based and Specialty Products for Organic Chemistry, Chromatography, and Purification

DISCOVER AND DOWNLOAD OUR BROCHURES METAL AND ORGANIC SCAVENGING SiliaMetS® - Metal Scavengers SiliaBond® – Organic Scavengers E-PAK® - Fixed Bed Flow-Through Purification Cartridges CHROMATOGRAPHY AND PURIFICATION SiliaFlash® - Irregular Silica Gels | SiliaSphere™ PC - Spherical Silica Gels SiliaBond® - Chromatographic Phases SiliaSep[™] - Flash Cartridges | SiliaPlate[™] - TLC Plates SAMPLE PREPARATION SiliaPrep[™] - Silica-based SPE Cartridges and Well Plates SiliaPrepX[™] - Polymeric SPE Cartridges and Well Plates ANALYTICAL AND PREPARATIVE CHROMATOGRAPHY SiliaSphere[™] - Spherical Silica Gels SiliaChrom® - HPLC Columns **ORGANIC SYNTHESIS** SiliaBond® - Reagents and Oxidants SiliaCat® - Heterogeneous Catalysts **PEPTIDE SYNTHESIS** Peptide Synthesis and Purification Solutions Amine Free Basing and TFA Removal **R&D SERVICES** Metal and Organic Scavenging Screenings | Organic Synthesis Chromatography and Purification | Material Science Method Development, Optimization, and Transfer **Technical Support** At SiliCycle, we are committed to providing the best technical support possible. Our worldwide Technical Support Group of highly qualified M. Sc., Ph. D. Chemists and Engineers will answer your questions and provide solutions to your most advanced chemistry and purification needs. Contact us at support@silicycle.com or call us. SiliCycle Inc. Phone: +1 418.874.0054 Email: info@silicycle.com 2500, Parc-Technologique Blvd, Toll Free +1 877.745.4292 (North America only) Quebec City (Quebec) G1P 4S6 Website: www.silicycle.com Fax: +1 418.874.0355 CANADA 🔛 🚺 Follow us: in 💓 🗗 You Tube < Overseas Offices SiliCycle Europe 8 SiliCycle India SiliCycle Shanghai india@silicycle.com +33 1 87 65 06 57 europe@silicycle.com contact@silicyclechina.com © 2023 SiliCycle Inc.

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