

ISOLUTE® Si-Thiol

Metal Scavenger

Key Facts



Stoichiometric



Shelf Life

Capacity
(mmol/g)

BSE/TSE



Scalable

Particle Size
(μm)Thermally &
Mechanically
StableGood
Laboratory
PracticeBulk Density
(g/L)

Specifications

Chemical Name:	Silica 1-propanethiol; 3-Mercaptopropyl silica gel (Si-Thiol; Si-SH)
Solid-Support Type:	Silica
Appearance:	Free flowing off-white powder
Application:	Scavenger of electrophiles eg. alkyl-, benzyl- and allyl- halides, acid chlorides, isocyanates; Metal scavenger (e.g., Pd, Pt, Cu, Hg, Ag and Pb)
Typical Conditions:	Add 3-5 equiv. Stir for 15 min-2 h, then 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

Palladium-catalyzed reactions are very popular in organic synthesis. Some of the most widely practiced examples include Suzuki-Miyaura cross coupling reactions, Heck reactions, Buchwald aminations, Wacker-type oxidation, hydrogenation, allylation, reductive de-allylation and indole formation.

Despite the widespread use of these palladium-mediated reactions, removal of residual palladium during workup and product isolation remains a major problem. Reducing the palladium content to the low parts per million (ppm) levels, as is required for active pharmaceutical ingredients, is particularly challenging.

Biotage has developed a portfolio of scavenging products designed to remove these transition metal impurities. ISOLUTE® Si-Thiol was developed to address the need for a versatile metal scavenger that can be deployed in a multitude of different cases. It is the workhorse of the scavenging world and a first port of call for many screening and process applications.

ISOLUTE® Si-Thiol is the silica-bound equivalent of 1-propanethiol which is primarily used in the pharmaceutical industry as a metal scavenger for metals such as Pd, Pt, Cu, Hg, Ag and Pb but it can also be useful for covalent scavenging of alkyl, benzyl and allyl halides as well as a variety of other electrophiles including acid chlorides and isocyanates.

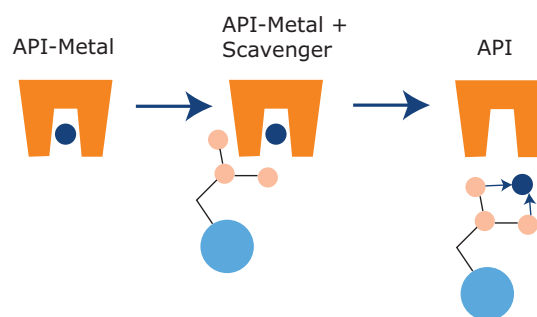
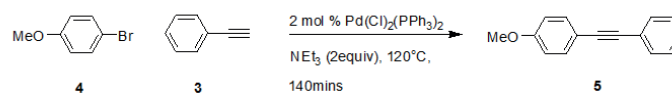


Figure 1. Metal Scavenging

In 2006, Li reported a modified Palladium-catalyzed cross-coupling reaction under copper, amine and solvent free conditions. Due to the lack of other contaminating metal, we used this as a model reaction for demonstrating metal scavenging with Biotage Si-Thiol. In our example study, alkyne (5) was prepared in previously published yields and purities (Scheme 1), however the product was found to contain high levels of contaminating



Scheme 1: Model copper-free, amine-free Sonogashira reaction

palladium. This initial reaction offered a starting point for to explore removal of Pd.

Simple treatments with Si-Thiol metal scavenger significantly reduced the palladium content (Figure 2).

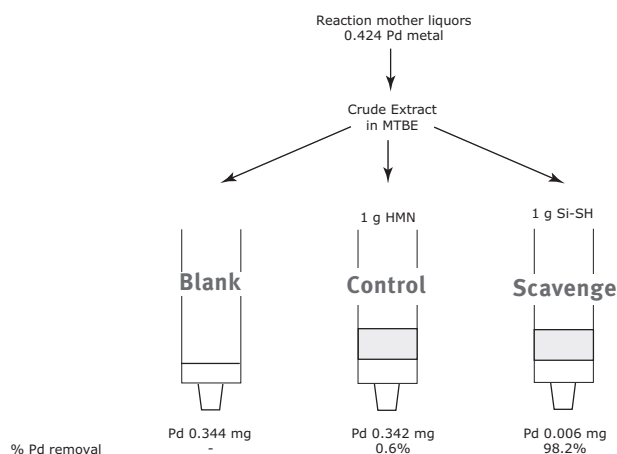


Figure 2. The effect of Si-Thiol.

It is sometimes impractical to perform scavenging reactions for long periods of time so we have additionally considered the effect of time on scavenging using Si-Thiol (Figure 2).

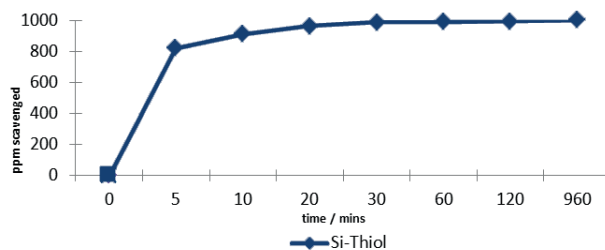


Figure 2. Batch scavenging kinetics using 3 eq of Si-Thiol metal scavenger in a 1000ppm Pd solution of Pd(OAc)₂, over time.

Based on previous optimization 3 eq of scavenger was used and scavenging measured over time. In the case of the Pd(OAc)₂, Si-Thiol was able to remove the Pd from solution at 3 equiv. after 960 minutes however 98.6% and 99.1% of the palladium had been removed by the 30 and 60 minute marks, respectively.

Ordering Information

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

EUROPE

Main Office: +46 18 565900
 Fax: +46 18 591922
 Order Tel: +46 18 565710
 Order Fax: +46 18 565705
 order@biotage.com
 Support Tel: +46 18 56 59 11
 Support Fax: +46 18 56 57 11
 eu-1-pointsupport@biotage.com

NORTH & LATIN AMERICA

Main Office: +1 704 654 4900
 Toll Free: +1 800 446 4752
 Fax: +1 704 654 4917
 Order Tel: +1 800 446 4752
 Order Fax: +1 704 654 4917
 ordermailbox@biotage.com
 Support Tel: +1 800 446 4752
 us-1-pointsupport@biotage.com

JAPAN

Tel: +81 3 5627 3123
 Fax: +81 3 5627 3121
 jp_order@biotage.com
 jp-1-pointsupport@biotage.com

CHINA

Tel: +86 21 68162810
 Fax: +86 21 68162829
 cn_order@biotage.com
 cn-1-pointsupport@biotage.com

KOREA

Tel: +82 31 706 8500
 Fax: +82 31 706 8510
 korea_info@biotage.com
 kr-1-pointsupport@biotage.com

INDIA

Tel: +91 11 45653772
 india@biotage.com

Distributors in other regions
 are listed on www.biotage.com

Part Number: PPS374.V.2

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