

# CombiZorb MP-Sulfonyl chloride

### Structure:



#### **Application Examples:**

Representative procedure: to a vial containing 0.6 gram MP-Sulfonyl chloride (1 mmol), 5 mmol n-PrOH and 3 mL of dichloromethane/pyridine (2:1) were added under nitrogen. The mixture was magnetically stirred for 4 hours at room temperature before filtration. The solid was then washed with 2mL MeOH, 3mL dichloromethane, 2mL MeOH, 3mL dichloromethane, and then 2 mL acetonitrile. A solution of piperidine (2 mmol) and N, N-di isopropylethylamine (DIEA, 5 mmol) in acetonitrile (3 mL) was added to the solid obtained above, and the mixture was agitated under nitrogen at 50-60°C for 16 hours. 1.5 g of MP-Isocyanate (2 mmol) was then added and the mixture was agitated at room temperature for another 2 hours before filtration. The corresponding N-propyl piperidine was obtained in the filtrate.

#### **Synthesis of Tertiary Amines**



#### **Recommended Storage:**

Keep cool and dry under argon,  $<25^{\circ}$ C. This product is highly reactive with water. To prevent exposure to moisture, it is recommended that you handle the packing in an inert atomosphere such as argon or nitrogen. In addition, it is recommended that the bottle be filled with argon and securely recapped, after use, to insure the best storage conditions.

#### **Reference:**

1. W. Huang, B. He Chin. J. Reactive Polymers 1992, 1, 61

2. T. Takahashi, S. Ebata and T. Doi *Tetrahedron Lett.* 1998, 39, 1369

3. E. W. Baxter, J. K. Rueter, S. Nortey and A. B. Reitz *Tetrahedron Lett.* 1998, 39, 979



#### **Characterization:**

Spherical, macroporous polystyrene/DVB particle; Density: 0.6 g/ml; Particle Size: 30-100 um; swelling: 30-50 % in THF or  $CH_2Cl_2$ .

#### **Capacity:**

1.6-2.0 mmol/g (determined by uptake titration of chlo-ride)

#### **Application:**

Scavenging alcohols, phenols, and amines, more widely used as solid phase reagents in some "catch and release" applications[2,3].

### Typical Conditions to make solid support tosylates:

5 equiv. alcohols relative to the MP-Sulfonyl chloride; 2-5 h, room temperature. The scavenger is compatible with more common solvents than gel-type polystyrene based analogues, including THF, dichloromethane, DMF and acetonitrile. Pyridine or other tertiary amines are needed to catalyze reaction and neutralize the acid formed. In general, it is unnecessary to swell the particle to get access to the functional sites.

#### Table: Synthesis of Tertiary amines from Alcohols

RCH <sub>2</sub> OH	R <sup>1</sup> R <sup>2</sup> NH	$\mathbf{R}^{1}\mathbf{R}^{2}\mathbf{NCH}_{2}\mathbf{R}$	Solvent for Loading	Yield (%)
n-PrOH	HN	n-PrN	DCM/ Pyridine(2:1)	85%
n-PrOH	HNO	n-PrN_O	DCM/ Pyridine(2:1)	87%
EtOCH <sub>2</sub> CH <sub>2</sub> OH	HNNMe	EtOCH <sub>2</sub> CH <sub>2</sub> N_NMe	DCM/ Pyridine (2:1)	83%
EtOCH <sub>2</sub> CH <sub>2</sub> OH	HN_NMe	EtOCH <sub>2</sub> CH <sub>2</sub> N_NMe	Acetonitrile/ Pyridine (2:1)	78%

Determined by GC

## **Certificate of Analysis**

Product Name	CombiZorb MP-Sulfonyl chloride
Lot Number	

Functional Groups -SO<sub>2</sub>Cl

Test Specification Result **Off-white or light yellow** Pass Appearance >1.6 mmol/g Capacity Swelling in dichloromethane <50% **Particle Size** >90% at 25 to 130 µm Pass Non-volatile Extractible <0.5 % Pass (by dichloromethane) IR spectroscopy Consistent with proposed structure Pass

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Certified By \_\_\_\_\_

Quality Control Date\_\_\_\_\_