

# CombiZorb **MP-Sulfonyl** hydrazide

### Structure:



#### **Characterization:**

Spherical, macroporous polystyrene/DVB particle; Density: 0.6 g/ml; Particle Size: 30-100 um; swelling: 30-50 % in THF or CH<sub>2</sub>Cl<sub>2</sub>.

#### **Capacity:**

1.5-2.5 mmol/g (determined by uptake of 2,4-dichloro benzaldehyde and confirmed by elemental analysis of the derivative)

#### Application:

Scavenging aldehydes and ketones and used as solid phase reagents in some "catch and release" applications[1, 2].

#### **Typical Conditions:**

3 equiv. relative to the aldehydes or ketones to be removed; 2-16 h, 20-60°C ; a small amount of acid catalyst may help in some cases; 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. In general, it is unnecessary to swell the particle to get access to the functional sites.

#### Table: Scavenging of a aldehydes and ketones with MP-Sulfonylhydrazide (3 equiv.RT)

	Solvent	Catalyst	Time(h)	%residue
benzaldehyde	$CH_2Cl_2$	none	2	<1
benzaldehyde	MeOH	none	2	<1
hexanal	$CH_2Cl_2$	none	2	<1
cyclohexanone	$CH_2Cl_2$	none	2	19
cyclohexanone	$CH_2Cl_2$	acetic acid	2	<1
cyclohexanone	MeOH	none	2	5
acetophenone	$CH_2Cl_2$	acetic acid	2	16
acetophenone	$CH_2Cl_2$	acetic acid	16	<1
acetophenone	MeOH	acetic acid	2	3
pentanedione	MeOH	none	2	4
Determined by CC				

Determined by GC

#### **Application Examples:**

Representative procedure: phenyl hydrazine (1 mmol) was dissolved in 2mL of MeOH in a vial, 1.5 mmol 2,4pentadione was added into the solution at room temperature under N<sub>2</sub>. The vial was sealed and shaken mildly for 1 h before 0.6g (1 mmol) CombiZorb MP-Sulfonyl hydrazide was added. The vial was sealed under N<sub>2</sub> again and mildely shaken for another two hours. The mixture was then filtered and the solid was washed with 1 mL MeOH. The filtrate was combined and pure 1phenyl 2,4 di-methyl pyrazole was obtained upon evaporation of MeOH. The purity was determined by GC.

#### **Pyrazole**



R = phenyl, 4-methoxyphenyl, m-tolyl;

Unlike gel-type polystyrene scavengers, macroporous scavengers can be used in alcohols with good efficiency.

#### **Recommended Storage:**

Keep cool and dry under argon,  $<5^{\circ}$ C.

#### **Reference:**

#### For general use of solid-supported sulfonyl hydrazides as scavengers, see:

1. Y. Hu, S. Baudart and J. Porco, Jr.; J. Org. Chem. 1999, 64, 1049

2. O. Galioglu and A. Auar; Eur. Polym. J. 1989, 25,313

#### For the application of this unique scavenger, see:

3. Q. Wang et al. at ACS National Meeting at San Francisco, 2000, March. 26-30, Organic Div.; Paper # 182



## **Certificate of Analysis**

Product Name	CombiZorb	MP-Sulfonyl	hydrazide
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Lot Number

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

Test	Specification	Result
Appearance	Off-white or light yellow	Pass
Capacity	>1.5 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

FOR RESEARCH AND DEVELOPMENT USE ONLY

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

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