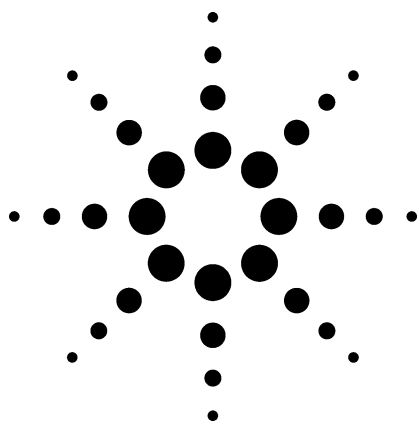
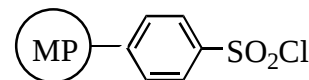




CombiZorb MP-Sulfonyl chloride

Structure:



Characterization:

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

Capacity:

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

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

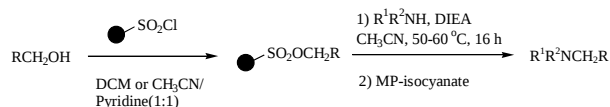
RCH_2OH	$\text{R}'\text{R}''\text{NH}$	$\text{R}'\text{R}''\text{NCH}_2\text{R}$	Solvent for Loading	Yield (%)
n-PrOH		n-PrN	DCM/ Pyridine(2:1)	85%
n-PrOH		n-PrN	DCM/ Pyridine(2:1)	87%
$\text{EtOCH}_2\text{CH}_2\text{OH}$		$\text{EtOCH}_2\text{CH}_2\text{NMe}$	DCM/ Pyridine (2:1)	83%
$\text{EtOCH}_2\text{CH}_2\text{OH}$		$\text{EtOCH}_2\text{CH}_2\text{NMe}$	Acetonitrile/ Pyridine (2:1)	78%

Determined by GC

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°C. This product is highly reactive with water. To prevent exposure to moisture, it is recommended that you handle the packing in an inert atmosphere 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



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Certificate of Analysis

Product Name **CombiZorb MP-Sulfonyl chloride**

Lot Number _____

Functional Groups **-SO₂Cl**

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

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Certified By _____

Quality Control Date_____