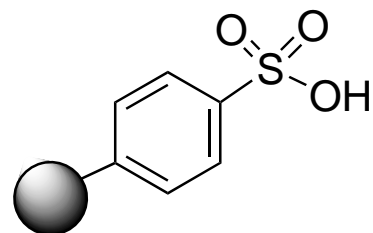


MP-TsOH

Resin Bound Acid



Resin Type: Macroporous poly(styrene-*co*-divinylbenzene)

Capacity: Typical capacity 4.0 mmol/g, minimum capacity 3.7 mmol/g (based on uptake of benzylamine)

Bead Size: 375–575 microns, 25–40 mesh (mean value)

Chemical Name: Macroporous polystyrene sulfonic acid (0.5% inorganic antistatic agent)

Application: Scavenging and “catch and release” of amines, acid catalysis

Typical Scavenging Conditions: Approx. 2–3 equiv of resin relative to amine, 0.5–1 h, 20 °C

Compatible Solvents: DCM (3.0 mL/g), THF (3.1 mL/g), DMF (3.1 mL/g), MeOH (3.05 mL/g)

MP-TsOH resin is a sulfonated macroporous polystyrene resin that is a resin-bound equivalent of *p*-toluenesulfonic acid (TsOH). The resin may be used as an equivalent to the strong cation-exchange resin, Amberlyst A-15 (Rohm and Haas).¹⁻⁴ MP-TsOH has been optimized for use as a bound reagent or scavenger resin for the synthesis of small molecules. The sulfonic acid groups in MP-TsOH are readily accessible for removal of basic compounds, *e.g.* primary, secondary, and tertiary amines, by quaternary salt formation. In addition, MP-TsOH does not contain dark leachable impurities derived from overoxidation of the polystyrene backbone observed in higher loading sulfonic acid resins.⁵

Representative amine scavenging examples (batch mode) as a function of time are provided in **Table 1**. MP-TsOH provides a useful alternative to quenching reactions with aqueous or soluble organic acids. MP-TsOH may also be used in cartridge applications to perform catch-and-release of amine derivatives in analogy to silica-derived SCX columns.⁶⁻⁸ MP-TsOH circumvents the contamination of amine products with particulates that sometimes occur with silica-derived SCX columns. Representative amine scavenging examples (cartridge mode) as a function of time are provided in **Table 2**.

PART NUMBER	QUANTITY
800461	10 g
800462	25 g
800463	100 g
800464	1000 g

We have found that catch and release purification of amines with MP-TsOH columns to be effective in retaining amines with a wide range of basicity, including N-methylmorpholine, aniline, aminothiazole and nitroaniline. Full retention of weakly basic amines such as nitroaniline was achieved when DCM was used as the solvent, however retention is less efficient in THF or DMF. The use of greater than three equivalents of resin is recommended for weakly basic amines in these solvents.

Table 1. Amine Removal by MP-TsOH (Batch Mode)

Amine	MP-TsOH (equiv)	% Scavenged	
		20 min	1 h
Propylamine	3	100	100
3-(morpholino)propylamine	3	100	100
Aniline	3	100	100
Nitroaniline	3	75	96
2-Aminothiazole	3	100	100

Table 2. Amine Removal by MP-TsOH (Cartridge Mode)

Amine	MP-TsOH (equiv)	% Scavenged 5 min
Propylamine	5	100
3-(morpholino)propylamine	5	100
Aniline	5	100
2-Aminothiazole	5	98

REPRESENTATIVE PROCEDURE

Catch and Release

The amine solution is incubated with MP-TsOH (3 equivalent relative to the amine) for 30 min. The resin is then filtered followed by DCM washing to remove non-basic impurities. Treating the resin with 2 M ammonia in methanol releases the amine as a free base. The same catch and release experiment can also be performed using MP-TsOH cartridges.⁹

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