Metal Scavenger User Guide

Case Studies | Methods | Ordering Information

Biotage®

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What are Metal Scavengers?

Biotage supported resin and silica based metal scavengers are effective reagents for the removal of trace metals (such as PGM's) from catalyzed reactions to final API's. This technology can be applied to a variety of different industries, from pharmaceutical to fine chemical; from agrochemical to water and waste treatment.

Figure 1. Biotage® Metal Scavenging Tool Kit.

Tool Kit of Metal Scavengers

- Now incorporating the industry leading universal scavenger for metal scavenging / removal work, Biotage® MP-TMT
- Higher metal affinity – low Kd(Pd) relative to API Kd(Pd) – excellent metal scavenging
- Fast kinetics / filtration at room temperature – enhanced by heating
- Diverse solid supports – silica and polystyrene backbones
- Work Flow Compatible – generic, use in diverse conditions
- Minimal loss of API – less / no non-specific binding
- Higher purity of final product – no leachables
- Short development time – faster method optimization

Solid supported metal scavengers have been shown to be highly selective, and also offer cost-effective scavenging, enabling chemists to reach metal purity goals[1]. Precious metals such as Pd, Pt, Ru and Rh in organometallic catalysts are becoming more widespread in industrial synthesis due to atom economy and green credentials[2,3]. These PGM group catalyzed reactions are commonly used in the manufacture of active pharmaceutical ingredients (API's) and fine chemicals. Achieving purity levels for metal contamination is becoming increasingly challenging, with limits of 5 ppm (ingested)*, and 1 ppm (parenteral) API now the benchmarked standard[4]. This poses particular difficulties in downstream purification of Pd based chemistries.

Traditional methods to remove metals include chromatography, activated carbon, extraction, distillation and recrystallization but these can offer poor selectivity and lead to high API loss. Biotage has assembled an optimized tool kit (K-MS-2) to facilitate the process of metal screening and method development.

Biotage are leaders in metal scavenging in a variety of different application areas.

Features

- Mechanically robust, and may be stirred vigorously without breakdown
- Bioanalytical grade silica or ultra clean polystyrene polymers
- Can also be added in batch processes and stirred
- Optimum flow characteristics as fixed beds
- May be stored in closed containers at RT, or longer term in a cool dry (4 °C) environment

* at time of writing

References

1. Welch, C.J.; Albaneze-Walker, J.; Leonard, W.R.; Biba, M.; DaSilva, J.; Henderson, D.; Laing, B.; Mathre, D.J.; Spencer, S.; Bu, X.; Wang, T.; *Org. Proc. Res. Dev.* **2005**, 9, 198–205
2. King, A. O.; Yasuda, N. *Topics in Organometallic Chemistry*, **2004**, 6, 205–245.
3. Carey, J.S.; Laffan, D.; Thompson, C.; Williams, M.T. Org. *Biolmol. Chem.* **2006**, 4, 2337–2347.
4. European Agency for the Evaluation of Medicinal Products, *Guidelines on the Specification Limits for Residues of Metal Catalysts or Metal Reagents*, 2008. Accessed December **2009**: http://www.emea.europa.eu/pdfs/human/swp/444600en.pdf

Metal Scavenger Selection Guide

Product	Particle Size (µm)	Ligand-M Strength	Compatible API's	Batch Performance	Fixed Bed (Cartridge) Performance	Main Metals Scavenged	Typical Catalysts	Typical Reactions	Other Metals Scavenged	Solvent Compatibility	Typical Load vs. API (flow)	Typical Load vs. API (batch)
MP-TMT	150–350	■	ABN*	■	■	Pd	$Pd(OAc)_2$, $Pd(PPh_3)Cl_2$, $Pd(PPh_3)_4$, $Pd(dppf)Cl_2$	Suzuki, Heck, Sonogashira, Ulman etc.	Rh, Ru, Pt, Ni	All¨	n/a	2–20 wt%
Si-TMT	40–63	■	ABN*	■	■	Pd	$Pd(OAc)_2$, $Pd(PPh_3)Cl_2$, $Pd(PPh_3)_4$, $Pd(dppf)Cl_2$	Suzuki, Heck, Sonogashira, Ulman etc.	Rh, Ru,Pt, Ni	All¨	5–10 eq	2–20 wt%
Si-Thiol	40–63	■	ABN*	■	■	Pd	$Pd(OAc)_2$, $Pd(PPh_3)Cl_2$, $Pd(PPh_3)_4$, $Pd(dppf)Cl_2$	Suzuki, Heck, Sonogashira, Ulman etc.	Rh, Ru, Pt, Ni	All¨	5–10 eq	2–20 wt%
SCX-2	40–63	■	AN**	■	■	Fe	M˘(I)	Various	M˘(I)	All¨	5–10 eq	2–20 wt%
Si-Trisamine	40–63	■	BN***	■	■	Pd	$Pd(OAc)_2$, $Pd(PPh_3)Cl_2$, $Pd(PPh_3)_4$, $Pd(dppf)Cl_2$ M(II)	Suzuki, Heck, Sonogashira, Ulman etc.	Ni, Sn, Fe, Cu, Zn, M(II)	All¨	5–10 eq	2–20 wt%

■ Excellent/Preferred
■ Very Good
■ Good

*ABN for Acids, bases, neutral products, 2 > pH > 10, extremes possible for short times
**AN for Acids, neutral products, 2 > pH > 10, extremes possible for short times
***BN for Bases, neutral products, 2 > pH > 10, extremes possible for short times
˘M = Transition metal
¨All common laboratory and process solvents

Advantages of Biotage Metal Scavengers

No Leaching	Used in final/late stage API synthesis, each product supplied with full QC ISO certification, detailing extractables, LOD, elemental analysis, FTIR, physical inspection, particle size, MSDS, TSE statement supplied as standard. Further information in support of regulatory compliance available on request.
High Selectivity	Excellent extraction of metal
Excellent Range of Metal Affinities	Tuneable by metal or oxidation state, variety of scavengers as standard
Many Oxidation States Covered	Can be applied to mother liquors or extracted product of reaction
Solvent Compatibility	Scavengers work in both organic or aqueous or mixtures of solvents
Fast Kinetics	Room temperature kinetics, can also be heated
Easily Scalable	From mg screening experiments to multi-Kg applications
Generic Method for Batch	High efficiency generic add, stir, filter protocols
Generic Method for Flow	High efficiency fixed-bed format protocols
Cost Efficient	Reduces solvent, reduces labor, high metal recovery, reduces waste solvent disposal
Controlled Loading	Each batch lot controlled, very consistent lot-lot reproducibility
Thermally and Mechanically Stable	Works well with overhead stirring or mechanical shaking, withstands temperatures up to approx. 150–200 °C (i.e. microwave chemistry heating conditions)

Biotage has a flexible cartridge packing facility to accommodate many scale-up paths and options, from grams to Multi-kg and in a variety of formats for processing.

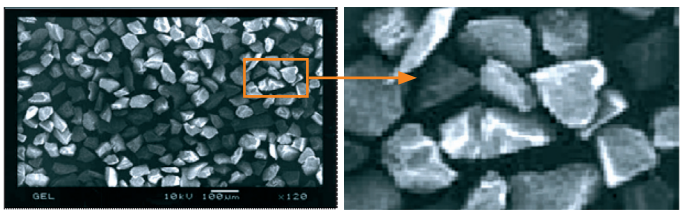

Figure 2. Scanning Eelectron Micrograph image of silica scavengers (x 120 magnification).

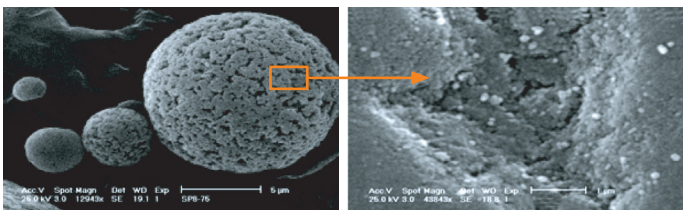

Figure 3. SEM image of MP-resin; permanently open pores allow greatest solvent compatability.

Regulatory Support Information

Biotage metal scavengers are made in an ISO9001:2008 compliant manufacturing facility and have served GMP pharmaceutical, drug discovery and fine chemical industries for many years. Each material is batch and lot controlled, with benefits of full traceability.

The metal scavengers are industry proven and supported by a comprehensive regulatory qualification support package incorporating:

- Certificate of Analysis
  - Extractables
  - Chemical loading
  - Lot information
  - Batch identity
- MSDS
- BSE/TSE statement
- Full Instructions and Suggestions for Use

Case Studies

Case 1 – Removal of Pd from Suzuki Reaction Using Biotage® MP-TMT (xs. catalyst)

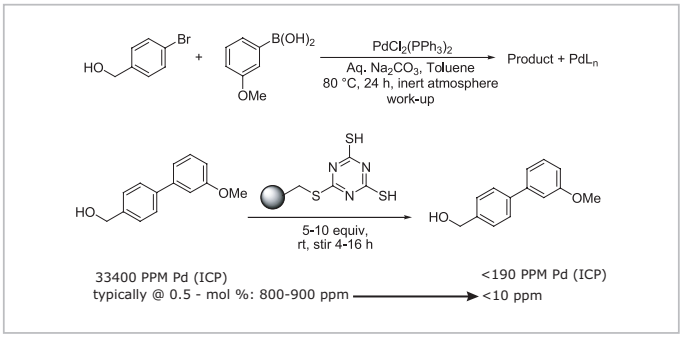

Figure 4. Removal of Pd from Suzuki reaction using Biotage® MP-TMT.

Conclusion

Adding 5 equivalents of Biotage® MP-TMT (with respect to Pd catalyst) to the extracted biaryl product of the Suzuki reaction and stirring at RT reduced Pd contamination 100–1000 fold. It is typical to start with initial Pd contamination approx. 800 ppm, resulting in final scavenged Pd at <10 and typically <5 ppm in final product.

Case 2 – Comparison of Different TMT Resins[5]

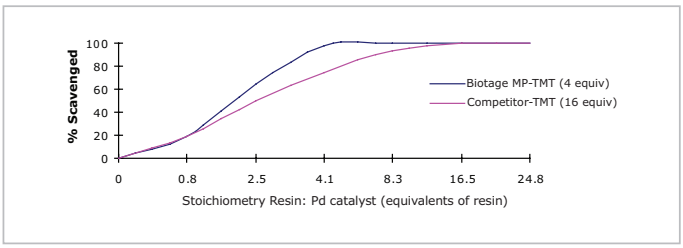

Figure 5: Biotage® MP-TMT or Bound-TMT resin was exhaustively stirred with $Pd(Cl)_2(PPh_3)_2$ in THF/DMF (50:50) (2 mL / total 8 µmol/852ppm Pd) for 16 hours at RT to investigate total capacity. Residual Pd was determined following scavenging.

Conclusion

Not all Resin-TMTs are the same. Under these conditions, for 100% scavenging, it was necessary to add 16 equivalents of a competitor-TMT, compared to only 4 equivalents of Biotage® MP-TMT. The difference was due to the precision engineered polymer back bone which gives more scavenging per unit mass of resin in the case of the Biotage scavengers. This results in lower actual costs.

References

5. Davies G.; Jordan S. and Rana, S. OPRD FT Lauderdale, April **2009.**

Case 3 – Removal of Pd from a Pd(dppf) Cl_2 Solution at RT by Fixed Bed Methods

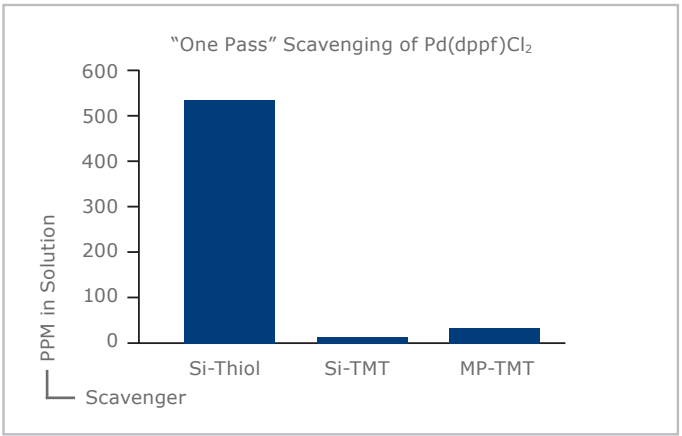

Figure 6. Initial Concentration 1000 ppm Pd, Fixed bed: 500 mg / 3 mL std fritted polyprop cartridge, Gravity flow, one pass. Si-TMT in fixed bed format was most effective in removal of the contaminated Pd solution.

Conclusion

Whilst Si-Thiol is a very good all-rounder, in very challenging situations (for example if there is sulfur in the target product) alternatives may be more efficient. Si-TMT in fixed bed format was more effective for metal scavenging than Si-Thiol, under identical conditions. Due to the smaller particle size; the Si-TMT was more effective than MP-TMT; as the bed was better packed. Metal scavengers can be applied directly to reaction mother liquors or to later stage, post extraction and purification. Many of end-users apply the scavengers to final stage API synthesis as they are most effective in those environments.

Case 4 – Comparison of Supported TMT Ligands with Thiol[6]

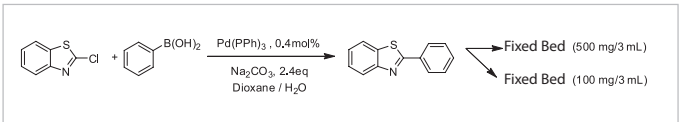

Figure 7. In metal scavenging cartridges %Pd reduction was monitored by ICP, following one-pass gravity flow through cartridges filled with either Biotage® Si-TMT or Biotage® Si-Thiol.

Scavenger		500 mg/3 mL	100 mg/3 mL
Si-Thiol	ICP Pd reduction	86%	45%
Si-TMT	ICP Pd reduction	>99%	95%

Conclusion

In very challenging scavenging situations, it is important to use the scavengers that have high Pd affinity. The TMT ligands Biotage use are the strongest available; allowing more efficient scavenging with less scavenger material.

References

6. S. Rana, Unpublished Results, Biotage 2008. Method as per: Heo,Y.; Song, Y.S.; Kim, B.T.; Heo, J. *Tetrahedron Lett.* 47 (2006), 3091–3094.

Generic Metal Scavenging Methods

Screening Protocol – Batch (Best for Resins)

1. Add solid scavenger to the product dissolved in a suitable solvent. In initial scoping to select the best scavenger, add 0.5–1 equivalents of scavenger directly to the solution of API or synthetic intermediate from which metal residues are to be removed.
2. No pre-wetting of the scavenger is required for batch stirring.
3. Agitate the mixture for at least an hour at room temperature for initial investigations.
4. Scavenging progress can be followed by normal analytical techniques. In many cases a strong indication of the progress of metal removal can be gained by the loss of color from the solution. Often the scavenging appears to be complete within 5–10 minutes at room temperature and this can be checked by measuring the metal content.
5. Coloration of the white, off-white or cream colored scavenger is an indication of metal removal from the solution.
6. At the end, the metal scavenger is removed via filtration, typically using a sintered funnel, glass wool or SPE cartridge. The scavenger is washed with additional solvent and combined solution concentrated.
7. At this stage, the best scavenger(s) procedure can be scaled directly. Further optimization of scavenger quantity, temperature, scavenging time and solvent type can be undertaken if required (see next page).
8. Cartridge or slurry applications are possible.

Typical results Pd (800 ppm) ▸ ‹10 ppm in one application (best for resin scavengers).

Screening Protocol – Flow (Resins and Silicas)

The metal scavenging kit (K-MS-2)kit contains Si-TMT in a 500 mg/6 mL SPE cartridge. Due to the highly efficient nature of the Pd binding onto the scavenger, this fixed-bed format, using Si-TMT is directly scalable to multi-Kg scale. As a guide, based on typical values for the 9 factors in figure 10 (P.T.O.), a 500 mg cartridge of Si-TMT in a 6 mL cartridge will completely remove all traces of Pd from a 1 mol% catalytic screening/test reaction at 1 mmol (product) scale.

1. Equilibrate cartridge by passing 3–5 bed volumes of API/product solvent through. This will remove any air gaps and enhance the scavenging process. This step is not critical if the cartridge is to be used under capacity.

Add dissolved product to cartridge and allow to flow through at RT under gravity. The solution may be ‘pushed’ using slight air or nitrogen pressure or it may be ‘pulled’ gently, using light vacuum (this method is especially amenable to processing on Biotage® FlashVac).

2. Pd (even in ‘clear colorless’ solutions), on scavenging will cause Si-TMT to change color. It is normal to see a dark brown/black band of Pd at the top of the silica bed. Breakthrough of Pd into the eluant was not observed, even following multiple (50+) bed volume washes using solvent under extreme testing.
3. Once product has passed through, wash cartridge with 5 bed volumes of solvent to ensure full recovery.
4. Products are not retained on the Si-TMT support as the silica is neutral, simple washing with bed volumes of solvent is enough to elute all product. Depending on the structure, some compounds may require more washing for complete elution.

Typical results Pd (800 ppm) ▸ ‹10 ppm in one pass flow approach (usually better with silica scavengers). Repeating this process will reduce Pd to even lower levels.

Optimizing Generic Scavenging Methods

Equivalents of Scavenger

If ICP data is available, typically 2–4 equivalents of scavenger with respect to metal can be used as a starting point in the calculation, or 50 wt% scavenger to metal in the API is usually a good place to start if ICP is not available. Once the preferred scavenger is identified then the scavenging process can be further optimized by reducing the number of equivalents. Depending on product and solvent conditions, it is possible to use 5–20 wt% scavenger with respect to API to remove metals to ‹5 ppm.

Temperature

Many scavenging process steps can be operated at RT in reasonable timescales (typically 15 min to 2 hr). Faster rates of metal removal can be achieved by heating or via fixed bed protocols. Although similar performance was seen at RT compared to elevated temperatures under identical conditions; Biotage media can withstand elevated temperatures without degradation and can be added directly to warm reaction mixtures and process streams.

pH of the Aqueous Reaction Mixtures

Biotage scavengers can be used across a wide pH range (pH 2–10), thought should be given to the optimum pH range for abstraction of target metal or organic impurity. High pH (above pH 10) should generally be avoided with silica supported scavengers; resin based scavengers should be tested if it is necessary to work at this level of pH for extended times.

Conclusion

“Neutral” ligands (such as Si-Thiol, MP-TMT) have very wide pH scope. Charged ligands can be switched on and off depending on pH.

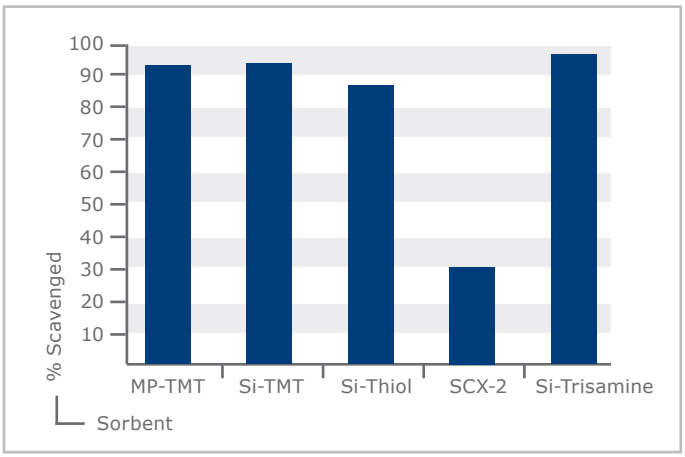

Figure 8. Batch Scavenging (2 hrs stir) 1000 ppm $Pd(PPh_3)_2Cl_2$ @ 10 °C.

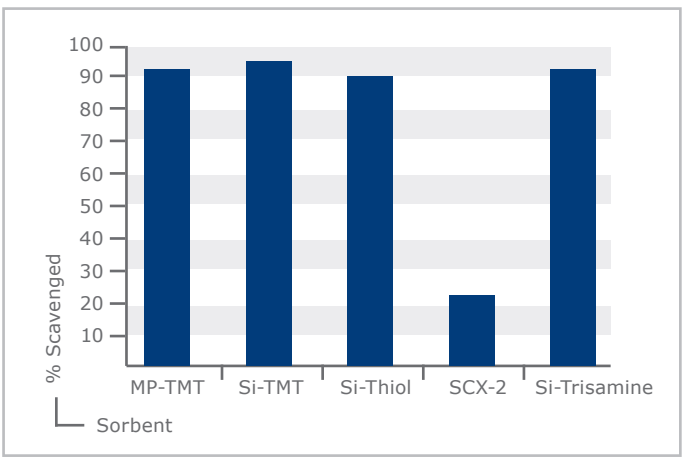

Figure 9. Batch Scavenging (2 hrs stir) 1000 ppm $Pd(PPh_3)_2Cl_2$ @>40 °C.

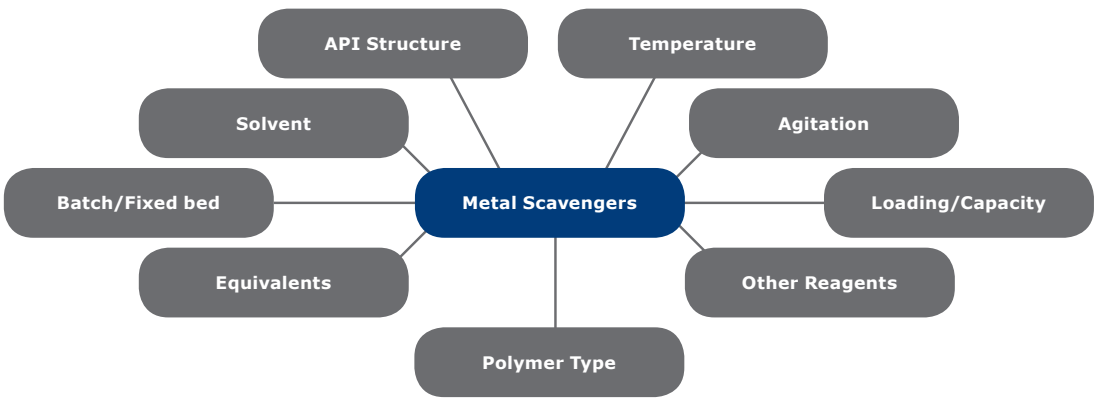

Figure 10. Factors involved in effective metal scavenging.

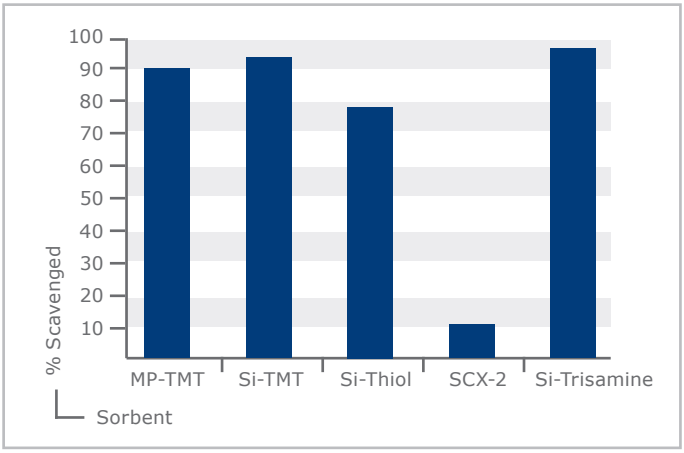

Figure 11. Batch Scavenging 1000 ppm $Pd(PPh_3)_2Cl_2$ @ pH 1.

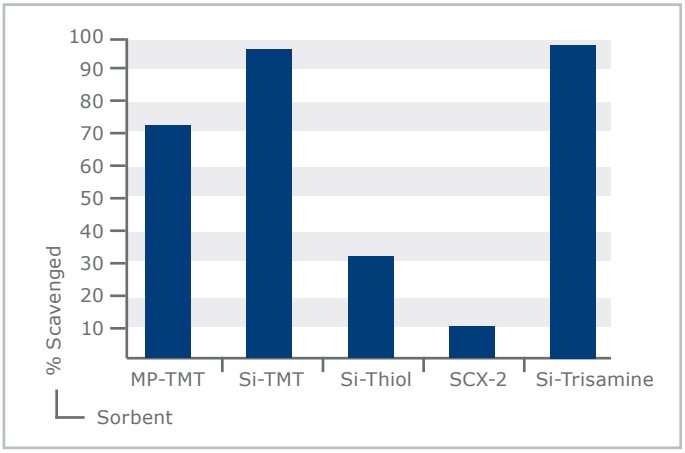

Figure 12. Batch Scavenging 1000 ppm $Pd(PPh_3)_2Cl_2$ @ pH 1.9.

Agitation Rate

For batch processes, higher agitation rates can enhance scavenging performance by improving mass transport. Typically, gentle to moderate agitation rate using magnetic, overhead or shaking methods is sufficient though.

Other Reagents Present in the Reaction Mixture

Reactants in the reaction mixture may interact with the scavengers. Clearly there should be no interaction between the functional groups on the scavenger and the other reagents, otherwise this may necessitate the use of additional equivalents of the scavenger.

Solvent

Macroporous resin (MP) and silica supported (Si) scavengers can be used in a wide range of aqueous and organic solvents, including THF, DMF, ethyl acetate, toluene, MeOH, ethers, chlorinated solvents and water. Co-solvent/solvent substitution should be considered if kinetics are slow. Biotage has excellent support and advice in this respect.

Batch/Flow

Si scavengers are usually more efficient in fixed beds or in flow due to their smaller particle size and enhanced packing, resins are typically employed in batch stirring processes, where additional mechanical rigidity and robustness is desired.

Structure of the API

If the product or API of interest has a strong affinity for Pd, it will be necessary to use a more powerful metal scavenger to perform the optimization. In these cases, depending on the format (batch or flow) we would recommend using the MP-TMT or the Si-TMT scavengers.

Loading and Capacity

Loading capacity is not the critical factor for metal scavenging, activity of the resin or silica is the paramount. A lower loaded Si-TMT for example can outperform a more highly loaded Si-Thiol depending on the product being scavenged. Si-TMT used in fixed bed, can outperform the identical quantity of Si-TMT in batch stirring due to the kinetic amplification that fixed-bed formats can offer. The key is to consider the effect of format, ligand and work-flow. Biotage has 30+ years' experience in silica and polymer chemistry and can assist in such matters.

Ordering Information

Biotage® MP-TMT – Palladium Scavenger

Chemical Name: Macroporous polystyrene-2,4,6-trimercaptotriazine

Resin Type: Highly cross-linked macroporous poly(styrene-co-divinylbenzene)

Capacity: › 0.5 mmol/g (elemental nitrogen analysis)

Bead Size: 150–355 microns; 45–100 mesh (›90% within)

Application: Scavenging palladium from aqueous and nonaqueous solutions

Typical Scavenging Conditions: 5 equivalents Biotage® MP-TMT relative to palladium content, 16–24 h, room temperature

Compatible Solvents: THF (4.3 mL/g), dichloromethane (DCM) (3.8 mL/g), toluene (‹4.9 mL/g), acetonitrile (ACN) (‹4.6 mL/g), methanol (MeOH) (4.3 mL/g), water (3.7 mL/g)

Storage: Cool, dry location

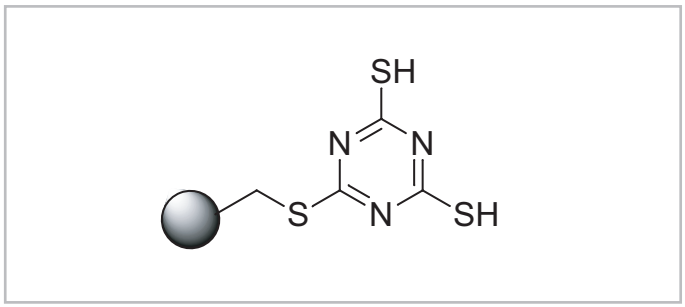

Figure 13. Biotage® MP-TMT structure.

Part Number	Quantity
801506	3 g
801469	10 g
801470	25 g
801471	100 g
801472	1000 g

Biotage® Si-TMT – Scavenger/Reagent

Material Type: 2,4,6-trimercaptotriazine silica

Solid-Support Type: Silica

Typical Capacity: 0.3 mmol/g

Size: 60 µm

Appearance: Free flowing off-white powder

Application: Metal scavenging

Typical Conditions: 3 equivalents ISOLUTE® Si-TMT, 30 min, room temperature

Compatible Solvents: Water, Acetonitrile (MeCN), methanol, Dimethylsulfoxide (DMSO),Dichloromethane (DCM), tetrahydrofuran (THF), N,N-Dimethylformamide (DMF), Dioxane

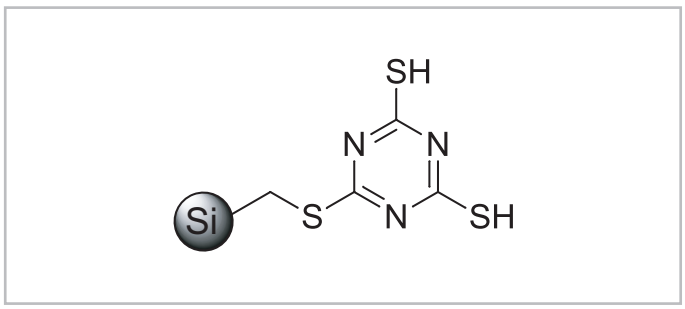

Figure 14. Biotage® Si-TMT structure.

Part Number	Quantity
9538-0010	10 g
9538-0025	25 g
9538-0100	100 g
9538-1000	1000 g

Biotage® Si-Thiol – Metal Scavenger/Reagent

Chemical Name: Silica 1-propanethiol;
3-Mercaptopropyl silica gel (Si-Thiol; Si-SH)

Solid-Support Type: Silica

Typical Capacity: 1.3 mmol/g

Size: 63 µm

Appearance: Free flowing off-white powder

Application: Metal scavenger (e.g. Pd, Pt, Cu, Hg, Ag and Pb); electrophile scavenger (e.g. alkyl-, benzyl- and allyl- halides, acid chlorides, isocyanates)

Typical Conditions: Add 2–6 equivalents to crude reaction mixture, stir for 1–12h, and then filter

Storage: Cool (4 ˚C), dry location

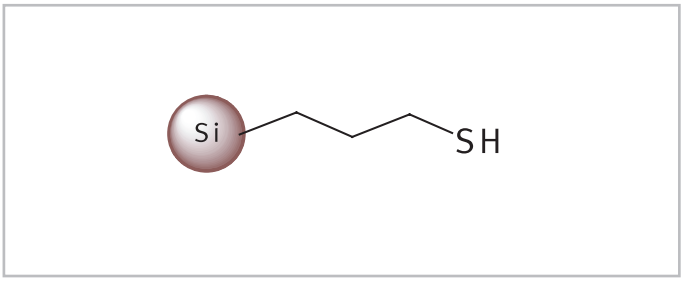

Figure 15. Biotage® Si-Thiol structure.

Part Number	Quantity
9180-0010	10 g
9180-0025	25g
9180-0100	100 g
9180-0500	500 g
9180-1000	1000 g

Biotage® Si-Trisamine – Scavenger/Reagent

Material Type: Propyl tris-(2-aminoethyl)amine silica

Solid-Support Type: Silica

Typical Capacity: 1.2–2.1 mmol/g based on benzoyl chloride uptake

Size: 65 µm (average)

Extraction Residue: ‹0.1%

Application: Metal scavenging, scavenging acid chlorides, sulfonyl chlorides, isocyanates, chloroformates, anhydrides and other electrophiles

Compatible Solvents: water, acetonitrile, methanol, DMSO, DCM, THF, DMF, Dioxane

Storage: Cool (4 ˚C), dry location

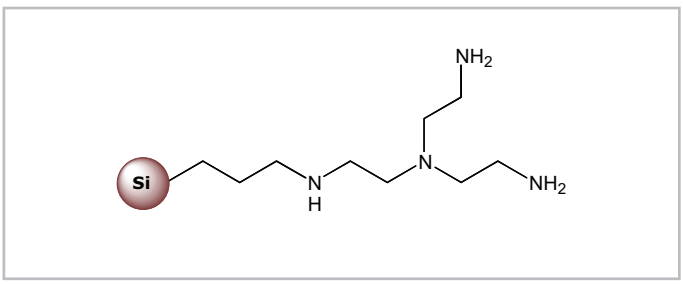

Figure 16. Biotage® Si-Trisamine structure.

Part Number	Quantity
9495-0010	10 g
9495-0025	25g
9495-0100	100 g
9495-0500	500 g
9495-1000	1000 g

Biotage® Si-Propylsulfonic Acid (SCX-2) – Scavenger/Reagent

Chemical Name: Silica Propylsulfonic Acid (Si-propylsulfonic acid; SCX-2)

Solid-Support Type: Silica

Typical Capacity: 0.7 mmol/g

Size: 52 µm

Appearance: Free flowing off-white powder

Application: Scavenger of amine, Catch-and-Release, Acid catalyst, metal scavenger

Typical Conditions: Stir crude reaction mixture with 2–4 equivalents for 1 h and filter

Compatible Solvents: Methanol (MeOH), Dichloromethane (DCM), Acetonitrile (MeCN), Acetone, Ethyl Acetate (EtOAc), N,N-Dimethylformamide (DMF), Dimethylsulfoxide (DMSO)

Storage: Cool (4 °C), dry location

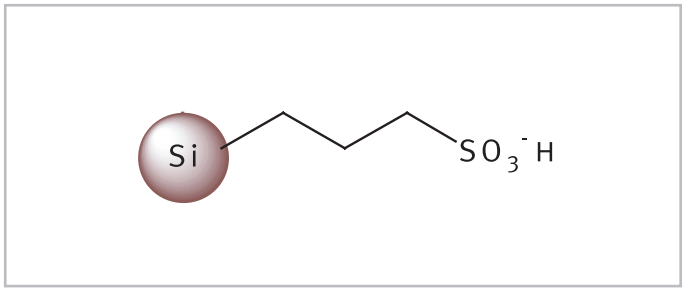

Figure 17. Biotage® SCX-2 structure.

Part Number	Quantity
9536-0010	10 g
9536-0025	25 g
9536-0100	100 g
9536-1000	1000 g

Biotage® Metal Scavenging Tool Kit

- MP-TMT 3 g
- SCX-2 3 g
- Si-TMT 3 g
- Si-TMT 500 mg/6 mL cartridges Pk/5
- Si-Thiol 10 g
- Si-Trisamine 10 g

Figure 18. Biotage® Metal Scavenging Tool Kit.

Part Number	Quantity
K-MS-2	1

Biotage Resin Family Selection Guide

Product	Metal Scavenging	Peptide/ Coupling Chemistry	Reductive Amination	Oxidation	C-C Bond Formation	Work-up Scavenging
MP-TMT	■					
Si-TMT	■					
Si-Thiol	■					
Si-Trisamine	■					■
SCX-2	■		■			■
PS-Carbodiimide		■		■		
PS-TBD		■			■	
PS-HOBt		■				■
PS-DIEA		■				■
PS-DMAP		■				■
ACTU		■				
MP-Triacetoxyborohydride			■			
MP-Cyanoborohydride			■			
MP-Borohydride			■			
PS-$TsNHNH_2$			■			■
PS-Isocyanate			■			■
MP-Isocyanate			■			■
Si-Carbonate					■	■
PS-PPh_3-Pd					■	
PS-PPh_3					■	■
PS-Benzaldehyde			■			■
PS-TEMPO			■	■		
MP-TsOH	■		■			■
PS-Trisamine	■		■			■
MP-Trisamine	■		■			■
Si-Triamine	■		■		■	■

■ Excellent/Preferred

■ Very Good

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