ORGANIC LETTERS

2006
Vol. 8, No. 15
3259−3262

Catalyst-Free Chemoselective *N-tert*-Butyloxycarbonylation of Amines in Water

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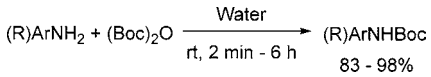
Received May 7, 2006

ABSTRACT



Catalyst-free *N-tert*-butyloxycarbonylation of amines in water is reported. The *N-t*-Boc derivatives were formed chemoselectively without any isocyanate, urea, *N,N*-di-*t*-Boc, and *O*/*S*-*t*-Boc as side products. Chiral amines, esters of α-amino acids, and *β*-amino alcohol afforded optically pure *N-t*-Boc derivatives. Amino alcohol and 2-aminophenol afforded the *N-t*-Boc derivative without oxazolidinone formation. Selectivity was observed during competition of aromatic amine vs aliphatic amine, amine vs amino acid ester, amine vs amino alcohol, and primary amine vs secondary amine.

The presence of the amine functionality in a wide range of biologically active compounds makes protection of amines an important and frequently needed exercise in synthetic organic/medicinal chemistry.[1] Acylation[2] is an easy method of protection but the harsh reaction conditions[3] required to regenerate the amine from the acylated derivative are not suitable for a multifunctional substrate and a protecting group that is cleavable under mild conditions is required. As the *N-tert*-butylcarbamates are stable toward a variety of routinely adopted experimental conditions such as in the presence of nucleophiles but are easily converted to the parent amines under mild acidic conditions,[1a] the *N-tert*-butyloxycarbonylation is an elegant strategy to protect amines.[4,5] Amines are converted to *N-t*-Boc derivatives by reaction with (i) di-*tert*-butyl dicarbonate [$(Boc)_2O$] in the presence of DMAP[6]/inorganic bases,[7] (ii) 4-(dimethylamino)-1-*tert*-butoxycarbonylpyridinium chloride[8]/tertafluoroborate[9] in aq NaOH, (iii) 2-*tert*-butyloxycarbonyloxyimino-2-phenylacetonitrile in the presence of Et_3N in H_2O−dioxane,[1a] (iv) *tert*-butyl-2-pyridyl carbonate in the presence of Et_3N in H_2O−DMF,[1a] and (v) *tert*-butyl-1-chloroalkyl carbonates in the presence of K_2CO_3 in H_2O−THF.[1a] These methodologies have various drawbacks such as the requirement of long reaction times, special efforts to prepare the *tert*-butyloxycarbonylation reagents,[1a,8,9] and auxiliary substances (e.g., solvents, bases, etc.). The high toxicity of DMAP[13] is a serious concern for the use of DMAP and the *tert*-butyloxycarbonylation reagents[8,9] derived from DMAP. Further, the base-catalyzed reactions are often associated with the forma-

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10.1021/ol0611191 CCC: $33.50 © 2006 American Chemical Society
Published on Web 06/20/2006

tion of isocyanate,[6d,14] urea,[6d] and *N*,*N*-di-Boc derivatives.[6d,15] The limited number of Lewis acid-catalyzed procedures[16] circumvented the problem of side reactions but some of them are not devoid of other drawbacks such as long reaction time, the need to use solvent,[13a−d] and hazards (e.g., the use of sulfuric acid at 500 °C to prepare yttria-zirconia,[16a] $ZrCl_4$ is moisture sensitive and decomposes on storing liberating HCl fumes), etc.

The current environmental concerns encourage the development of "greener" conditions, where possible, and the tight legislation on the maintenance of greenness in synthetic processes insists on preventing generation of waste, avoiding the use of auxiliary substances (e.g., organic solvents, additional reagents)[18] and minimizing the energy requirement.[19] The use of water as reaction medium[20] has received considerable attention in the context of green chemistry for several reasons: (i) it is cheap, safe, and environmentally benign, (ii) reactions in aqueous medium eliminate the additional efforts in making the substrates and reagents dry before use and thus reduce/eliminate the consumption of drying agents, energy, and time, (iii) and the unique physical and chemical properties of water can be utilized to realize reactivity or selectivity that cannot be attained in organic solvents.[21] In pursuit of our recent efforts to develop environmentally friendly synthetic methodologies by carrying out reactions in water,[22] we observed that there are limited examples of organic reactions that have been carried out in water in the absence of any catalyst[23] but there is no report of catalyst-free *N*-*tert*-butyloxycarbonylation of amines in water.

Herein we report an efficient method for chemoselective *N*-*tert*-butyloxycarbonylation of amines in water at room temperature in the absence of any acid/base catalyst. As comparable yields were obtained in using tap and distilled water, the reactions were carried out in tap water so that the efforts and energy consumption for preparing distilled water are avoided. The critical amount of water required was found to be 1 mL/mmol of the amine. The use of a lesser amount of water led to incomplete conversion but the use of >1 mL/mmol did not have a significant influence on the reaction rate and product yield. The products were isolated by filtration (for solid products) or extraction with EtOAc (for liquid products) and were of sufficient purity (spectral data) and did not require further efforts of purification. To address the question of whether the Lewis acid character of the glassware is contributing to the reaction, the *N*-*tert*-butyloxycarbonylation of aniline was carried out with $(Boc)_2O$ in a plastic vessel and the *N*-*t*-Boc derivative was obtained 92% yield after 0.5 h. Various aromatic, heteroaromatic, aryl alkyl, and alkyl amines were converted to the *N*-*t*-BOC derivatives in excellent yields after 2 min to 6 h (Table 1). No competitive formation of isocyanate,[6d,11] urea,[6d] and *N*,*N*-di-BOC[6d,12] was observed (IR, GCMS). Substrates having an OH/SH group (entries 6−8, 32, and 33) afforded the *N*-*t*-Boc derivatives without *O*/*S*-*t*-Boc formation (IR).[6d] The chemoselectivity was further demonstrated in the case of 2-aminophenol (entry 7) and amino alcohol (entry 33) that did not form oxazolidinone[6d,24] and aminoacetaldehyde dimethyl acetal (entry 28) that is sensitive to acids. Chiral amine (entry 29), esters of α-amino acids (entries 30−32), and amino alcohol (entry 33) gave optically pure *N*-*t*-Boc derivatives (as determined by optical rotation and comparison with literature values)[25] and demonstrated the mildness of this procedure.

The reactions were monitored by TLC and IR and could also be followed up visually. For solid amines, a clear solution was obtained after addition of $(Boc)_2O$ to the magnetically stirred suspension of the amine in water. Commencement of slow effervescence took place with concomitant formation of the *N*-*t*-Boc derivative (TLC). The formation of solid residue indicated the completion of the reaction (TLC, IR). In case of liquid amines, transparent droplets were formed in mixing the amine and $(Boc)_2O$ in water and a white emulsion appeared on the surface alongside the walls of the reaction vessel with slow effervescence. Finally, a white solid settled down and indicated the completion of the reaction (TLC, IR).

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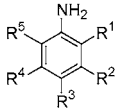
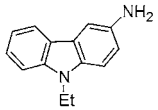
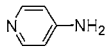
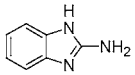
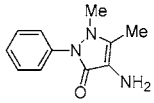
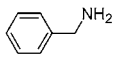
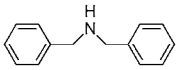
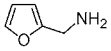
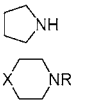
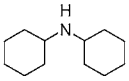
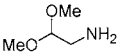
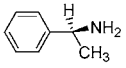
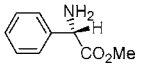
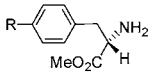
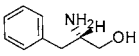
Table 1. *N-tert*-Butyloxycarbonylation of Amines in Water[a]

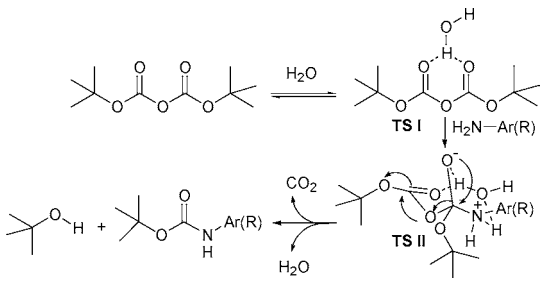
entry	substrate	time (h)	yield (%)[b,c]
			
1	$R^1 = R^2 = R^3 = R^4 = R^5 = H$	0.5	95
2	$R^1 = R^2 = R^4 = R^5 = H$; $R^3 = Me$	1	96
3	$R^1 = Me$; $R^2 = R^3 = R^4 = R^5 = H$	1.5	92
4	$R^1 = R^2 = Me$; $R^3 = R^4 = R^5 = H$	2	90
5	$R^1 = R^3 = R^5 = Me$; $R^2 = R^5 = H$	2	90
6	$R^1 = R^2 = R^4 = R^5 = H$; $R^3 = OH$	1	87
7	$R^1 = OH$; $R^2 = R^3 = R^4 = R^5 = H$	2	90
8	$R^1 = R^2 = R^4 = R^5 = H$; $R^3 = SH$	1	83
9	$R^1 = R^2 = R^4 = R^5 = H$; $R^3 = F$	1	90
10	$R^1 = R^2 = R^4 = R^5 = H$; $R^3 = Br$	1	86
11	$R^1 = Me$; $R^2 = R^4 = R^5 = H$; $R^3 = Br$	1.5	90
12	$R^1 = R^4 = R^5 = H$; $R^3 = F$; $R^2 = Cl$	6	92
13	$R^1 = R^2 = R^4 = R^5 = H$; $R^3 = OCH_2Ph$	1	90
14		0.5	92[d]
15		0.5	92
16		2	92
17		2	90[d]
18		5 min	96
19		5 min	92
20		0.5	95
21		2 min	90
22	X = CH_2; R = H	2 min	95
23	X = O; R = H	2 min	98
24	X = CH_2; R = $CH_2CH_2NH_2$	5 min	95
25	X = O; R = $CH_2CH_2NH_2$	5 min	90
26		20 min	90
27		2	90
28		10 min	95
29		10 min	96
30		1	91
31	 R = H	1	90
32	R = OH	1	90
33		20 min	92

[a] The amine (2.5 mmol) was treated with $(Boc)_2O$ (1.1 equiv) in water (2.5 mL) at room temperature (~30−35 °C). [b] Yield of the purified product. [c] The products were characterized by IR, NMR, and MS and all unknown compounds gave correct elemental analyses. [d] The reaction was carried out with 1.5 equiv of $(Boc)_2O$.

The role of water may be explained by Scheme 1.[26] Hydrogen bond formation between water and the carbonyl oxygen atoms of $(Boc)_2O$ causes "electrophilic activation" (TS **I**) making the carbonyl group more susceptible to nucleophilic attack. The oxygen atom of water in turn forms a hydrogen bond with the hydrogen atom of the amine and increases the electron density at the nitrogen atom (nucleophilic activation). Electrostatic attraction between the carbonyl group and the nitrogen atom forms the TS **II**. Intramolecular nucleophilic attack by the nitrogen atom on the carbonyl carbon followed by elimination of CO_2, ^{t}BuOH, and H_2O forms the carbamate.

The dual activation role of water through cooperative hydrogen bond formation (Scheme 1) accounted for the present observations (Table 1). The faster rate of reaction with aliphatic amines was due to their better nucleophilicity. However, the slower rate of carbamate formation with dicyclohexylamine (entry 27) was the result of steric hindrance of the cyclohexyl groups in the TS **II**. The longer time required for 2-aminophenol (entry 7) compared to that of 4-aminophenol (entry 6) may be due to the fact that in case of the former, intramolecular hydrogen bond formation

Scheme 1. Dual Activation Role of Water during Catalyst-Free *N-tert*-Butyloxycarbonylation of Amines



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between the *O*-H hydrogen atom and the nitrogen atom reduced the nucleophilicity of the NH_2 group. This intramolecular hydrogen bond formation impaired the hydrogen bond formation involving the *N*-H hydrogen atom and the oxygen atom of the water molecule in the TS **II** and decreased the reaction rate. The distinct involvement of the hydrogen bond between the *N*-H hydrogen of the amine and the oxygen atom of water in the TS **II** can be realized by comparison of entry 18 vs 20 and entry 29 vs 30. In 2-furfurylamine and methyl (*S*)-phenylglycinate, intramolecular hydrogen bond formation of the *N*-H hydrogen with the oxygen atom of the furan ring and the oxygen atom of the carbonyl group, respectively, should make the nitrogen atom more nucleophilic, but contrary to this expectation, the reaction rate was slower for these substrates. The decrease in the rate of reaction in the case of 2-furfurylamine and methyl (*S*)-phenylglycinate was due to the intramolecular hydrogen bond formation in these substrates that did not permit the hydrogen bond formation between the *N*-H hydrogen and the oxygen of the water molecule in the TS **II** to take place as efficiently as in the case of benzylamine and (*S*)-α-methylbenzylamine. As the amide carbonyl oxygen atom in 4-aminoantipyrine (entry 17) is more electron rich compared to the oxygen atom in 2-furfurylamine and the carbonyl oxygen atom of methyl (*S*)-phenylglycinate, the intramolecular hydrogen bond formation between the amide oxygen atom and the *N*-H hydrogen is more pronounced in 4-aminoantipyrine resulting in much decrease in the rate of carbamate formation. In case of 2-aminobenzimidazole (entry 16), the *N*-H hydrogen atom of the imidazole ring undergoes hydrogen bond formation with a separate water molecule, which in turn is involved in hydrogen bond formation with the nitrogen atom of the 2-NH_2 group and reduces its nucleophilicity. The trapping of a separate water molecule between the ring *N*-H and the 2-NH_2 competes with the formation of the TS **II** and contributes to retard the carbamate formation.

The difference in the rate of reaction of the amine group under various electronic and steric environments encouraged us to study the selective *N*-*t*-Boc formation during competitions of an aromatic amine vs an aliphatic amine, an amine vs ester of an amino acid, an amine vs an amino alcohol, and a primary amine vs a secondary amine. The reaction of aniline (1 mmol) and benzylamine (1 mmol) with $(Boc)_2O$ (1 mmol) afforded the corresponding *N*-*t*-Boc derivatives in 30% and 70% yields, respectively, after 5 min (GCMS). The *N*-*t*-Boc of (*S*)-α-methylbenzylamine was formed as the only product during the reaction of the mixtures of (i) (*S*)-α-methylbenzylamine (1 mmol) and methyl (*S*)-phenylglycinate (1 mmol) and (ii) (*S*)-α-methylbenzylamine (1 mmol) and (*S*)-phenylalalinol (1 mmol), separately, with $(Boc)_2O$ (1 mmol) in 90% and 92% isolated yields, respectively, after 10 min. The *N*-*t*-Boc-cyclohexylamine was formed exclusively when a mixture of cyclohexylamine (1 mmol) and dicyclohexylamine (1 mmol) was treated with $(Boc)_2O$ (1 mmol) for 60 min (IR, TLC comparison with authentic compounds). The preferential *N*-*t*-Boc formation of cyclohexylamine over dicyclohexylamine is due to the steric factor as otherwise the later is expected to form the *N*-*t*-Boc derivative selectively because a secondary amine is more nucleophilic than a primary amine. When a mixture of benzylamine (1 mmol) and dibenzylamine (1 mmol) was treated with $(Boc)_2O$ (1 mmol) for 5 min a 64:36 selectivity (GCMS) was observed in favor of the *N*-*t*-Boc of dibenzylamine. This result established that in the absence of an appreciable amount of steric effect, the *N*-*t*-Boc of the secondary amine is formed preferably. To further demonstrate this, a mixture of piperidine (1 mmol) and 1-(2-aminoethyl)-piperidine (1 mmol) was treated with 1 equiv of $(Boc)_2O$ for 1 min and the corresponding *N*-*t*-Boc derivatives were formed in a ratio of 67:33 (GCMS). Similarly the reaction of a mixture of morpholine (1 mmol) and 4-(2-aminoethyl)morpholine (1 mmol) with $(Boc)_2O$ (1 mmol) for 1 min exhibited a 88:12 selectivity in favor of *N*-*t*-Boc-morpholine (GCMS).

We have described herein an efficient methodology for water mediated *N*-*tert*-butyloxycarbonylation of amines at room temperature. The advantages such as the (i) catalyst-free and nonhazardous conditions, (ii) operation at room temperature, (ii) high yields, (iii) excellent chemoselectivity, and (iv) ease of product isolation/purification fulfill the triple bottom line philosophy of green chemistry[27] and make the present methodology environmentally benign.

Acknowledgment. We thank DST, New Delhi, India for financial support.

Supporting Information Available: Typical experimental procedure, spectral data of all compounds, scanned spectra of unknown compounds. This material is available free of charge via the Internet at http://pubs.acs.org.

OL0611191

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