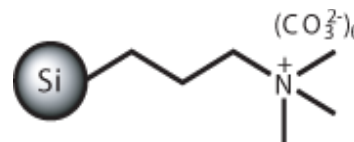


Application Note P1.0

TFA Removal using SiliaPrep Carbonate

Specifications

Product Name:	SiliaPrep Carbonate (Si-CO ₃)
Product Number:	SPE-R66030B-xxx
Active Loading:	0.7 mmol/g
Silica Characteristics:	40-63 μm, 60 Å Off-white powder
Solvent Compatibility:	All solvents, aqueous and organic
Prolonged Storage:	Keep dry
Related Publications:	1) Org. Lett., 4(7) 2002, 1167 2) Org Lett., 5(24) 2003, 4721



Typical Applications:

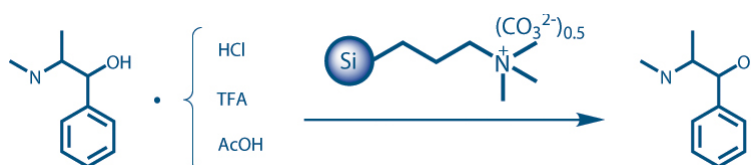
- TFA Removal
- Amine Free Basing
- Scavengers for acids
 - Acidic Phenols (including HOBt)
 - Boronic Acids
 - Carboxylic Acids
 - Imidazoles
- General Base (quenching reaction)

Description of SiliaPrep Carbonate

SiliaPrep Carbonate (or Si-CO₃) is the silica bound equivalent of tetramethyl ammonium carbonate. It can be used as a general base to quench a reaction, to free base amines in their ammonium salt form and to scavenge acids and acidic phenols, including HOBt, which is widely used in amide coupling reactions.

SiliaPrep Carbonate is also very efficient at scavenging boronic acids mainly used in coupling reactions (especially Suzuki): 99% removal of phenylboronic acid with one equivalent in DCM at room temperature after 1h.

APPLICATION: TFA Removal-Amine Free Basing



TYPICAL EXPERIMENTAL PROCEDURE

Amine free basing using SiliaPrep Carbonate

1. SiliaPrep Carbonate (2-4 eq. of Si-CO₃ in respect to the amine) is conditioned with THF.
2. Load the amine solution (in THF) onto the SiliaPrep Carbonate.
3. Free salt amine is eluted with THF under gravity.

Note: Other solvents can be used.
Related publication: Org. Lett., 4 (7) 2002, 1167

Amine free basing using SiliaPrep Carbonate

Amine salts		Yield ^A (%)	Purity ^B (%)
Ephedrine •	HCl	98.7	94.4
	TFA	100.0	98.9
	AcOH	100.0	99.2

^A Yields refer to the mass of isolated product.
^B Purity determined by GC-FID.

SiliaPrep Carbonate vs SiliaBond® Carbonate (bulk)

For your convenience SiliCycle offers supported carbonate either in cartridge and bulk formats. Most of the time SiliaPrep SPE Cartridges allow optimal scavenging efficiency explained by an higher number of equivalents/theoretical plates at the same time (see results below). Among the advantages of using SiliaPrep Carbonate is the possibility of automation, the rapidity of execution, and the higher efficiency for the same number of equivalents.

APPLICATION: HOBt Scavenging

TYPICAL EXPERIMENTAL PROCEDURE

Scavenging HOBt with SiliaBond Carbonate (bulk)

1. Add 2-4 eq. of SiliaBond Carbonate to the HOBt solution (in DMF).
2. Stir for 1 hour at room temperature.
3. Remove the SiliaBond® Carbonate by filtration rinsing with DMF.
4. Solvent evaporation gives the HOBt free solution.

Scavenging HOBt with SiliaPrep Carbonate

1. SiliaPrep Carbonate (2-4 eq. of Si-CO₃ in respect to HOBt) was conditioned with DMF.
2. Onto the SiliaPrep™ Carbonate was loaded the HOBt containing solution.
3. Amide (compound of interest) was eluted with DMF.
4. Solvent evaporation lead to the HOBt free solution.

Note: Other solvents can be used.
Related publication: Org. Lett., 5 (24) 2003, 4721

HOBt Scavenging with SiliaBond & SiliaPrep Carbonate

Si-CO ₃ # eq.	Reaction Time	Final HOBt concentration ^a	Reactivity
3	5 min.	32 ppm	99.4%
	1 hour	32 ppm	99.4%
	SiliaPrep	< 5 ppm	100%
4	5 min.	22 ppm	99.6%
	10 min.	22 ppm	99.6%
	1 hour	21 ppm	99.6%
	SiliaPrep	< 5 ppm	100%

^aDetermined by GC-MS. Initial HOBt concentration: 5 000 ppm in DMF.



Ordering Information

SiliaPrep Carbonate Formats

Formats	Qty/ Pk	Product Number
SiliaPrep SPE Cartridges		
1 mL/50mg	100	SPE-R66030B-01B
1 mL/100mg	100	SPE-R66030B-01C
3 mL/200mg	50	SPE-R66030B-03G
3 mL/500mg	50	SPE-R66030B-03P
6 mL/500mg	50	SPE-R66030B-06P
6 mL/1g	50	SPE-R66030B-06S
6 mL/2g	50	SPE-R66030B-06U
12 mL/2g	20	SPE-R66030B-12U
25 mL/5g	20	SPE-R66030B-20X
SiliaPrep Large Reservoir Volume SPE Cartridges		
10 mL/200mg	50	SPC-R66030B-10G
10 mL/500mg	50	SPC-R66030B-10P



SiliaPrep Carbonate Formats

Formats	Qty/ Pk	Product Number
SiliaPrep 96 Well Plates		
2 mL/50mg	1	96W-R66030B-B
2 mL/100mg	1	96W-R66030B-C
2 mL/200mg	1	96W-R66030B-G
Bulk		
10g, 25g, 100g, 500g, 1kg, 5kg, 10kg, 25kg		R66030B

These products are also available in 24, 48 and 384 well plates.

Contact us for a quote.