

SiliaMets®
Specification Sheet

SiliaMets® Triamine

Product number: R48030B
Particle size: 40-63 µm
Pore diameter: 60 Å
Particle shape: Irregular Silica Gel



Product description

Better known for their electrophile scavenging efficiency, and their base reagent quality, SiliaBond Amine, SiliaMets Diamine and Triamine are also proven scavengers for metals. They are very useful for scavenging Pd, Pt, Cr, W and Zn.

Product specifications

Properties	Specifications	Analysis methods
Particle size distribution (%) (Under 40 µm / Over 63 µm)	≤ 5 / ≤ 5	Sieving
Specific surface area (m ² /g)	480-550	Brunauer, Emmett and Teller (BET) Analysis
Pore diameter (Å)	55-65	
Pore volume (mL/g)	0.70-0.85	

Test report

Properties	Specifications	Analysis methods
Nitrogen content (N%)	≥ 4.65	Elemental analysis
Volatile content (% , 110°C)	≤ 4.00	Humidity measurement
Color	Off-white	Visual test

Physical properties

Properties	Specifications	Analysis methods
Molecular loading (mmol/g)	≥ 1.11	Based on nitrogen content
Nitrogen loading (mmol/g)	≥ 3.33	
Surface coverage (µmol/m ²)	≥ 2.73	Based on molecular loading
Purity (%)	≥ 99.95	GC-MS
Endcapping	Yes	-
Typical tap density (g/L)	736	-
Solvent compatibility	All solvents, aqueous and organic	-

Storage conditions and shelf-life

Prolonged storage	Shelf - life
Keep cool & dry (<8°C)	2 years

Recommended conditions for this product are to keep it dry in a tightly closed container. However, for prolonged storage, optimal recommended conditions is to always keep product in a cool place (< 8°C) under inert atmosphere (*argon*) when possible.

Screening in Batch Reactor Mode (*Bulk*)

To select the best scavenger for initial screening experiments, do the following steps for each SiliaMets Metal Scavengers included in the kit. Use 4-8 molar equivalents of each SiliaMets in respect to the residual metal concentration.

1. Dissolve the crude product to be treated in a suitable solvent (*or use directly the crude reaction mixture*) and prepare vials containing the same solution volume.
2. Directly, add each SiliaMets included in the kit to these vials.

Note: no pre-wetting of the SiliaMets is required. See "Determining the Optimal Amount of SiliaMets to use" on page 96, of the SiliCycle Catalog for the Pharmaceutical Industry.

3. For initial tests, stir the solution for at least one hour at room temperature.
4. Scavenging progress can be followed by normal analytical techniques. The scavenging progress can be estimated by looking at the color of the solution. When the scavenging is almost complete, the solution is less colored and SiliaMets becomes colored. In some occasional cases, if all the samples are still coloured, try one or all of the following: let them react for a longer period of time; add more equivalents of the SiliaMets, increase the temperature of the reaction.
5. At the end of the scavenging, filter off the SiliaMets using a fritted funnel or filtration device.
6. Wash the SiliaMets with additional solvent for total recovery of the API (*or compound of interest*) and concentrate the solution under vacuum.
7. Analyze the residual metal concentration of each vial to identify the most efficient SiliaMets Metal Scavenger.

Note: you can choose more than one scavenger.

8. If you are satisfied with the scavenging efficiency of the best SiliaMets, direct scale-up is possible. Otherwise, scavenging optimization can be done with SiliaMets identified in #7 (*see next section*).

Experiment Optimization

If, upon completion of the screening procedure, the scavenging is not complete or you wish to either reduce the number of equivalents or the reaction time, optimization steps can be undertaken.

Various parameters can be changed, one at a time or simultaneously, to improve the metal removal efficiency.

Note: you can mix multiple SiliaMets to get superior efficiency.

Number of SiliaMets® Equivalents

For initial screening experiments we suggest that 4-8 molar equivalents be used, in respect to the residual metal concentration of each SiliaMets. Once the preferred scavenger is identified, further optimization can be done to reduce the number of equivalents used (*typically down to 2-4 equivalents*).

Subsequent Treatments

In some cases (*equilibrium process or the presence of multiple species*), multiple treatments with SiliaMets is suitable instead of a single treatment with a larger amount.

For optimal results, filtration between each treatment can allow a higher scavenging efficiency.

Reaction Time

In some cases, where increasing the temperature is impossible, longer contact time with the scavenger can allow a higher scavenging efficiency.

Conditions: Pd(OAc)₂, THF, SiliaMets Thiol, RT.

Temperature

In the initial screening, we suggest the scavenging experiments be run at room temperature. Usually, metal scavenging is completed after one hour or so. However, when shorter scavenging times are required, higher scavenging rates can be achieved by increasing the temperature. SiliaMets can be safely used at elevated temperature without degradation and can be added either at room temperature or directly to a warm solution.

Solvent

SiliaMets can safely be used in a wide range of organic and aqueous solvents commonly used in laboratory and in process, such as DMF, DMSO, THF, 2-butanone, alcohols, ethers, chlorinated solvent, etc.

Mixing Rate

SiliaMets are mechanically stable and offer excellent scavenging efficiency in batch processes agitated by overhead and magnetic stirrers, as well as orbital shaking under low to moderate agitation rates.

If required, mixing rates can be increased to get better scavenging results. With faster stirring, you improve SiliaMets dispersion in solution.



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