### Technical Note 511

# **MP-Carbonate**

## Resin-Bound Base

**Chemical Name:** Macroporous triethylammonium methylpolystyrene carbonate (0.5% inorganic antistatic agent)

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

Capacity: 2.5–3.5 mmol/g (based on nitrogen elemental analysis)

**Bead Size:** 350–1250 microns, 18–52 mesh (95% within)

**Application:** General base, ammonium salt neutralization, scavenging acids and acidic phenols. The neutralization of insoluble amine hydrochlorides requires the use of 0.05–0.1 equiv of disopropylethylamine as a soluble transfer base.

Typical Scavenging Conditions: 3 equiv relative to substrate, 0.5–2 h, 20 °C

Compatible Solvents: DCM (3.0 mL/g), DCE (3.0 mL/g), THF (2.8 mL/g), DMF (2.9 mL/g)

MP-Carbonate resin is a macroporous polystyrene anion-exchange resin that is a resin-bound equivalent of tetraalkylammonium carbonate. MP-Carbonate may be used as a general base to quench reactions, neutralize amine hydrochlorides or to scavenge a variety of acidic molecules such as carboxylic acids or acidic phenols.

Removal of excess carboxylic acids or acidic phenols, e.g. phenol or nitrophenol, from solution generally requires 3–4 equiv of MP-Carbonate. Removal of excess hindered phenol requires larger amounts of resin, typically up to 5-fold excess of MP-Carbonate. Complete removal takes from 30 minutes to 2 hours. Upon completion of the scavenging, the resin is rinsed three times with a suitable solvent, including DCM, THF, or EtOH. Representative acids and phenol scavenging examples are presented in **Table 1**.

MP-Carbonate is also very useful for neutralizing trialkylammonium salts, e.g. hydrochlorides and trifluoroacetates, to generate the free base. Applications include neutralizing reactants, products and ammonium salts of volatile amines, e.g. DIEA or TEA, produced in a chemical transformation. The latter case allows for neutralization and amine removal in the concentration step, circumventing an aqueous workup. In cases where the ammonium salt is insoluble, a catalytic amount of DIEA (0.05–0.1 equiv) can be added as a soluble transfer base.

PART NUMBER	QUANTITY	
800267	10 g	
800268	25 g	
800269	100 g	
800314	1000 g	



#### REPRESENTATIVE PROCEDURE:

## Neutralization of Amine Hydrochloride Salt

**Reaction:** Ephedrine hydrochloride salt (1 equiv) was converted to the free amine with MP-Carbonate (4 equiv) in DCM or methanol for one hour. Since ephedrine hydrochloride is not soluble in DCM, a catalytic amount of DIEA (0.05 equiv) was added as a transfer base and was removed during sample concentration.

**Reaction Workup:** The resin was removed by filtration and washed 2 x with DCM. The filtrate was concentrated to give ephedrine in 100% yield (MeOH) and 82% yield (DCM) (NMR purity: 100%).

#### REFERENCES

1. Parlow, J. J.; Naing, W.; South, M. S.; Flynn, D. L. Tetrahedron Lett. 1997, 38, 7959.

Table 1. Comparative Scavenging Times in Dichloromethane

Substrates	MP-Carbonate (equiv)	Time (h)	% Scavenged
Benzoic acid	3	1	100
Hexanoic acid	4	1	100
Bromobenzoic acid	3	1	100
Phenol	4	1	100
Nitrophenol	2	1	100
2-Allylphenol	6	1	93
2,6- Dimethylphenol	7	1	80

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