ISOLUTE® Si-TMT

Metal Scavenger

Specifications

Chemical Name:
2,4,6-trimercaptotriazine silica

Solid-Support Type: Silica

Typical Capacity: 0.3 mmol/g

Size: 60 µm

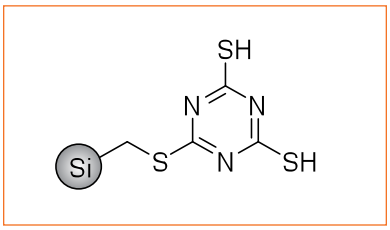
Appearance: Free flowing off-white powder

Applications: Metal scavenging

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

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

ISOLUTE® Si-TMT



ISOLUTE Si-TMT is the silica bound equivalent of 2,4,6-trimercaptotriazine (TMT). Si-TMT has been shown to efficiently scavenge residual palladium from palladium-catalyzed reactions. The function group in Si-TMT is attached to a bioanalytical grade silica platform, thus the use of this material does not introduce impurities to the reaction system. There are numerous advantages of functionalized Isolute Silica.

Solvent Independent

Silica neither shrinks nor swells in any solvent, which makes it ideal for limited volume reactors (e.g. microwave vials, 96 well plates, SPE columns).

Fast Kinetics

Since the functional groups are on the surface and in pores of the silica, similar to highly cross-linked macroporous polystyrene beads, the rate of reaction is not controlled by diffusion as with the 1% crosslinked PS- polymer matrices.

Simplified Workflow

Reaction mixtures containing silica-based reagents or scavengers can be loaded directly onto a flash cartridge (after solvent evaporation) for flash chromatography, without the need for filtration or work-up.

Si-TMT can be added to a reaction in "batch mode" and stirred in the traditional ways, "flow-through mode" in a cartridge, or used in a microwave reactor. The material is stable at room temperature and under atmospheric conditions.

Si-TMT is an effective scavenger for transition metals from aqueous or organic solutions. Si-TMT was applied successfully to the flow through, one-pass scavenging of palladium acetate (Figure 1), dichlorobis(triphenylphosphine)palladium (II) (DCB) (Figure 2) and tetrakis(triphenylphosphine)palladium (0) (KIS) (Figure 3) using MP-TMT from Biotage, Si-Thiol from Biotage, and competitor supported thiourea scavenger.

Figure 1

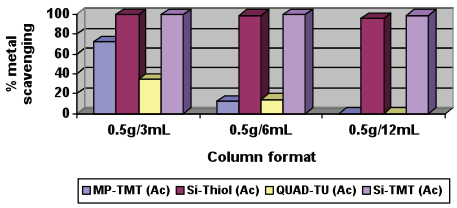


Figure 1. Palladium acetate scavenging efficiency using Si-TMT in column format in comparison with popular solid supported scavengers

Figure 2

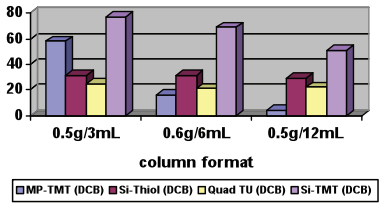


Figure 2. Dichlorobis(triphenylphosphine)-palladium (II) (DCB) scavenging efficiency using Si-TMT in column format in comparison with popular solid supported scavengers

Figure 3

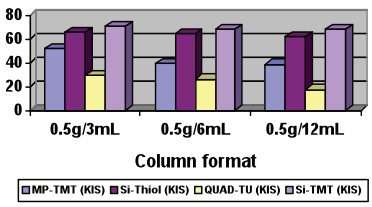


Figure 3. tetrakis(triphenylphosphine)-palladium (0) (KIS) scavenging efficiency using Si-TMT in column format in comparison with popular solid supported scavengers

Biotage

Thermally and Mechanically Stable

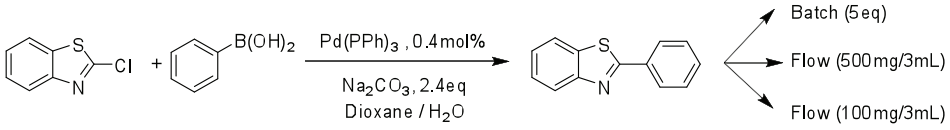
Most silica based products, like polymers, can withstand temperatures over 200 °C and are, equally suitable for microwave synthesis. These products are stable to mechanical agitation and can be used in conjunction with both magnetic and mechanical stirring.

Easy to use and handle

Silica does not carry a static charge and therfore is easy to weigh and handle. These products do not stick to glassware and do not require extensive washing for high product recovery.

Application: Suzuki Coupling

A Suzuki coupling was performed (Scheme 1) and scavenging experiments were performed on the crude reaction mixture in both batch and flow mode. The results, as shown in Table 1., show that Si-TMT is more efficient in scavenging palladium residues than other silica based metal scavengers.



Scheme 1: Suzuki Coupliing

	Batch Studies (5 equiv.)	Flow (cartridge) scavenging	
		500mg/3mL	100mg/3mL
Si-Thiol (Biotage)	91%	86%	45%
Si-Thiol (Competitor S)	82%	91%	45%
Si-TMT (Biotage)	>99%	>99%	95%

Table 1: Results of metal scavenging experiments following reaction shown in Scheme 1. Table shows percentage of reduction of palladium as measured by ICP.

Ordering Information for ISOLUTE Si-TMT

Part Number	Size	Description
9538-0025	25 g	Scavenger of heavy metal ions such as Pd^{2+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Pb^{2+}, Ru^{2+} and Zn^{2+}
9538-0100	100 g	
9538-1000	1 Kg	

www.biotage.com

North America
Main Office: +1 434 979 2319
Toll Free: +1 800 446 4752
Fax: +1 434 979 4743
Order Tel: +1 434 220 2687
Order Fax: +1 434 296 8217
ordermailbox@biotage.com

Europe
Main Office: +46 18 56 5900
Fax: +46 18 59 1922
Order Tel: +46 18 56 57 10
Order Fax: +46 18 56 57 05
order@eu.biotage.com

Japan
Tel: +81 422 28 1233
Fax: +81 422 28 1236
jp_order@biotage.com

To locate a distributor please visit our web site at www.biotage.com.

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