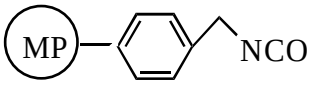
CombiZorb MP-Isocyanate

Structure:



Characterization:

Spherical, macroporous polystyrene/DVB particle; Density: 0.6 g/ml; Particle Size: 30-100 µm; swelling: 20-30 % in THF or CH_2Cl_2.

Capacity:

0.9-1.2 mmol/g (determined by benzylamine uptake).

Application:

Scavenging primary and secondary amines, anilines, hydrazines[1], and other nucleophiles.

Typical Conditions:

2-4 equivalents relative to the nucleophiles; 2-5 h, ambient temperature; shake or gentle agitation. The scavenger is compatible with more common solvents than gel-type polystyrene based analogues, including THF, Toluene, DMF, i-PrOH and even methanol (may use up to 50-60% loading capacity in methanol). As primary and secondary amines are substantially more reactive than alcohol towards isocyanate, they can be selectively removed in presence of alcohols. In general, it is unnecessary to swell the particle to get access to the functional sites.

Table 1. Scavenging of a variety of nucleophiles with 2.5 equiv. MP-Isocyanate

Nucleophile	Solvent	Temp (°C)	Time (h)	Scavenged (%)[A]
benzylamine	CH_2Cl_2	20	0.5	>99%
benzylamine	acetonitrile	20	0.5	>99%
benzylamine	i-PrOH	20	0.5	92%
benzylamine	MeOH	20	0.5	91%
morpholine	THF	20	1	>99%
1-methyl piperazine	THF	20	1	>99%
tryptamine	THF	20	1	94%
phenyl hydrazine	THF	20	1	>99%
aniline	THF	50	1	75%

A) Determined by GC

Application Example:

0.6 mL of Benzylamine in dichloromethane (0.5M) was mixed with 0.4 mL of phenyl isocyanate in dichloromethane (0.5M) and an extra 0.5 mL dichloromethane in a vial. The mixture was shaken at room temperature for 1 hour. 1mL MeOH and 0.4g CombiZorb MP-Isocyanate was added; the mixture was shaken for 2 more hours at room temperature before filtration. GC analysis of the filtrate showed only peaks of phenyl benzyl urethane and internal standards. Upon evaporation of solvents, the phenyl benzyl urethane was obtained in 87% yield.

Recommended Storage:

Keep cool and dry under argon, <25°C

Reference:

For general use of solid-supported isocyanates as scavengers, see:

1. R. J. Booth & J. C. Hodges J. Am. Chem. Soc., 1997, 119, 4882.

2. S. W. Kaldor et al. Tetrahedron Lett.,1996, 37, 7193

3. J. Rebek et al. J. Am. Chem. Soc. 1975, 97, 4407

For the application of this unique scavenger, see:

4. Q. Wang et al. at ACS National Meeting at New Orleans, 1999, Aug. 22-26, Organic Div.; Paper # 337

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Certificate of Analysis

Product Name **CombiZorb MP-Isocyanate**

Lot Number _____

Functional Groups **-NCO**

Test	Specification	Result
Appearance	Off-white or light yellow	Pass
Capacity	>0.9 mmol/g	
Swelling in dichloromethane	<50%	
Particle Size	>80% at 25 to 130 µm	Pass
Extractible	<0.2 AU/g/mL	Pass
IR spectroscopy	Consistant with proposed structure	Pass

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Certified By _____ **Quality Control Date**_____