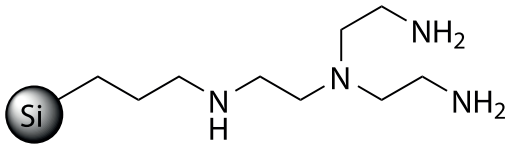
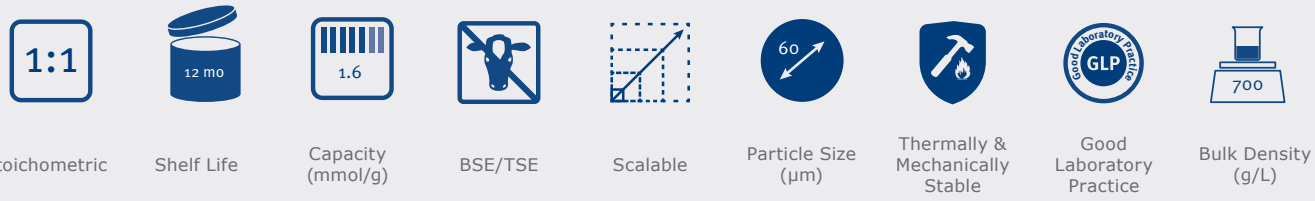
ISOLUTE® Si Trisamine

Metal Scavenger



Key Facts



Stoichometric | Shelf Life | Capacity (mmol/g) | BSE/TSE | Scalable | Particle Size (µm) | Thermally & Mechanically Stable | Good Laboratory Practice | Bulk Density (g/L)

Specifications

Chemical Name:	Propyl tris-(2-aminoethyl) amine silica
Solid-Support Type:	Silica
Applications:	Metal scavenging, scavenging acid chlorides, sulfonyl chlorides, isocyanates, chloroformates, anhydrides and other electrophiles
Typical Scavenging Conditions:	Si-Trisamine (3 equiv.) agitated with metal or electrophile at room temperature for 30 min.
Compatible Solvents:	Water, acetonitrile, methanol, DMSO, DCM, THF, DMF, Dioxane
Storage:	Cool, dry location

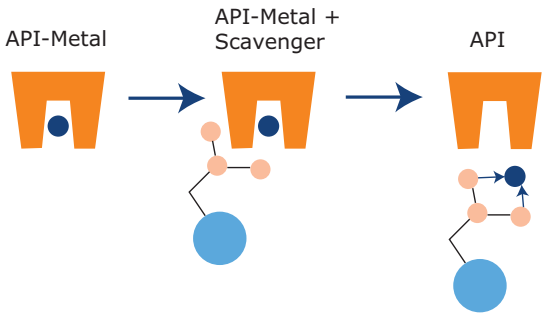


Figure 1. Metal Scavenging

ISOLUTE® Si-Trisamine is a silica bound equivalent of a trisamine base and can scavenge a variety of electrophiles, including aldehydes, acid chlorides, sulfonyl chlorides, isocyanates, isothiocyanates and heavy metal ions such as Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Pb^{+2}, Ru^{+2} and Zn^{2+}.

Si-Trisamine has a number of uses, it is a very powerful scavenger of transition metals and can also be used as a work-up tool to remove electrophiles or acids in either batch or flow-mode (cartridge) following traditional reflux heated or microwave synthesized reactions.

Metal Scavenging

Heavy metals such as palladium, copper and tin are widely used in diverse transformations and their removal following organic synthesis has become a vital part of the API scale-up process. Si-Trisamine was applied successfully in the scavenging of palladium acetate, copper sulfate and nickel acetate from aqueous and organic solutions (**Figure 1**).

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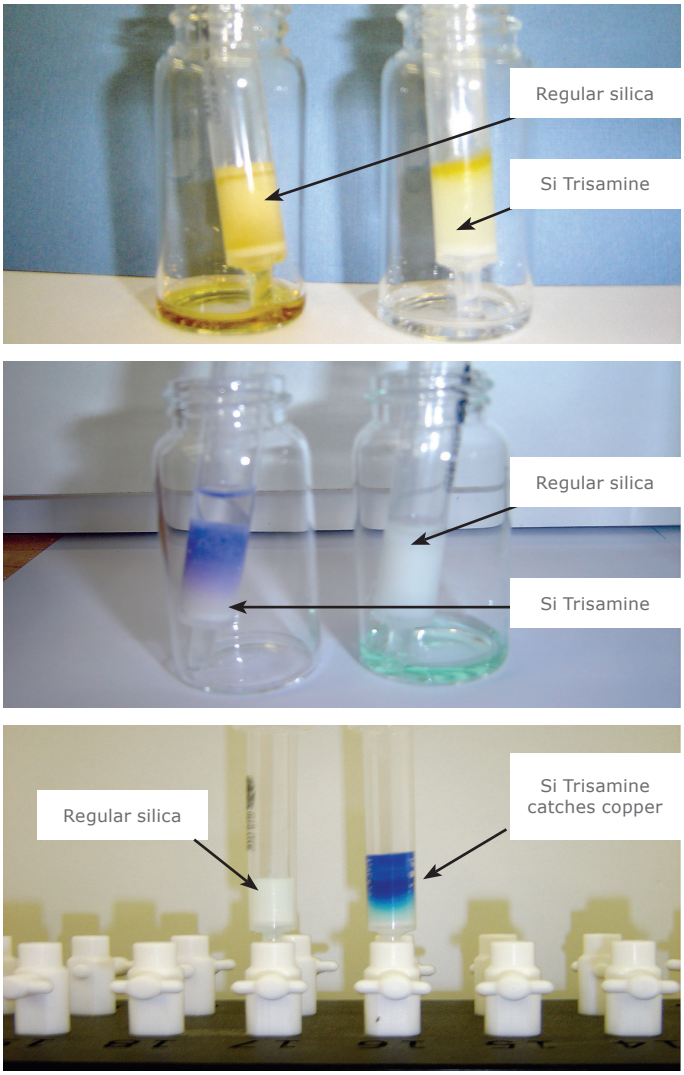

Figure 1. (Top) Scavenging of a 50 mg/50 mL THF/DMA solution of $Pd(OAc)_2$ using a 500 mg/3 mL Si-Trisamine cartridge. Total residence time was 30 seconds, and the solution was allowed to flow through under gravity.

(Middle) Scavenging of a 250 mg/5 mL aqueous solution of $Ni(OAc)_2$.

(Bottom) Scavenging of a 500 mg aqueous solution of $CuSO_4$.

Medicinal Chemistry

Sulfonamides and amides are important pharmacophores, commonly synthesized via activation of the corresponding acid followed by reaction with an amine substrate. Si-Trisamine can be used to scavenge the excess activated electrophile, eliminating the need for column chromatography. The use of Si-Trisamine as a scavenger of excess 2,3,4,5,6-pentafluorobenzoyl chloride, benzoyl chloride or p-toluene sulfonyl chloride allowed efficient isolation of a variety of alkylated adducts **(Figure 2)**.

Si-Trisamine is effective at scavenging acidic species from solution. Acid/base kinetics are generally very fast so that a single pass of the acidic species through a Si-Trisamine cartridge (5 equiv. excess) with residence time of less than a minute is enough to effect complete scavenging of the acidic component. This was demonstrated visually using an acidic dye **(Figure 3)**.

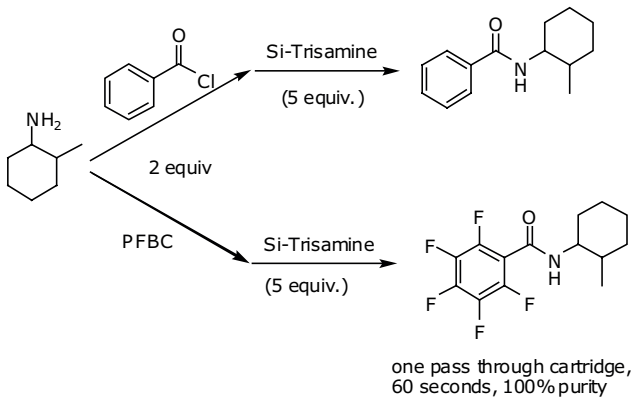

Figure 2. Generalized reaction scheme of acid chlorides with the Si-Trisamine reagent. Complete scavenging was achieved even in the case of the electron withdrawing pentafluorobenzoyl chloride substrate in cartridge mode. Although the 'batch' stoichiometric excess of Si-Trisamine was only 5 equivalents, the scavenging was complete in one pass with a flow through time of 60 seconds.

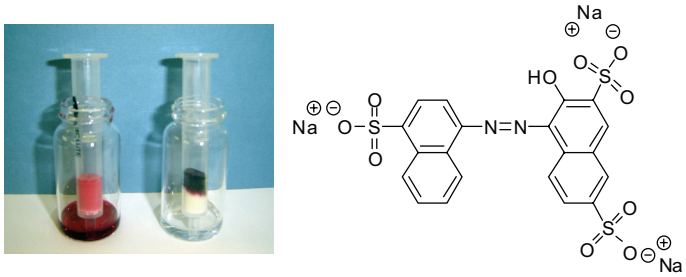

Figure 3. One pass flow through scavenging/catching of an acid water soluble dye (Amaranth/Acid Red) in a 500 mg/3 mL cartridge of Si-Trisamine. Total residence time was approximately 30 seconds. The image on the left is a blank/control experiment comprising of a 500 mg/3 mL cartridge of endcapped C18 silica. The effect of Si-Trisamine is quantitative. The elution solvent contained no red dye.

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Ordering Information

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

ISOLUTE® Si-Trisamine is included in a comprehensive metal scavenging screening tool kit, an essential tool to quickly and effectively find the right metal scavenger for the application in hand. With protocols, guidance and recommendations, this all in one kit showcases all of the key metal scavengers.

Part Number: K-MS-2.

bsi. ISO 9001 Quality Management FM 31206 ISO 14001 Environmental Management EMS 640981

Biotage holds certification for both ISO9001 Quality Management and ISO14001 Environmental Management.

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