

#### **Characterization:**

Spherical, ultra-pure silica; Density: 1.3 g/ml; Particle Size: 20-80 µm; no swelling.

#### Capacity:

1.2-1.6 mmol/g (determined by acid/base titration and confirmed mmol by reaction with 4-chlorobenzoyl chloride).

#### **Application:**

Scavenging acids, acid chlorides, acid anhydrides, chloroformates, isocyanates<sup>1,3</sup>, aldehydes<sup>2</sup>, and many other electrophiles.

# CombiZorb S-Triamine Structure:

#### **Typical Conditions:**

2-3 equivalents relative to acids, and isocyanates, 3-5 equivalent relative to acid chlorides, anhydrides, chloroformates and aldehydes; 0.5-2 h, 20-30°C (except for aldehydes with which mild heating and a small amount of acid catalyst may be needed); shake or gentle agitation; compatible with any solvents used for your reactions, no swelling needed. In general, there is no need to quench the reaction mixture with water when using scavengers, however, if water is used, this scavenger is compatible with an aqueous solution.

#### **Recommended Storage:**

Keep under N2 in a dry and cool place.

Table 1. Scavenging of a Variety of Electrophiles

Electrophile	CombiZorb S-Triamine (equiv.) <sup>A</sup>	Solvent	Conditions	Scavenged (%) <sup>B</sup>
4-chlorobenzoyl chloride	4	$\mathrm{CH_{2}Cl_{2}}$	1h, 20°C	>99%
2-phenylbutyryl chloride	4	$\mathrm{CH_{2}Cl_{2}}$	1h, 20°C	>99%
Phenyl chloroformate	4	$\mathrm{CH_{2}Cl_{2}}$	1h, 20°C	>99%
Chloroacetic anhydride	4	$\mathrm{CH_{2}Cl_{2}}$	1h, 20°C	>99%
Phenyl isocyanate	2	$\mathrm{CH_{2}Cl_{2}}$	1h, 20°C	>99%
Benzaldehyde	3	THF/MeOH (1:2)	1h, 60°C	>99%

A) Relative to electrophiles without use of additional base

#### **Application Example:**

Benzylamine (0.4 mmol) and CombiZorb S-Tertiary amine (1.4 g, 1.2 mmol) were premixed in a vial with 2 mL CH<sub>2</sub>Cl<sub>2</sub>, 4-chlorobenzoyl chloride (0.6 mmol) was added into the vial at room temperature and gently stirred for 1 h. CombiZorb S-Triamine (0.4 g, 0.6 mmol) plus 1 mL acetonitrile was added into the reaction mixture and stirred for 1 h. The solid scavengers were removed by filtration and washed twice with 0.5 mL CH<sub>2</sub>Cl<sub>2</sub>. All filtrates were collected and solvents were evaporated to give benzyl 4-chlorobenzamide in 95% yield with purity >99% (determined by GC).

#### Reference:

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

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

2. D. L. Flynn, et al. J. Am. Chem. Soc., 1997, 119, 4874.

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

3. L. A. Thompson, et al. at Lake Tahoe Symposium on Molecular Diversity, Lake Tahoe, CA. 1999, Jan. 28.

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



B) Determined by GC

## **Certificate of Analysis**

Product Name CombiZorb S-Triamine					
Lot Number					
Functional Groups -NH, -N	H <sub>2</sub>				
Test	Specification	Result			
Appearance	Off-white	Pass			
Capacity	>1.2 mmol/g				

>80% at 30 to 70  $\mu m$ 

<0.2 AU/g/mL

**Pass** 

**Pass** 

FOR RESEARCH AND DEVELOPMENT USE ONLY

Particle Size

Non-volatile extractible

Certified By	Quality Control Date
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