

Si

# SiliaBond<sup>®</sup>

## Metal Scavengers



**SILICYCLE** <sup>®</sup>  
UltraPure **SILICA GELS**

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# Introduction

Removing residual metals from post reactions can be a major issue. The toxic nature of transition metals has led to the reduction of tolerated residual concentration in active pharmaceutical ingredients (APIs) to single digit ppm. However, many important key steps, such as coupling reactions (Suzuki, Heck, Stille, Sonogashira, etc), ring closing metathesis (RCM), and hydrogenation, still require the use of metal catalysts.


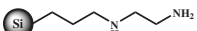
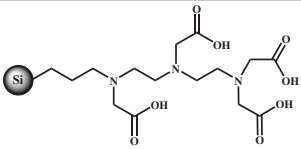
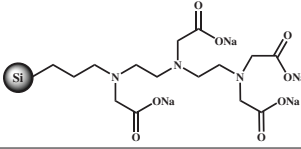
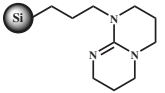
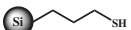
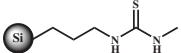
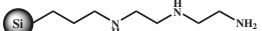
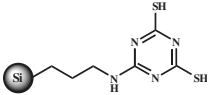
Purification of the API from remaining metal by traditional methods (chromatography, activated carbon, distillation, recrystallisation, ultrafiltration, or reverse osmosis) often leads to problems such as high costs, time consuming, low efficiency, and API losses. SiliCycle® has come with an efficient alternative: **SiliaBond Metal Scavengers**. These scavengers are made with SiliCycle's high purity silica gel which does not suffer from polymers' shortcomings, (i.e.: limited choice of solvents, excessive swelling, extensive washings, poor selectivity, and static charge).

**SiliaBond Metal Scavengers** have significantly improved the way APIs can be synthesized. Any metal containing catalyst can be used, even downstream, as there are efficient scavengers available for metals such as Pd, Pt, Sn, Ru, Rh, Ni, Cr, etc. Treatment with **SiliaBond Metal Scavengers** will selectively reduce the metal concentration without affecting the API.

This brochure includes useful information and tips on **SiliaBond Metal Scavengers** (properties and selection table), uses, experimental procedure, and results. You can take advantage of SiliCycle's expertise in metal removing capabilities by using our newest available confidential service: **Metal Scavengers Screening Service**. Our R&D team can test your solutions and find the optimal conditions to remove any leftover metal catalyst. Contact us for more details.

# SiliaBond Metal Scavengers Product Line

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

| SiliaBond Metal Scavengers |                 |  |
|----------------------------|-----------------|--|
| Name                       | Product Number* | Structure  |
| SiliaBond Amine            | R52030B         |    |
| SiliaBond Diamine          | R49030B         |    |
| SiliaBond TAAcOH           | R69030B         |   |
| SiliaBond TAAcONa          | R69230B         |  |
| SiliaBond TBD              | R68530B         |  |
| SiliaBond Thiol            | R51030B         |  |
| SiliaBond Thiourea         | R69530B         |  |
| SiliaBond Triamine         | R48030B         |  |
| SiliaBond DMT              | R79030B         |  |

AVAILABLE  
SOON

\* Formats: 10 g, 25 g, 100 g, 500 g, 1 kg, 5 kg, 10 kg, 25 kg

# SiliaBond Metal Scavenger Kit

For your convenience, SiliCycle has combined the most popular metal scavengers into a kit for screening purposes.

## SiliaBond Metal Scavenger Kit

Bulk format: K30730B (5 g, 10 g, 25 g, 100 g)

SiliaPrep SPE Cartridges: SPE-K30730B-06U (2 g/6 mL)  
(10 cartridges of each product)

### Products Included

|                   |                    |
|-------------------|--------------------|
| SiliaBond Diamine | SiliaBond Thiol    |
| SiliaBond TAAcOH  | SiliaBond Thiourea |
| SiliaBond TAAcONa | SiliaBond Triamine |

## SiliaBond Metal Scavengers Characteristics

SiliaBond Metal Scavengers are made of SiliaFlash® F60 (40-63  $\mu\text{m}$ , 60 Å, 500  $\text{m}^2/\text{g}$ ) and endcapped to avoid non-specific binding (undesirable interactions with the free silanols). Therefore, they present the following characteristics:

- **No leaching (chemical stability):** no API contamination by the metal scavenger.  
Each SiliaBond product manufactured by SiliCycle is submitted to a very rigorous quality control in order to provide customers with default-free products and insure 100% satisfaction. The quality control analysis of the final material includes loading and reactivity determination, loss on drying as well as leachables and extractables analysis ( $\geq 99.995\%$ ). Certificates of analysis are sent with each SiliaBond Metal Scavenger product.
- **High selectivity:** total recovery of the API.
- **Very good metal affinity:** efficient for a wide range of metal catalysts.
- **Solvent compatibility:** can be used in any solvent, aqueous (pH 2 to 12) and organic.
- **Fast kinetic:** even at room temperature.
- **Easily scalable:** from mg up to multi-ton scale.
- **Thermally and mechanically stable:** works well with overhead stirring and can withstand very high temperatures; suitable for use in microwave synthesizers.
- **Ease of use:** no swelling or static charge.
- **Flexible formats:** amenable to use in SiliaSep & SiliaPrep Cartridges.
- **Cost efficient:** low cost per gram of metal scavenged.
- **Controlled loading:** consistent and accurate loading insure lot-to-lot reproducibility.

As SiliaBond Metal Scavengers are being used by many pharmaceutical industries and biotechnologies, SiliCycle has implemented a quality control procedure regarding possible leaching. SiliaBond products have been used in typical experimental conditions and under "drastic" conditions, and then analyzed by GC/MS and ICP-OES for leaching quantification. The solution must be free of contaminants for the product to pass the quality control test. Contact SiliCycle to get experiment results.

# SiliaBond Metal Scavengers Selection Table

When selecting a metal scavenger, every component present must be considered; metal catalyst, solvent, residual reagents, by-products, structure of the API (or molecule of interest), and temperature have their importance. SiliCycle has created the selection table shown below to help you select the more efficient scavenger for a specific metal. However we highly recommend to test various **SiliaBond Metal Scavengers** with your solution since some parameters may affect the efficiency of the scavenging.

**SiliaBond Metal Scavenger Selection Table**

| Scavenger   | Ag | Cd | Cr | Co | Cu | Fe | Hg | Ni | Pb | Pd (II) | Pd (0) | Pt | Rh (I) | Rh (II) | Rh (III) | Ru (II) | Ru (IV) | Sn | V | Zn |
|-------------|----|----|----|----|----|----|----|----|----|---------|--------|----|--------|---------|----------|---------|---------|----|---|----|
| Si-Amine    |    | ■  | ■  | ■  | ■  |    | ■  |    | ■  | ■       |        | ■  | ■      | ■       |          |         |         |    |   | ■  |
| Si-Diamine  |    | ■  | ■  | ■  | ■  | ■  | ■  | ■  | ■  | ■       | ■      | ■  | ■      | ■       |          | ■       | ■       |    |   | ■  |
| Si-TAAcOH   |    |    |    |    |    | ■  |    | ■  |    | ■       | ■      |    | ■      | ■       |          |         |         | ■  |   |    |
| Si-TAAcONa  |    |    | ■  | ■  | ■  | ■  |    | ■  |    | ■       | ■      |    | ■      | ■       | ■        | ■       | ■       | ■  |   | ■  |
| Si-TBD      |    |    | ■  | ■  |    | ■  |    |    |    | ■       |        |    |        |         |          |         |         |    |   |    |
| Si-Thiol    | ■  |    |    |    | ■  |    | ■  |    | ■  | ■       | ■      |    | ■      | ■       | ■        |         | ■       | ■  |   |    |
| Si-Thiourea | ■  |    |    |    |    |    | ■  |    |    | ■       | ■      |    | ■      | ■       | ■        |         |         | ■  | ■ | ■  |
| Si-Triamine |    |    | ■  | ■  | ■  | ■  |    | ■  | ■  | ■       | ■      | ■  |        |         |          |         |         |    |   | ■  |
| Si-DMT      |    |    |    |    |    |    |    | ■  |    | ■       | ■      | ■  | ■      | ■       | ■        | ■       | ■       |    |   |    |

■ Preferred scavengers   ■ Scavenges   ■ Reported in the literature or by customers

Visit our website to see metal scavenger video.

## Typical Experimental Conditions

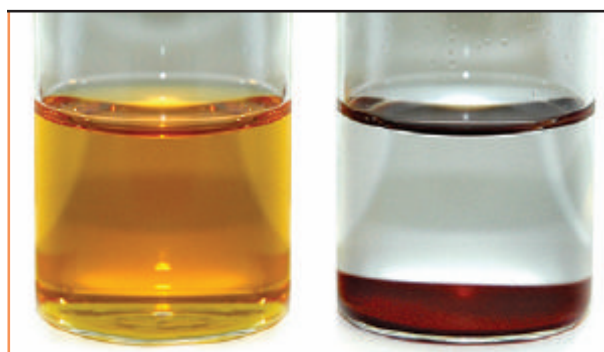
### STEP 1: SCREENING

To find out which **SiliaBond Metal Scavenger** will better suit your need, we recommend performing some screening (on small scale) using the **SiliaBond Metal Scavenger Kit** (Product # K30730B) to quickly determine which scavenger presents the highest efficiency and make experiment optimization from there.

## TYPICAL EXPERIMENTAL PROCEDURE

Prepare six vials containing the same quantity of the solution to be treated with the **SiliaBond Scavengers** included in the kit.

1. For each **SiliaBond Metal Scavenger**, calculate the amount needed (4-8 eq.) in respect to the residual metal concentration. (See "Calculation of the amount of scavenger to use" section.)
2. Add the **SiliaBond** to the solution and stir for one hour at room temperature.
3. Filter off the **SiliaBond** using a fritted funnel or filtration device, and wash the scavenger with additional solvent for total recovery of the API.
4. Analyze the residual metal concentration of each vial to identify the most efficient scavenger (the one with the lowest residual metal concentration). You can choose more than one scavenger.
5. Follow the optimization steps with the **SiliaBond Metal Scavenger** identified in #4.



Note: the scavenging progress can be estimated by looking at the color of the solution as demonstrated in the figure. When the scavenging is almost completed, the solution is colorless and **SiliaBond Metal Scavenger** becomes coloured. If all the samples are still coloured, let them react for a longer time (ex.: 4 hours) and/or use more equivalents (at least 8 eq.).

## STEP 2: OPTIMIZATION

If, upon completion of the screening procedure, the scavenging is not completed and you are not satisfied with the scavenging efficiency or you wish to reduce the number of equivalents and the reaction time, the following steps should be undertaken:

- Increase the temperature of the scavenging reaction
- Modify the number of molar equivalents of the **SiliaBond Metal Scavenger**
- Change the reaction time
- Try a different and/or a mixture of **SiliaBond Metal Scavengers**

These parameters can be changed one at a time or simultaneously to improve the metal removal efficiency. It is important to underline that these scavengers are stable and work in any solvent. All these parameters (temperature, number of equivalents, reaction time and solvent) were investigated in the scavenging study presented in this brochure.

### STEP 3: BULK (OR SPE) PROCEDURE

Once the experimental conditions have been optimized (solvent, number of equivalents, temperature, and time), add the **SiliaBond Metal Scavenger** to the solution and stir mechanically at selected temperature for the determined period of time. Filter off the scavenger using a filtration device and wash the scavenger with solvent for total API recovery.

Alternatively, a packed cartridge (**SPE** or **Flash cartridge**) can be employed for metal removal. We recommend using at least 4 equivalents of the scavenger and eluting by gravity. At the end, rinse the cartridge with 2-3 column volumes.



### CALCULATION OF THE AMOUNT OF SILIABOND METAL SCAVENGER TO USE

To get an effective metal removal, the amount of **SiliaBond Metal Scavenger** used is very important. You can determine by two different ways how much scavenger will be needed; from the residual concentration (more accurate method) or from the amount of metal catalyst used, (when the residual metal concentration is unknown).

#### From residual metal concentration (ppm)

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

##### Data needed:

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

1. Determine the amount (g) of palladium to be scavenged

$$\text{Pd (mg)} = \frac{500 \text{ ppm} \times 800 \text{ g of material}}{1000} = 400 \text{ mg of Pd in 800g of material}$$

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

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

$$\text{Scavenger (g)} = \frac{3.76 \text{ mmol of Pd}}{1.2 \text{ mmol/g scavenger}} = 3.13 \text{ g of scavenger (1 eq)}$$

To scavenge 400 mg of palladium, 3.13 g of **SiliaBond Thiol** is needed if using only one equivalent. However, it is highly recommend trying a minimum of 4 equivalents at first. In this case, the amount of **SiliaBond Thiol** will be 4 times higher (4 x 3.13 g = 12.40 g).

#### From amount of metal catalyst used

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

##### Data needed:

- **Amount of metal catalyst used:** Ex. 10 g of Pd(PPh<sub>3</sub>)<sub>4</sub>
- **Metal catalyst molecular weight:** Pd(PPh<sub>3</sub>)<sub>4</sub> = 1155.56 g/mol

1. Determine the amount (g) of palladium to be scavenged

$$\text{Pd (mmol)} = \frac{10 \text{ g of Pd(PPh}_3)_4 \times 1000}{1155.56 \text{ g/mol}} = 8.65 \text{ mmol of Pd (max to be scavenged)}$$

The amount of **SiliaBond Thiol** to be used can then be determined as stated above (see 2. Calculate the amount of scavenger to use).

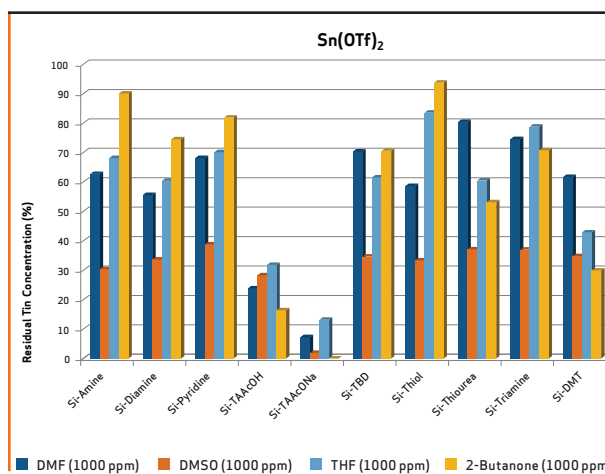
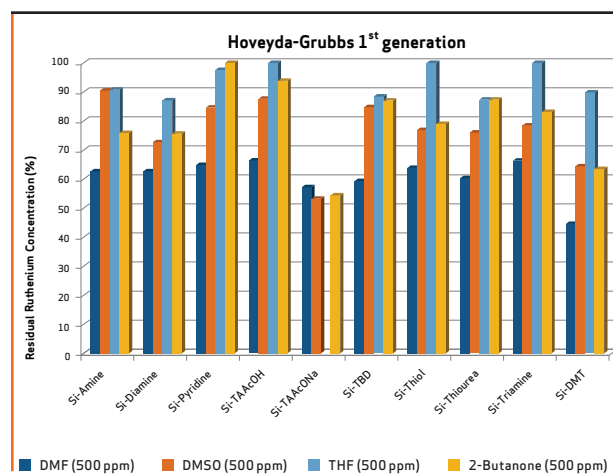
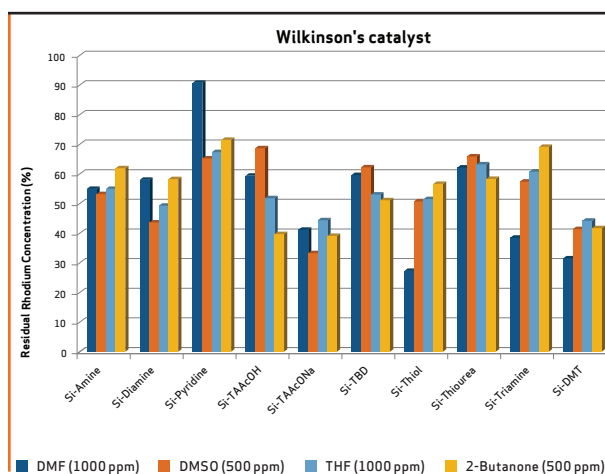
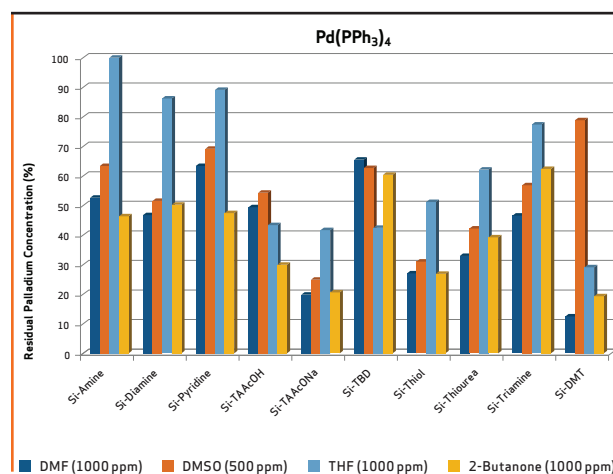
In this particular case, 1 equivalent of **SiliaBond Thiol** corresponds to 7.20 g.

# Metal Scavenging Study

A metal scavenging study using **SiliaBond Metal Scavengers** products was conducted on various metal catalysts commonly used in organic synthesis. In this study, we examined the effectiveness of using silica-bound metal scavengers to remove metals. Various parameters were studied for their influence on the scavengers' robustness as well as to establish the best conditions to bring the level of metal down to an acceptable level for the pharmaceutical industry (i.e.: solvent, number of equivalents, temperature, reaction time, and nature of the catalyst).

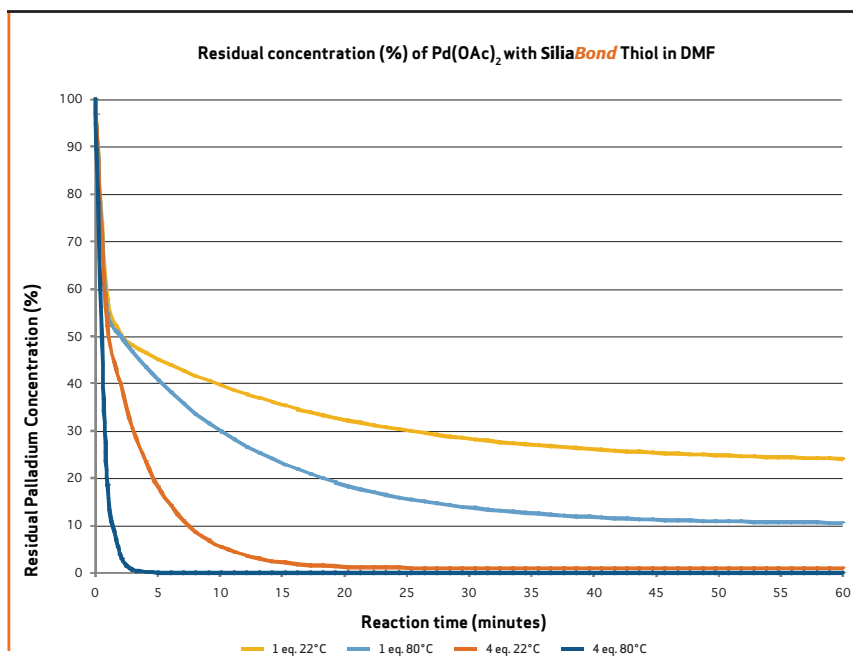
## SOLVENT EFFECT ON SCAVENGING EFFICIENCY

**SiliaBond Metal Scavengers** can safely be used in any organic and aqueous solvents. However, the nature of the solvent does sometimes influence the scavenging efficiency, as demonstrated in the graphs below using one equivalent of the **SiliaBond Metal Scavenger** for one hour at 50°C.



## REACTION TIME AND NUMBER OF EQUIVALENTS EFFECT ON SCAVENGING EFFICIENCY

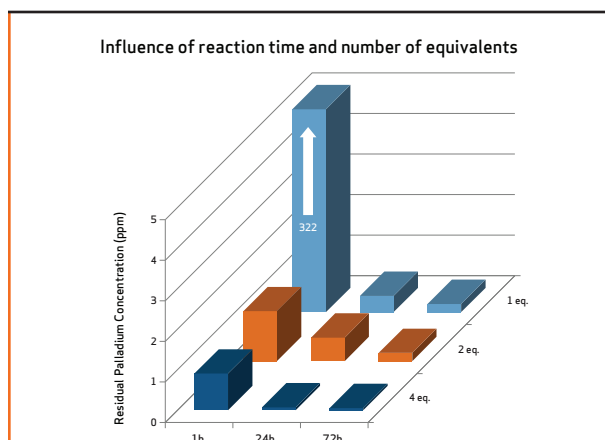
The fast scavenging kinetic of **SiliaBond Metal Scavengers** allows the reduction of the residual metal concentration to very low ppm in a few minutes. However, various parameters affect the kinetic. The influence of the reaction time, the temperature, and the number of equivalents used are shown in the graphs below.



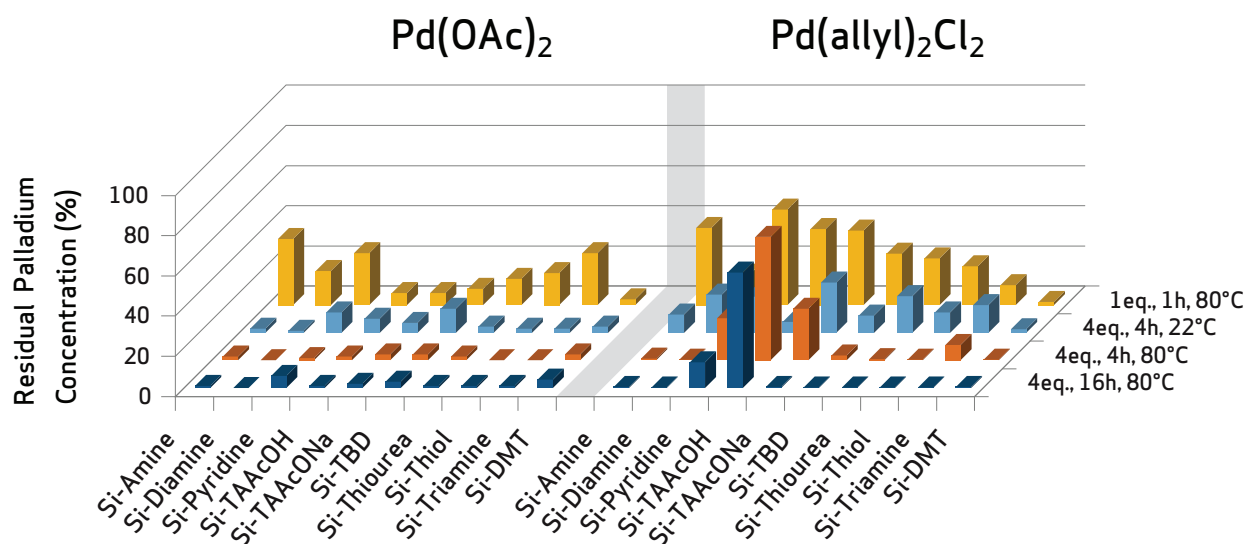
Conclusion: Number of equivalents and temperature both have an effect on the scavenging efficiency. As the temperature is increased, the residual amount of Pd decreases dramatically.

Please note that it is also possible to get low residual concentration (<1 ppm) with only one equivalent of scavenger, for example by using THF as a solvent (see graph at right).

Conditions: Pd(OAc)<sub>2</sub>, THF, **Si-Thiol**, RT.



# Scavenging Palladium Catalysts



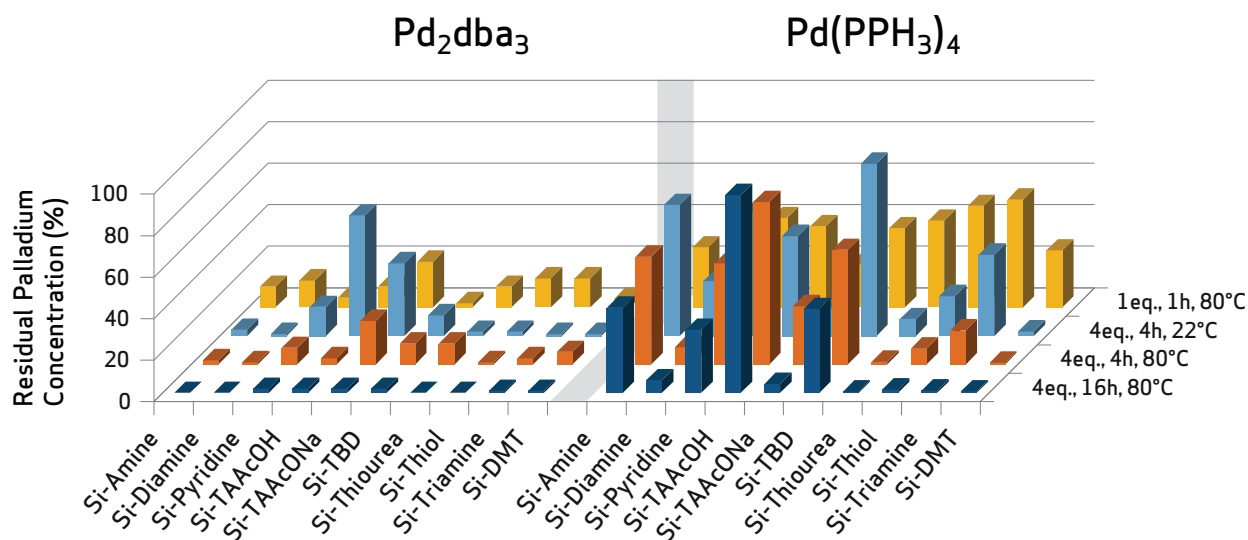
| Pd(OAc) <sub>2</sub> : Residual Palladium Concentration |                 |          |
|---|-----------------|----------|
| Scavengers  | Conditions      | Pd (ppm) |
| SiliaBond Amine   | 4eq., 4h, 80°C  | 13.5     |
|   | 4eq., 16h, 80°C | 4.1      |
| SiliaBond Diamine                                       | 4eq., 4h, 22°C  | 10.8     |
|   | 4eq., 4h, 80°C  | 2.9      |
|   | 4eq., 16h, 80°C | 3.8      |
| SiliaBond TAAcOH  | 4eq., 4h, 80°C  | 12.5     |
|   | 4eq., 16h, 80°C | 10.5     |
| SiliaBond Thiol   | 4eq., 4h, 80°C  | 12.7     |
|   | 4eq., 16h, 80°C | 6.27     |
| SiliaBond Thiourea                                      | 4eq., 4h, 80°C  | < 1      |
| SiliaBond Triamine                                      | 4eq., 4h, 80°C  | < 1      |

Initial concentration: 1 000 ppm in DMF

| Pd(allyl) <sub>2</sub> Cl <sub>2</sub> : Residual Palladium Concentration |                 |          |
|---|-----------------|----------|
| Scavengers  | Conditions      | Pd (ppm) |
| SiliaBond Amine   | 4eq., 4h, 80°C  | 8.6      |
|   | 4eq., 16h, 80°C | 3.9      |
| SiliaBond Diamine   | 4eq., 4h, 80°C  | 3.6      |
|   | 4eq., 16h, 80°C | 3.1      |
| SiliaBond TAAcONa   | 4eq., 16h, 80°C | 4.0      |
| SiliaBond TBD   | 4eq., 16h, 80°C | 3.0      |
| SiliaBond Thiol   | 4eq., 4h, 80°C  | 5.1      |
|   | 4eq., 16h, 80°C | 2.7      |
| SiliaBond Thiourea  | 4eq., 4h, 80°C  | 3.6      |
|   | 4eq., 16h, 80°C | 2.6      |
| SiliaBond Triamine  | 4eq., 16h, 80°C | 2.5      |
| SiliaBond DMT   | 4eq., 4h, 80°C  | 2.5      |

Initial concentration: 1 000 ppm in DMF

# Scavenging Palladium Catalysts (CONTINUED)



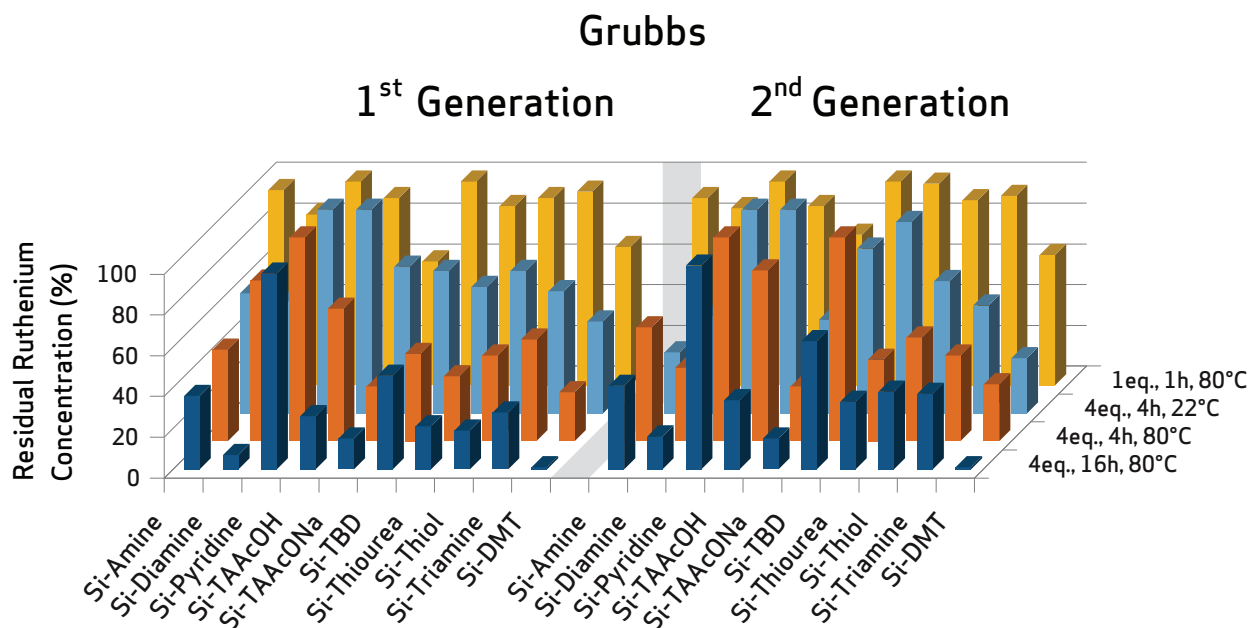
| Pd <sub>2</sub> (dba) <sub>3</sub> : Residual Palladium Concentration |                 |          |
|---|-----------------|----------|
| Scavengers  | Conditions      | Pd (ppm) |
| SiliaBond Amine   | 4eq., 16h, 80°C | 3.4      |
| SiliaBond Diamine   | 4eq., 4h, 22°C  | 11.6     |
|   | 4eq., 4h, 80°C  | 6.3      |
|   | 4eq., 16h, 80°C | 1.9      |
| SiliaBond Thiol   | 4eq., 16h, 80°C | 2.5      |
| SiliaBond Thiourea  | 4eq., 4h, 80°C  | 5.3      |
|   | 4eq., 16h, 80°C | 1.8      |
| SiliaBond Triamine  | 4eq., 16h, 80°C | 4.6      |
| SiliaBond DMT   | 4eq., 4h, 22°C  | 8.6      |

Initial concentration: 1 000 ppm in DMF

| Pd(PPh <sub>3</sub> ) <sub>4</sub> : Residual Palladium Concentration |                 |          |
|---|-----------------|----------|
| Scavengers  | Conditions      | Pd (ppm) |
| SiliaBond Thiol   | 4eq., 4h, 80°C  | 12.8     |
|   | 4eq., 16h, 80°C | 5.9      |
| SiliaBond Thiourea  | 4eq., 16h, 80°C | 16.7     |
| SiliaBond Triamine  | 4eq., 16h, 80°C | 11.6     |
| SiliaBond DMT   | 4eq., 4h, 80°C  | 7.6      |

Initial concentration: 1 000 ppm in DMF

# Scavenging Ruthenium Catalysts



Grubbs 1<sup>st</sup>: Residual Ruthenium Concentration

| Scavengers        | Conditions      | Ru (ppm) |
|-------------------|-----------------|----------|
| SiliaBond Diamine | 4eq., 16h, 80°C | 26.9     |
| SiliaBond TAAcONa | 4eq., 4h, 80°C  | 33.0     |
| SiliaBond DMT     | 4eq., 4h, 80°C  | 20.9     |
|                   | 4eq., 16h, 80°C | 3.8      |

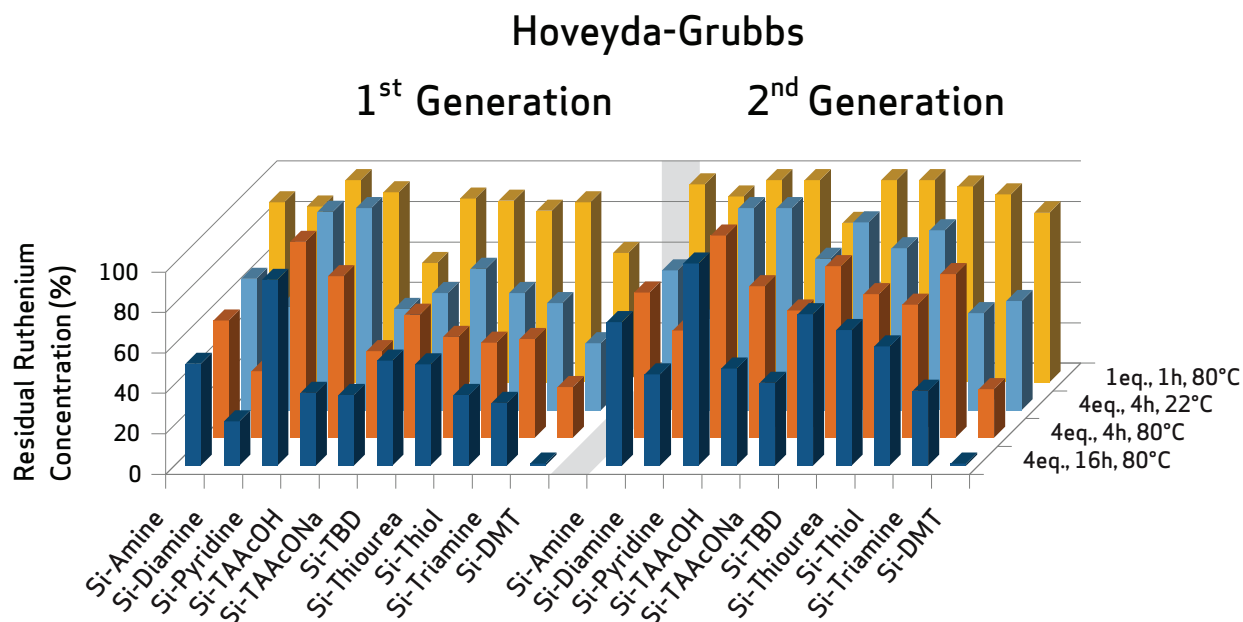
Initial concentration: 500 ppm in DMF

Grubbs 2<sup>nd</sup>: Residual Ruthenium Concentration

| Scavengers        | Conditions      | Ru (ppm) |
|-------------------|-----------------|----------|
| SiliaBond TAAcONa | 4eq., 4h, 80°C  | 33.8     |
| SiliaBond DMT     | 4eq., 4h, 80°C  | 40.8     |
|                   | 4eq., 16h, 80°C | 5.1      |

Initial concentration: 500 ppm in DMF

# Scavenging Ruthenium Catalysts (CONTINUED)



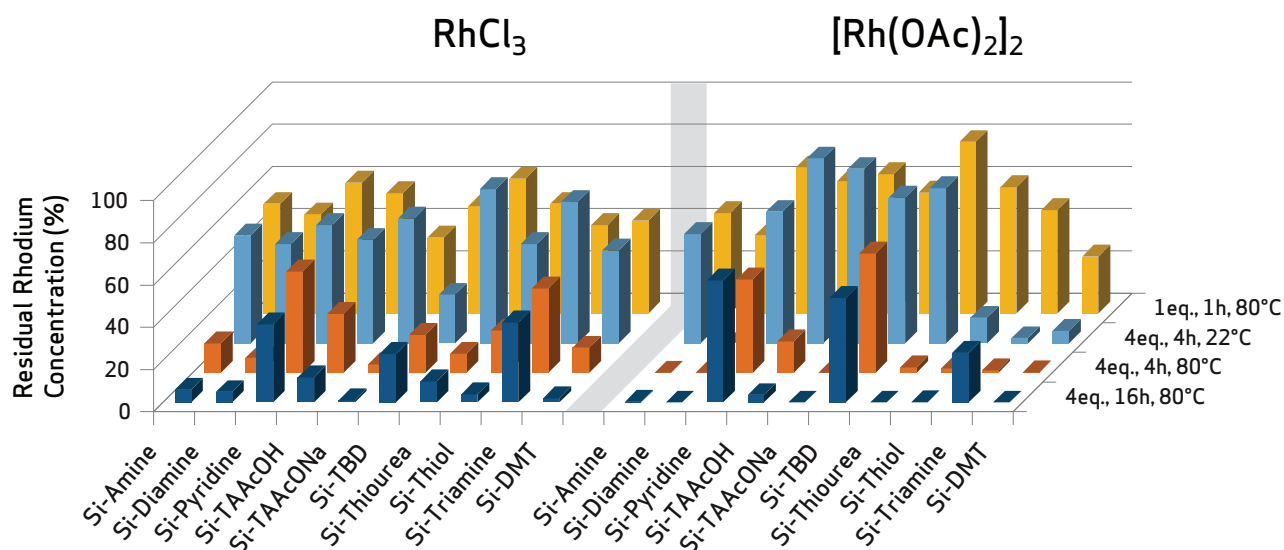
| Hoveyda-Grubbs 1 <sup>st</sup> : Residual Ruthenium Concentration |                  |          |
|---|------------------|----------|
| Scavengers  | Conditions       | Ru (ppm) |
| SiliaBond DMT   | 4 eq., 4h, 80°C  | 24.8     |
|   | 4 eq., 16h, 80°C | 7.1      |

Initial concentration: 500 ppm in DMF

| Hoveyda-Grubbs 2 <sup>nd</sup> : Residual Ruthenium Concentration |                  |          |
|---|------------------|----------|
| Scavengers  | Conditions       | Ru (ppm) |
| SiliaBond DMT   | 4 eq., 4h, 80°C  | 19.1     |
|   | 4 eq., 16h, 80°C | 6.1      |

Initial concentration: 500 ppm in DMF

# Scavenging Rhodium Catalysts



| RhCl <sub>3</sub> : Residual Rhodium Concentration |                 |          |
|--|-----------------|----------|
| Scavengers   | Conditions      | Rh (ppm) |
| SiliaBond TAAcONa                                  | 4eq., 16h, 80°C | 8.5      |
| SiliaBond Thiourea                                 | 4eq., 16h, 80°C | 28.1     |
| SiliaBond DMT                                      | 4eq., 16h, 80°C | 15.5     |

Initial concentration: 1 000 ppm in DMF

| [Rh(OAc) <sub>2</sub> ]: Residual Rhodium Concentration |                 |          |
|---|-----------------|----------|
| Scavengers  | Conditions      | Rh (ppm) |
| SiliaBond Amine   | 4eq., 16h, 80°C | < 1      |
| SiliaBond Diamine                                       | 4eq., 4h, 22°C  | 3.3      |
|   | 4eq., 4h, 80°C  | < 1      |
|   | 4eq., 16h, 80°C | < 1      |
| SiliaBond TAAcONa                                       | 4eq., 16h, 80°C | < 1      |
| SiliaBond Thiol   | 4eq., 4h, 80°C  | < 1      |
| SiliaBond Thiourea                                      | 4eq., 4h, 22°C  | 1.2      |
| SiliaBond Triamine                                      | 4eq., 4h, 80°C  | 12.6     |
|   | 4eq., 16h, 80°C | 9.0      |
| SiliaBond DMT   | 4eq., 4h, 80°C  | 1.9      |
|   | 4eq., 16h, 80°C | < 1      |

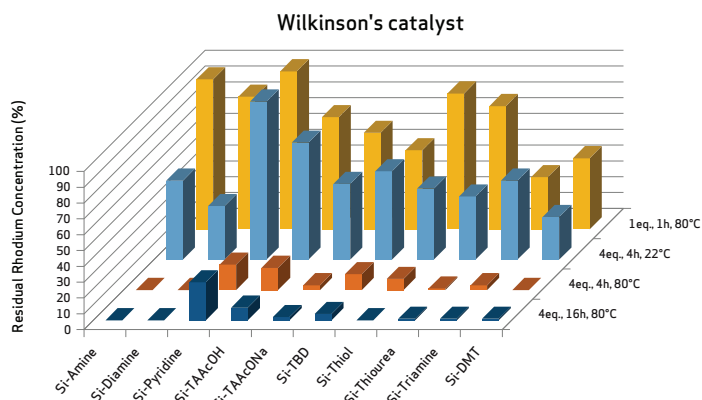
Initial concentration: 1 000 ppm in DMF

# Scavenging Rhodium Catalysts (CONTINUED)

Wilkinson's catalyst: Residual Rhodium Concentration

| Scavengers         | Conditions      | Rh (ppm) |
|--------------------|-----------------|----------|
| SiliaBond Amine    | 4eq., 4h, 80°C  | < 1      |
| SiliaBond Diamine  | 4eq., 4h, 80°C  | 2.2      |
|                    | 4eq., 16h, 80°C | 2.2      |
| SiliaBond Thiol    | 4eq., 16h, 80°C | < 1      |
| SiliaBond Thiourea | 4eq., 4h, 80°C  | 5.4      |
| SiliaBond Triamine | 4eq., 16h, 80°C | 10.5     |
| SiliaBond DMT      | 4eq., 4h, 80°C  | < 1      |
|                    | 4eq., 16h, 80°C | < 1      |

Initial concentration: 500 ppm in DMF

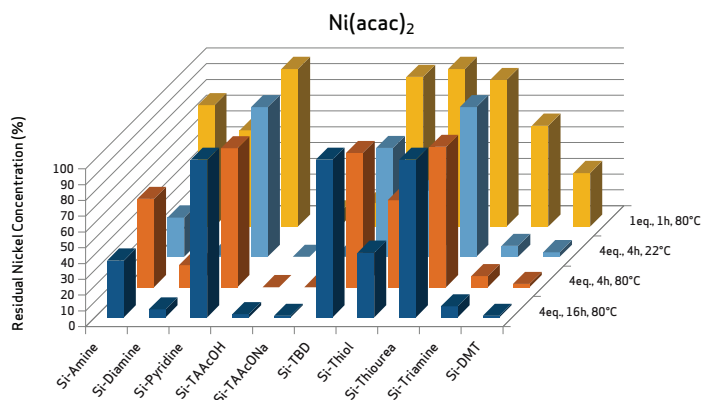


# Scavenging a Nickel Catalyst

Ni(acac)<sub>2</sub>: Residual Nickel Concentration

| Scavengers        | Conditions      | Ni (ppm) |
|-------------------|-----------------|----------|
| SiliaBond Diamine | 4eq., 4h, 22°C  | 8.8      |
| SiliaBond TAAcOH  | 4eq., 4h, 22°C  | < 1      |
| SiliaBond TAAcONa | 4eq., 4h, 22°C  | < 1      |
| SiliaBond DMT     | 4eq., 16h, 80°C | 11.1     |

Initial concentration: 500 ppm in DMF

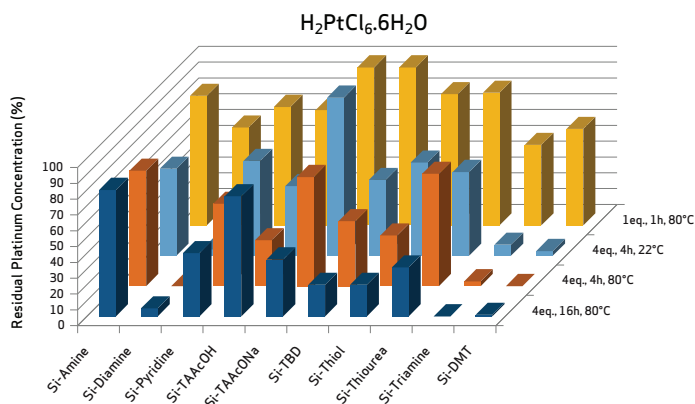


# Scavenging a Platinum Catalyst

$H_2PtCl_6 \cdot 6H_2O$ : Residual Platinum Concentration

| Scavengers         | Conditions      | Pt (ppm) |
|--------------------|-----------------|----------|
| SiliaBond Diamine  | 4eq., 4h, 80°C  | 2.2      |
| SiliaBond Triamine | 4eq., 16h, 80°C | 2.3      |
| SiliaBond DMT      | 4eq., 4h, 80°C  | < 1      |

Initial concentration: 1 000 ppm in DMF

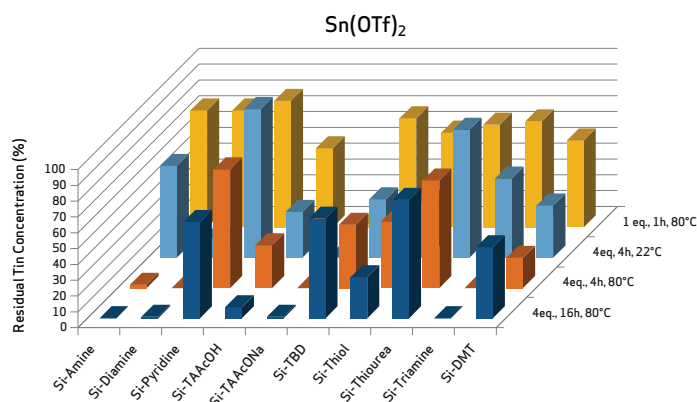


# Scavenging a Tin Catalyst

$Sn(OTf)_2$ : Residual Tin Concentration

| Scavengers         | Conditions      | Sn (ppm) |
|--------------------|-----------------|----------|
| SiliaBond Amine    | 4eq., 16h, 80°C | 2.3      |
| SiliaBond Diamine  | 4eq., 4h, 80°C  | < 1      |
| SiliaBond TAAcONa  | 4eq., 4h, 22°C  | < 1      |
|                    | 4eq., 4h, 80°C  | < 1      |
| SiliaBond Triamine | 4eq., 4h, 80°C  | < 1      |

Initial concentration: 1 000 ppm in DMF



# Flexible Formats: SiliaSep & SiliaPrep Cartridges

SiliaBond Metal Scavengers are amenable to use in different formats. For your convenience, SiliCycle offers SiliaBond Metal Scavengers in SiliaSep Flash Cartridges and SiliaPrep SPE Cartridges formats (see tables below for standard available cartridge formats).



## SiliaSep Flash Cartridges

| SiliaSep<br>(Isco™ compatible) | SiliaSep BT<br>(Biotage™ i compatible) | SiliaSep OT<br>(FlashMaster™ compatible) |                | SiliaSep BU<br>(Büchi™ compatible) |
|--------------------------------|--|--|----------------|------------------------------------|
|                                |  | silica weights                           | column volumes |                                    |
| 4 g                            | 12S                                    |  |                | 12S                                |
| 12 g                           | 12M                                    |  |                | 12M                                |
| 25 g                           | 40S                                    | 2 g                                      | 12 ml          | 40S                                |
| 40 g                           | 40M                                    | 5 g                                      | 25 ml          | 40M                                |
| 80 g                           | 40L                                    | 10 g                                     | 70 ml          | -                                  |
| 120 g                          | 65                                     | 20 g                                     | 150 ml         | -                                  |
| 220 g                          | 75S                                    | 25 g                                     | 276 ml         | -                                  |
| 330 g                          | 75M                                    | 50 g                                     | 377 mL         | -                                  |
| 750 g                          | 75L                                    | 70 g                                     | -              | -                                  |
| 1500 g                         | -                                      | 100 g                                    | -              | -                                  |
| -                              | -                                      | 200 g                                    | -              | -                                  |

# Flexible Formats: SiliaSep & SiliaPrep Cartridges (CONTINUED)

## SiliaPrep SPE Cartridges

(VOLUMES AND SORBENT WEIGHTS)\*

| 1 mL   | 3 mL   | 6 mL<br>(8 mL) | 12 mL<br>(15 mL) | 25 mL<br>(20 mL) |
|--------|--------|----------------|------------------|------------------|
| 50 mg  | 200 mg | 500 mg         | 2 g              | 5 g              |
| 100 mg | 500 mg | 1 g            |                  |                  |

\* Please refer to SiliaSep OT for > 25mL SPEs. Custom formats are available upon request.



**SiliaBond Metal Scavengers** are unique. These products have significantly changed how chemists can prepare API. Any synthetic pathway is now possible, even the ones using metal catalyst. Treatment with **SiliaBond Metal Scavengers** will lower the residual metal concentration to single digit ppm as demonstrated by the scavenging study. Various Palladium, Platinum, Rhodium, Ruthenium, Nickel, and Tin complexes were efficiently scavenged using different experimental conditions.

SiliCycle offers **SiliaBond Metal Scavengers** in **SPE** and **Flash cartridges** formats as well as a confidential **Metal Scavengers Screening Service** to take advantage of our expertise in metal removing.



**SOLUTION PROVIDER  
for metal removal!**

**We can help you. Contact us!**

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