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Short communication

Montmorillonite K 10 and montmorillonite KSF as new and reusable catalysts for conversion of amines to *N*-*tert*-butylcarbamates

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Abstract

Montmorillonite K 10 and montmorillonite KSF were found to be new and reusable catalysts for chemoselective conversion of amines to *N*-*t*-Boc derivatives at room temperature under solvent-free conditions without competitive formation of isocyanate, urea and *N*,*N*-di-*t*-Boc. Various aromatic, heteroaromatic and aliphatic amines afforded *N*-*t*-butylcarbamates in excellent yields on treatment with $(Boc)_2O$ after 5 min–2 h. Chiral amine and esters of α-amino acids afforded optically pure *N*-*t*-Boc derivatives in high yields. The catalytic efficiency of montmorillonite KSF was marginally inferior to that of montmorillonite K 10.

Keywords: Montmorillonite K 10; Montmorillonite KSF; Catalyst; *N*-*tert*-Butylcarbamates; Amines; Chiral α-amino acid esters; Di-*tert*-butyl dicarbonate; Chemoselective; Solvent-free; Room temperature

1. Introduction

Protection of amines is an important and frequently needed exercise in synthetic organic/medicinal chemistry [1]. Acylation [2] provides an easy process of protection but the harsh reaction conditions [3] required to regenerate the parent amine from the acylated derivative becomes detrimental for multifunctional substrate. The stability of *N*-*tert*-butylcarbamates under alkaline media and towards nucleophilic attack and the ease of deprotection under mild acidic conditions [1a] make *N*-*tert*-butoxycarbonylation an efficient strategy for protection of amines during synthesis of multifunctional target [4,5]. Conversion of amines to *N*-*tert*-butylcarbamates involves treatment with di-*tert*-butyl dicarbonate [$(Boc)_2O$] in the presence of DMAP [6]/inorganic bases [7], 4-dimethylamino-1-*tert*-butoxycarbonylpyridinium chloride [8]/tetrafluoroborate [9] in aqueous NaOH, 2-*tert*-butyloxycarbonyloxyimino-2-phenylacetonitrile in the presence of Et_3N in H_2O–dioxane [10], *tert*-butyl 2-pyridyl carbonate in the presence of Et_3N in H_2O–DMF [11], and *tert*-butyl 1-chloroalkyl carbonates in the presence of K_2CO_3 in H_2O–THF [12]. However, these methodologies have various drawbacks such as requirement of long reaction times, special efforts for preparation of *tert*-butoxycarbonylation reagents [8–12], auxiliary substances (e.g., solvents and other reagents) and potential hazards (e.g., the high toxicity of DMAP [13] does not qualify it and the *tert*-butoxycarbonylation reagents [8,9] derived from it to be safe for use). The base-catalysed reactions often lead to the formation of side products such as isocyanate [6d,14], urea [6d] and *N*,*N*-di-Boc derivatives [6d,15]. Recently, a few Lewis acid catalysts have been developed. These include yttria-zirconia in MeCN [16], $Zn(ClO_4)_2{\cdot}6H_2O$ [17]/$LiClO_4$ [18] in DCM and $ZrCl_4$ in MeCN [19]. Although these methods circumvented the problem associated with formation of the above mentioned side products, still they are not devoid of other drawbacks such as long reaction time, use of solvent and hazardness (e.g., preparation of yttria-zirconia involves use of sulphuric acid at 500 °C [16], perchlorates are strong oxidisers and explosive in nature [20] and $ZrCl_4$ is highly moisture sensitive, decomposes on storing and liberates corrosive HCl fumes), etc. These necessitate the development of new synthetic methodology.

2. Results and discussion

The side reactions under base-catalysed condensation may be explained in Scheme 1. Abstraction of proton form the amine by

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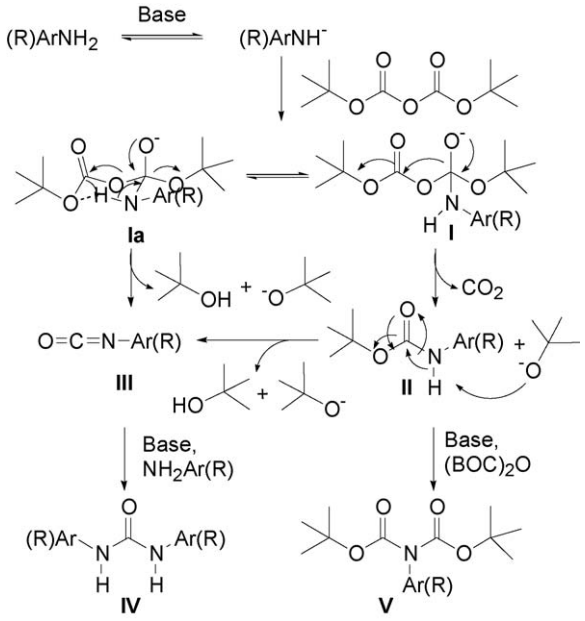
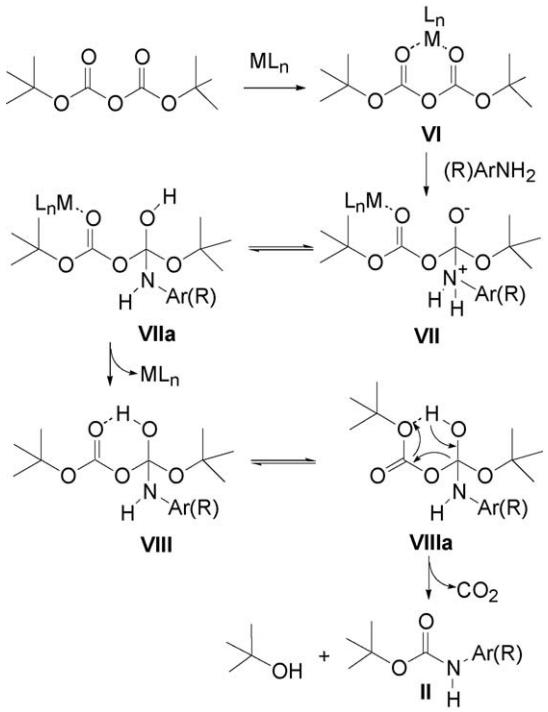


Scheme 1. Base-catalysed reaction of amines with $(Boc)_2O$.



Scheme 2. Lewis acid-catalysed reaction of amines with $(Boc)_2O$.

the base generates the amide anion that undergoes nucleophilic attack on $(Boc)_2O$ and generates the tetrahedral intermediate **I**. Elimination of CO_2 and $^tBuO^-$ form **I** leads to the formation of the *N*-*t*-Boc derivative **II**. The liberated $^tBuO^-$ undergoes proton exchange with the **II** to form the isocyanate **III** [21] that can further react with the starting amine in the presence of $^tBuO^-$ to the form urea derivative **IV**. In a parallel pathway, in the presence of $^tBuO^-$ **II** reacts with $(Boc)_2O$ to form the *N*,*N*-di-Boc derivative **V**. As in the transition state **Ia**, the *N*–H hydrogen and the oxygen atom of the liberated $^tBuO^-$ are so disposed that they can form a six-membered transition state through hydrogen bond formation, the isocyanate and/or *N*,*N*-di-BOC formation become unavoidable under base-catalysed reactions.

We reasoned that the side reactions may be eliminated if the in situ generation of $^tBuO^-$ can be avoided. We observed that in the limited examples of Lewis acid-catalysed reactions [16–19], the side products such as isocyanates, *N*,*N*-di-Boc derivatives and urea derivatives were not formed. A probable explanation for suppression of these side products under acid-catalysed reactions may be rationalized through Scheme 2. Coordination of the Lewis acid with $(Boc)_2O$ induces elcetrophilic activation via formation of the transition state structure **VI** which undergoes nucleophilic attack at one of the carbonyl carbons by the amine and generates the tetrahedral intermediate **VII**. Intramolecular proton transfer from the ammonium nitrogen atom to the oxyanioic site converts **VII** to **VIIa**. Intramolecular hydrogen bond formation leads to **VIII** and liberates the catalyst. Finally, the *N*-*t*-Boc amine is produced by rearrangement of **VIII/VIIIa** with the liberation of ^tBuOH instead of $^tBuO^-$.

Hence, we thought that the 'electrophilic activation' strategy should provide a better method of *N*-*tert*-butoxycarbonylation and introduced $Cu(BF_4)_2{\cdot}xH_2O$ as a new catalyst [22]. However, tight legislation on maintenance of greenness in synthetic pathways and processes demands to prevent waste, avoid use of auxiliary substances (e.g., solvents and additional reagents) and minimise energy requirement [23]. Thus, while designing new 'electrophilic activation' catalysts we kept in mind the aspect of maintaining greenness and were influenced by the awareness of the use of solid acids as environmentally friendly catalysts in organic synthesis [24]. Recently, we reported that commercially available montmorillonite K 10 efficiently catalysed the opening of epoxide rings by amines under solvent-free conditions and at room temperature [25] and planned to exploit the potential of montmorillonite clays (K 10 and KSF) as catalysts for conversion of amines to *N*-*t*-Boc derivatives.

Various aromatic, heteroaromatic, aliphatic and heterocyclic amines were treated with $(Boc)_2O$ (1 equiv) under solvent-free conditions at r.t. (~30–35 °C) in the presence of montmorillonite K 10 (Method A) and montmorillonite KSF (Method B). The *N*-*t*-Boc formation was completed after 5–120 min (Table 1). A few limited examples (entries 1–3, 6, 12 and 22) demonstrated that montmorillonite KSF was marginally inferior to montmorillonite K 10. No competitive side reactions such as the formation of isocyanate [6d,14], urea [6d] and *N*,*N*-di-BOC derivative [6d,15] were observed. Aliphatic amines were converted to the *N*-*t*-Boc derivative at a faster rate compared to that of aromatic amine. The *N*-*tert*-butyloxycarbonylation was compatible with various functional groups such as F, Cl, Br, OH, SH, OMe and OBn. However, substrates with strong electron-withdrawing group such as CN and NO_2 (entries 9 and 10) did not afford any significant amount of *N*-*t*-Boc derivative as these groups decreased the nucleophilicity of the nitrogen atom of the amine group. The progress of the reactions was monitored by TLC and IR. However, the reactions could also be followed up visually. In case of aromatic amines (solid amines),

Table 1
Montmorillonite K 10 and montmorilloniet KSF catalyzed formation of *N-tert*-butylcarbamates from various amines[a]

Entry	Amine	Time (min)	Yield[b] (%)
1	Aniline	15 (15)	100 (100)
2	4-Methoxyaniline	15 (20)	98 (90)
3	4-Methylaniline	30 (30)	95 (85)
4	4-Aminophenol	60	90
5	4-Aminothiophenol	60	92
6	4-Fluoroaniline	60 (60)	95 (95)
7	4-Bromoaniline	60	93
8	4-Chloroaniline	120	100
9	4-Nitroaniline	6 h	Nil[c]
10	4-Aminobenzonitrile	6 h	Nil[c]
11	2,4,6-Trimethylaniline	60	95
12	4-Aminopyridine	15 (15)	92 (90)
13	2-Aminobenzimidazole	60	90
14	4-Aminobenzothiazole	6 h	Nil[c]
15	Benzylamine	5	100
16	Dibenzylamine	10	90
17	Furfurylamine	15	100
18	Pyrrolidine	10	100
19	Morpholine	10 (15)	100 (100)
20	1-Amino-2-methyl-propan-2-ol	5	100
21	2,2-Dimethoxyethylamine	5	100
22	(*S*)-α-Methylbenzylamine	15	100
23	L-NH_2-Phg-OMe	30	90
24	L-NH_2-Phe-OMe	30	92
25	L-NH_2-Tyr-OMe	30	87
26	L-NH_2-Tyr-(OBn)OBn	45	90

[a]The substrate (2.5 mmol) was added to the magnetically stirred mixture of montmorillonite K 10/KSF (10%, w/w) and $(Boc)_2O$ (1 equiv) at r.t. under neat conditions. The figures in parentheses are the corresponding data for montmorillonite KSF-catalysed reactions.
[b] Yield of the isolated pure product.
[c] The starting material remained unchanged (TLC, GC–MS).

initially a clear solution was obtained after addition of amine to the magnetically stirred mixture of the catalyst and $(Boc)_2O$ with commencement of slow effervescence. Formation of solid residue indicated the completion of the reaction. For reactions that took long times (>30 min), although the effervescence could not be noticed distinctly, the reaction mixture solidified after completion of reaction. For aliphatic amines (liquid amines), an exothermic reaction took place immediately after addition of amine to the mixture of the catalyst and $(Boc)_2O$ with vigorous effervescence. The cessation of effervescence indicated completion of reaction. In case of α-methyl benzyl amine (liquid amine) (entry 22), solid residue formed after the reaction was completed. The reactions were easy to carry out and did not require aqueous work-up to isolate the product from the reaction mixture. The isolation of the products were achieved by dilution of the reaction mixture with EtOAc, filtration through a plug of cotton (to separate the catalyst) followed by evaporation of the solvent. In most of the cases, the products obtained after usual work-up were pure (spectral data). Wherever required, the crude products obtained after the usual work-up were triturated with EtOAc–hexane to afford the pure product. The catalyst could be easily recovered by putting the cotton plug in EtOAc in a beaker (25 mL) when the catalyst settled down. The cotton was removed and the EtOAc decanted off to recover the catalyst which was reused after activation on heating at 100 °C under reduced pressure (5 mm Hg) for 2 h and was found to retain its catalytic activity (see Section 4). Excellent chemoselectivity was observed for substrates having OH/SH group affording the corresponding *N-t*-Boc derivatives as the sole product and no significant *O*/*S-tert*-butyloxycarbonylation took place (IR) [6d,26]. Reaction with 2-methyl-2-propanol-1-amine (entry 20) resulted chemoselective formation of the *N-t*-Boc derivative without formation of oxazolidinone [6d,27]. The chemoselective *N-tert*-butylcarbamate formation was further demonstrated by the reaction with amino acetaldehyde dimethyl acetal (entry 21) that is acid sensitive. The mildness of the protocol was exemplified by the conversion of chiral amine (entry 22) and α-amino acid esters (entries 23–26) to the corresponding optically pure (as determined by the comparison of the optical rotation with authentic samples) [16,28–31] *N-t*-Boc derivatives in excellent yields. Recently it has been reported that montmorillonite catalysed the dealcoholysis of carbamates leading to the formation of isocyanates [32]. However, under the present study no isocyanate formation was observed (IR, GC–MS).

Following representative examples established the superiority of montmorillonite clays over the reported catalysts. Reaction of aniline afforded 90% yield after 14 h in the presence of yttria-zirconia (20%, w/w) in MeCN [16] and 92% yield after 12 h in DCM in the presence of $Zn(ClO_4)_2$ (5 mol%) [17]. Quantitative yields were obtained after 5 min in using montmorillonite K 10/KSF (10%, w/w) under solvent-free conditions. Use of $LiClO_4$ (20 mol%) afforded 86% yield of *N-t*-Boc morpholine after 5 h in DCM [18] and 85% yield was obtained after 4 h in MeCN yttria-zirconia (20%, w/w) [16] compared to quantitative yield obtained after 10 min under solvent-free condition in the presence montmorillonite K 10 (10%, w/w). Aminoacetaldehyde dimethyl ether was converted to *N-t*-Boc derivative in 90% yield after 16 h during $Zn(ClO_4)_2$-catalysed (5 mol%) reaction [17] but use of montmorillonite K 10 (10%, w/w) provided quantitative yield after 5 min. *N-tert*-Butoxycarbonylation of (*R*)-α-methylbenzylamine was achieved in 97% yield after 2.5 h in the presence of $Zn(ClO_4)_2$ (5 mol%) under neat condition [17] and *N-t*-Boc of (*S*)-α-methylbenzylamine was obtained in 85% yield after 5 h in DCM during $LiClO_4$ (20 mol%)-catalysed reaction [18]. Compared to these results, quantitative yields were obtained with (*S*)-α-methylbenzylamine under solvent-free conditions after 15 min in the presence of montmorillonite K 10 (10%, w/w). Reaction of (*S*)-phenylglycine methyl ester provided 88% yield after 5 h in DCM in the presence of $LiClO_4$ (20 mol%) [18]. In comparison to this, 100 and 95% yields were obtained after 15 min in the presence of montmorillonite K 10 (10%, w/w) and montmorillonite KSF (10%, w/w), respectively, under solvent-free conditions.

3. Conclusion

We have discovered that montmorillonite clays (K 10 and KSF) are highly efficient and reusable catalysts for *N-tert*-butoxycarbonylation of amines. The advantages include: (i) use of commercially available, inexpensive and environmentally friendly catalysts, (ii) solvent-free [33] and room temperature

reaction conditions, (iii) short reaction times, (iv) high yields and (iv) ease of product isolation/purification by non-aqueous work-up. The present methodology fulfils triple bottom line philosophy [34] of green chemistry.

4. Experimental

The ^{1}H and ^{13}C NMR spectra were recorded on Bruker Avance DPX 300 (300 MHz) spectrometer in $CDCl_3$ using TMS as internal standard. The IR spectra were recorded on Nicolet Impact 400 spectrometer as KBr pellets for solid and neat for liquid samples. The reactions were monitored by TLC (silica gel-G) and Shimadzu QP 5000 GCMS. Evaporation of solvents was performed at reduced pressure, using a Büchi rotary evaporator.

4.1. Typical procedures for synthesis of N-tert-butylcarbamates

Method A: Aniline (0.235 g, 2.5 mmol) was added to a magnetically stirred mixture of montmorillonite K 10 (25 mg, 10%, w/w) and di-*tert*-butyl dicarbonate (0.545 g, 2.5 mmol, 1.0 equiv) at room temperature. The mixture was stirred until completion of reaction (TLC, 15 min), diluted with EtOAc (10 mL) and filtered through a plug of cotton. The residue was washed with EtOAc (2 × 5 mL) and the combined filtrates were concentrated under vacuum to afford *tert*-butyl-*N*-phenylcarbamate (white solid, 0.485 g, 100%, entry 1; Table 1), identical (IR, ^{1}H and ^{13}C NMR and EIMS) to an authentic sample [6d]. The cotton plug containing the catalyst was dipped into EtOAc (15 mL) in a beaker (25 mL) when the montmorillonite K 10 settled down to the bottom of the beaker. The cotton was removed and the EtOAc decanted off. The recovered catalyst after being air dried and treated at 100 °C for 2 h under reduced pressure (5 mmHg) was reused to afford 95% yields during the reaction of aniline with di-*tert*-butyl dicarbonate.

Method B: Treatment of aniline (0.235 g, 2.5 mmol) with di-*tert*-butyl dicarbonate (0.545 g, 2.5 mmol, 1.0 equiv) in the presence of montmorillonite KSF (25 mg, 10%, w/w) at room temperature followed by usual work-up as described for Method A afforded *tert*-butyl-*N*-phenylcarbamate (white solid, 0.485 g, 100%, entry 1; Table 1), identical (IR, ^{1}H and ^{13}C NMR and EIMS) to an authentic sample [6d]. The catalyst was recovered and used after being reactivated as described in Method A providing 90% yields during the reaction of aniline (2.5 mmol) with di-*tert*-butyl dicarbonate.

The remaining reactions were carried out following these general procedures (Method A or Method B). The physical data (mp, IR, NMR and MS) of known compounds were found to be identical with those of authentic samples. Unknown compounds were characterised by spectral (IR, NMR and MS) and elemental analyses.

4.1.1. Spectral data of unknown compounds

4.1.1.1. Furan-2-ylmethylcarbamic acid tert-butyl ester (entry 17; Table 1). Yellow oil—IR (neat): 3346, 2979, 2932, 1702, 1508, 1367, 1250, 1171, 1006 and 753 cm^{-1}; ^{1}H NMR (300 MHz; $CDCl_3$) $\delta = 7.34$ (s, 1H), 6.30 (s, 1H), 6.20 (s, 1H), 4.86 (brs, 1H), 4.29 (d, 2H, $J = 5.04$ Hz), 1.45 (s, 9H). MS (EI) *m/z*: 197 (M^+). Anal. calcd. for $C_{10}H_{15}NO_3$: C, 60.90; H, 7.67; N, 7.10. Found: C, 60.73; H, 7.69; N, 7.12.

4.1.1.2. (2-Hydroxy-2-methyl-propyl)-carbamic acid tert-butyl ester (entry 20; Table 1). Colorless oil—IR (neat): 3390, 3370, 2970, 2925, 1698, 1509, 1355, 1250, 1164 and 1060 cm^{-1}; ^{1}H NMR (300 MHz; $CDCl_3$) $\delta = 3.48$ (s, 2H), 1.42 (s, 9H), 1.22 (s, 6H). MS (EI) *m/z*: 189 (M^+). Anal. calcd. for $C_9H_{19}NO_3$: C, 57.12; H, 10.12; N, 7.40. Found: C, 57.08; H, 10.14; N, 7.42.

4.2. Spectral data of respresentative compound N-tert-butyl-phenylcarbamate (entry 1; Table 1)

N-tert-Butyl-phenylcarbamate (white solid, 0.485 g, 100%) mp 132 °C. IR (KBr): 3314, 2985, 1689, 1598, 1531, 1245 and 1150 cm^{-1}. ^{1}H NMR ($CDCl_3$, 300 MHz) $\delta = 7.24$–7.36 (m, 4H), 6.99–7.04 (m, 1H), 6.55 (bs, 1H), 1.51 (s, 9H). ^{13}C NMR ($CDCl_3$, 75 MHz) $\delta = 152.7$, 138.32, 128.93, 122.98, 118.52, 80.45 and 28.32. MS (EI): *m/z* 193 (M^+) identical with those of an authentic sample [6d].

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