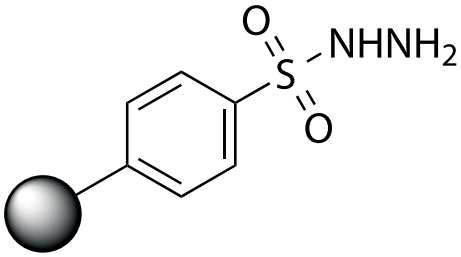
Biotage® PS-TsNHNH$_2$

Electrophile Scavenger



Key Facts

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

Specifications

Chemical Name:	Polystyrene sulfonyl hydrazide
Resin Type:	1% Cross-linked poly(styrene-co-divinylbenzene)
Application:	Scavenging aldehydes and ketones
Scavenging Conditions:	3 equivalents relative to carbonyl, 1–3 h, 20 °C, DCM. Ketones and hindered aldehydes are accelerated by the addition of HOAc (~10%) and/or heat.
Compatible Solvents:	DCM (7 mL/g), DCE (7 mL/g), THF (6.5 mL/g), DMF (7.2 mL/g)
Storage:	Cool, dry location

PS-TsNHNH$_2$ is a resin-bound equivalent of p-toluenesulfonyl hydrazide and is an excellent scavenger of aldehydes and ketones. Comparison with other polymeric benzyl hydrazide showed PS-TsNHNH$_2$ was a superior scavenger for carbonyls and much more stable to storage (the benzyl hydrazide resin decomposed on storage).

Removal of excess carbonyls from solution generally requires a three-fold excess of PS-TsNHNH$_2$. Addition of a catalytic amount of HOAc (5–10%) may be required for ketones and hindered aldehydes. HOAc is also required for sequestering aldehydes in DMF. Complete removal of common aldehydes occurs in 0.5 to 3 h and removal of a ketone takes from 2 to 16 h. Elevated temperatures were required for hindered ketones (e.g. 2,6-dimethylcyclohexanone). Upon completion of the scavenging, the resin is rinsed with a suitable solvent (i.e. those which swell polystyrene), and the product is isolated by concentration. PS-TsNHNH$_2$ was successfully used to work up the synthesis of alcohols by addition of a Grignard reagent to aldehydes.

PS-TsNHNH$_2$ is also potentially useful as a polymer-bound reagent. Bound sulfonyl hydrazones, formed by condensation with carbonyl compounds, can be utilized in further synthetic transformations. The high accessibility of tosyl hydrazide functional groups in PS-TsNHNH$_2$ afford high synthetic fidelity relative to reported systems.[1]

Representative Procedure

PS-Tosyl Hydrazine (0.6 g) was added to a mixture of 3-methoxybenzylaldehyde (20 µL, 1.5 mmol) and naphthalene (15 mg; internal standard) in THF (2 mL). This mixture was stirred for 30min, then filtered and washed with MeOH (8 mL). The filtrate was concentrated and analyzed by RP-HPLC.

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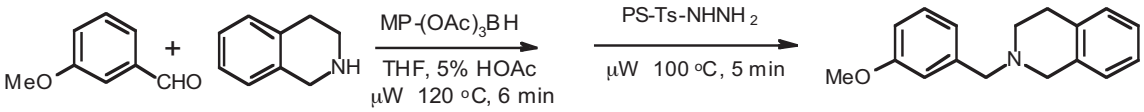

Scheme 2. One pot microwave-assisted reductive amination reaction followed by aldehyde scavenging.

Reductive Amination

This scavenger can be applied to the rapid clean up of tertiary amines prepared through reductive amination using MP-Triacetoxyborohydride and microwave irradiation (Scheme 1).

To a solution of the amine (0.5 mmol) and 3-methoxybenzylaldehyde (0.7 mmol) in a solution of THF and 5 % Acetic acid (2 mL) was added MP-Triacetoxyborohydride (523 mg, 2.39 mmol/g, 1.25 mmol). The vial was capped and heated to 120 °C for 6 min, then PS-TsNHNH$_2$ (0.6 g) added. The vial mixture was heated to 100 °C for 5 min, after which the reaction mixture was filtered. The solid was washed with a 1:1 solution of THF/MeOH (10 mL) and the filtrate was concentrated to afford product.

Ordering Information

Part Number	Quantity
800497	3 g
800270	10 g
800271	5 g
800272	100 g
800317	1000 g

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