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A protic ionic liquid catalyzed strategy for selective hydrolytic cleavage of *tert*-butyloxycarbonyl amine (*N*-Boc)†

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A simple, mild and efficient strategy for selective hydrolytic cleavage of the *N*-*tert*-butyloxycarbonyl (Boc) group is devised using a protic ionic liquid as an efficient catalyst. The deprotection reaction proceeded well for *N*-Boc protected aromatic, heteroaromatic, aliphatic compounds, and chiral amino acid esters and peptides. A wide range of labile protecting groups such as *tert*-butyl ester, *tert*-butyl ether, benzyloxycarbonyl (Cbz), TBDMS, *O*-Boc and *S*-Boc remained unaffected under the reaction conditions.

Due to environmental and economic issues as well as legislation, chemistry is driven to reduce waste, and reuse and recycle materials in order to meet the principles of green chemistry.[1] Thus the development of an environmentally benign, efficient and simple methodology for a fundamental organic transformation is in great demand. The protection and deprotection of functional groups is a common feature to synthesize multifunctionalized molecules in target oriented syntheses.[2] The choice of a suitable protecting group is often crucial in the context of simplifying the procedure, achieving the highest yield of the desired product, easy workup and separation. The protection of amines plays pivotal role in the synthetic organic chemistry. For instance, the *N*-Boc group is extensively used as a protecting group of amines in organic synthesis and amino acids in peptide and nucleoside chemistry. Consequently, a number of methods were developed for cleavage of the *N*-Boc group using strong acids,[3] Lewis acids[4] and microwave assisted neutral conditions[5] to liberate the parent amine. In some cases, basic conditions[6] such as aq. Na_2CO_3, Cs_2CO_3–imidazole and NaO^{t}Bu have been employed. The heterogeneous catalysis promoted *N*-Boc deprotection using sulfonic acid resin,[7a] montmorillonite K10,[7b] silica,[7c] heteropolyacids,[7d] HY-zeolite[7e] are also reported. However, most of the reported strategies suffer from serious drawbacks such as (i) longer reaction time, (ii) high temperature, (iii) low yield of products and (iv) exploiting expensive catalysts. Moreover, preparations of some of the catalysts are very tedious. Thus, organic synthesis professionals of industries and academia seek simple, efficient and milder methods for deprotection of this most frequently used protecting group, which should be selective enough for preserving the other functionalities in the molecule. G. Wang *et al.* (2009) reported[8] a special and efficient "green", catalyst-free, *N*-Boc deprotection in supercritical water under pressure. In their methodology, both aromatic and aliphatic *N*-Boc amines can be converted into the corresponding amines in high yields within 2–16 h, using distilled and deionized water (20 mL mmol^{-1}) at 150 °C. J. Wang and his colleagues (2009) described[9] a selective *N*-Boc deprotection method using boiling water as a reaction medium. In spite of the potentiality of these green methods, their major limitation is the use of sophisticated and costly technology, incompatibility of the deprotection reaction with ester functionality and longer reaction time. As an inexpensive and readily available reagent, imidazolium based protic ionic liquid has attracted considerable interest due to its less hazardous nature and efficiency in various organic transformations.[10] We have reported earlier an efficient method[11a] for the *tert*-butyloxycarbonylation of amines, amino acids/esters, alcohols and a green strategy for the selective hydrolytic cleavage of acetals and ketals[11b] using protic ionic liquid as an effective catalyst. In this communication, we disclose the efficacy of a protic ionic liquid as a catalyst for the selective deprotection of *N*-Boc group of a wide range of achiral and chiral compounds.

In our initial experiments we choose readily available *N*-Boc aniline as a model substrate and, the results for development and optimization of the deprotection studies is displayed in Table 1. On treatment of the *N*-Boc aniline (1 mmol) with 1 mmol of Bronsted acid ionic liquid (**I** or **II**) in water–dioxane (1 : 1) at 30 °C, the deprotection did not take place (Table 1, entry 1). However on rising the temperature to 70–72 °C

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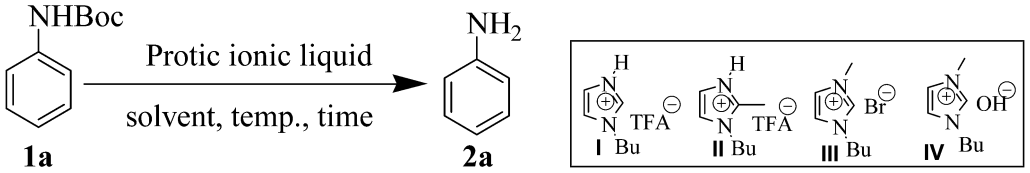
Table 1 Standardization of PIL catalyzed *N*-Boc cleavage of *N*-Boc aniline



Entry	Solvent	Temp. (°C)	Ionic liquid	Time (h)	Yield (%)
1	Dioxane–H_2O (1 : 1)	30	**I** or **II** (100 mol%)	12	0
2	Dioxane–H_2O (1 : 1)	70–72	**II** (100 mol%)	1	92
3	Dioxane–H_2O (1 : 1)	70–72	**II** (10 mol%)	6	85
4	Water	70–72	**II** (10 mol%)	6	Mixture
5	Dioxane–H_2O (1 : 1)	70–72	**III** (100 mol%)	12	0
6	Dioxane–H_2O (1 : 1)	70–72	**IV** (100 mol%)	12	0
7	Dioxane	80	**II** (100 mol%)	12	0
8	Water	80	None	4	0
9	Dioxane–H_2O (1 : 1)	80	None	4	0

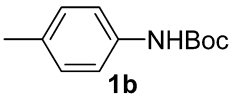
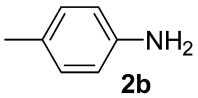
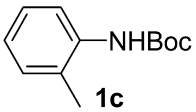
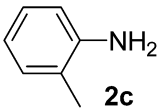
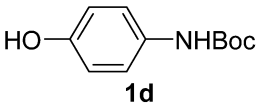
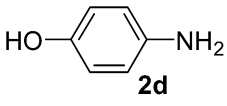
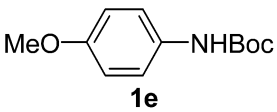
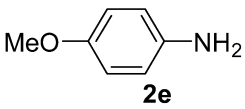
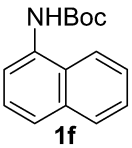
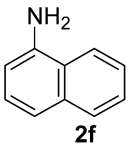
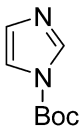
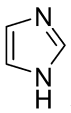
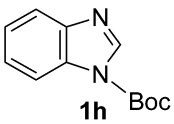
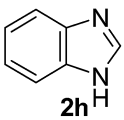
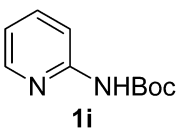
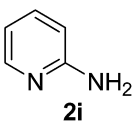
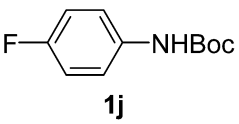
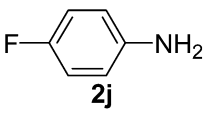
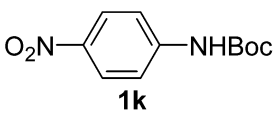
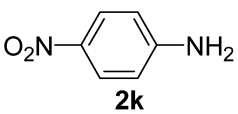
deprotection was smoothly accomplished after 1 h in 92% yield (Table 1, entry 2). Disappearance of the characteristic infrared absorption peak at 1690 cm^{-1} and ^1H NMR signal at δ 1.35 of **1a** confirmed the cleavage of *N*-Boc group. Usage of catalytic amount of **II** (10 mol%) was slowed down the reaction rate (6 h) and yield was also marginally dropped to 85% (Table 1, entry 3). Changing the solvent system dioxane–water (1 : 1) to water results incomplete conversion (Table 1, entry 4), probably due to the poor solubility of the precursor. It indicates that homogeneous solution of substrate in water–dioxane is essential for executing the catalytic cycle of the PIL. Since it was known that imidazolium cation has the capability to activate carbonyl group thus we turned our attention whether this cleavage reaction is compatible to other imidazolium based ionic liquid irrespective of their anionic counterparts. To investigate such role of imidazolium cation in this cleavage reaction we have employed 1-methyl-3-butyl imidazolium bromide (**III**) and 1-methyl-3-butyl imidazolium hydroxide (**IV**)[12] under the same reaction conditions. Unfortunately hydrolytic cleavage was unsuccessful (Table 1, entries 5, 6). These results revealed the proton present in ionic liquid **II** (PIL) is indispensable. Water has also important role with the protic ionic liquid as the deprotection reaction did not undergo in anhydrous condition even at 80 °C (Table 1, entry 7). The conversion of *N*-Boc aniline to aniline was also unsuccessful without protic ionic liquid either in water alone at 80 °C, (Table 1, entry 8, which was consistent with the results reported by G. Wang *et al.*[8]) or water–dioxane (Table 1, entry 9).

After the standard condition of the deprotection reaction is set, we have chosen several *N*-Boc protected aromatic and heteroaromatic compounds, which were subjected to deprotection under the optimized reaction conditions. We were pleased to observe that *N*-Boc protected *o*- and *p*-toluidine, 4-aminophenol, 4-methoxyaniline, 1-naphthylamine, imidazole, benzimidazole, 2-amino pyridine, 4-fluoroaniline and 4-nitroaniline undergo smoothly for deprotection under the catalysis of protic ionic liquid (**II**) to afford the desired amines in good to excellent yields (Table 2). It can be seen from the results summarized in Table 2 that the electron donating substituents such as methyl (Table 2, entries 1 and 2), hydroxyl (entry 3), methoxy (entry 4) accelerate the reaction rate whereas the electron withdrawing substituents fluoro (entry 9) and nitro (entry 10) decrease the rate of the reaction. *N*-Boc imidazole/benzimidazole (entries 6, 7) or *N*-Boc-2-amino pyridine (entry 8) also took longer reaction time. The slow reactivity of hetero-aromatic could be explained by the fact that the heterocyclic ring nitrogen got protonated by protic ionic liquid, which reduced the catalyst concentration. In the case of *N*-Boc protected 4-fluoroaniline and 4-nitroaniline the deprotection reactions were rather slow, but complete deprotection could be achieved at 80–82 °C in 72% and 75% yields respectively (Table 2, entries 9, 10). This is probably due to the solubility problem of the substrates in dioxane–water (1 : 1) medium.

The previous method is also applicable for deprotection of *N*-Boc protected aliphatic amines, which was working at a little bit higher temperature (80–82 °C). The *N*-Boc protected hexadecyl and octadecyl amines (Table 3, entries 1, 2) proceeded efficiently at 80 °C, whereas it was sluggish on use of *N*-Boc benzylamine and *N*-Boc dibenzylamines (Table 3, entries 3, 4). The *N*,*N*-di-Boc protected benzylamine was also cleaved to benzylamine by protic ionic liquid in water–dioxane (1 : 1) rather slowly (entry 5). The equimolar mixture of protected aromatic amine (*N*-Boc aniline) and aliphatic amine (*N*-Boc benzylamine) were treated together with equivalent amount of protic ionic liquid **II** in water–dioxane (1 : 1) at 70–72 °C, and complete conversion of *N*-Boc aniline to aniline was observed after 1 h with 90% yield (Table 3, entry 6). This experiment indicates that the deprotection strategy is highly selective for aromatic Boc-protected amino compounds over aliphatic *N*-Boc amines.

In contrary to cleavage of both the *N*-Boc and ester functional groups on use of supercritical water[8] or boiling water[9] herein *N*-

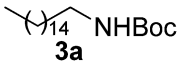
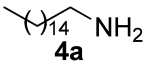
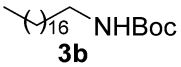
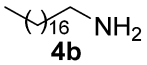
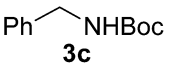
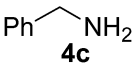
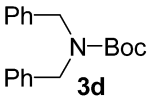
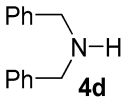
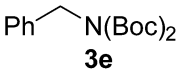
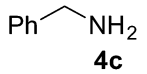
Table 2 Protic ionic liquid catalyzed deprotection of *N*-Boc aromatic amines[a]

Entry	Substrate	Product	Time (h)	Yield[b] (%)
1	 **1b**	 **2b**	1	98
2	 **1c**	 **2c**	1	92
3	 **1d**	 **2d**	2	82
4	 **1e**	 **2e**	1	85
5	 **1f**	 **2f**	2	89
6	 **1g**	 **2g**	3	77
7	 **1h**	 **2h**	4	65
8	 **1i**	 **2i**	5	55
9	 **1j**	 **2j**	5	72[c]
10	 **1k**	 **2k**	5	75[c]

[a] Reaction temperature 70–72 °C. [b] Yield reported on the basis of isolated product after column chromatography. [c] Reaction was carried out at 80–82 °C.

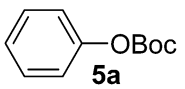
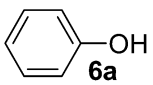
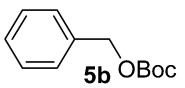
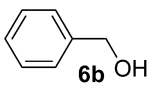
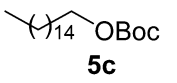
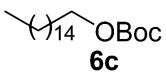
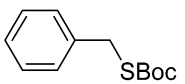
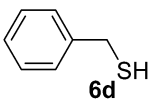
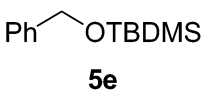
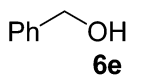
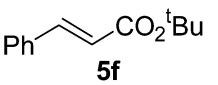
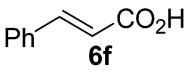
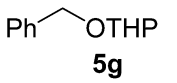
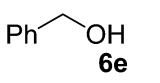
Boc deprotection were proceeds without cleavage of the ester functionality, *tert*-butyl ether group and *N*-benzyloxycarbonyl (Cbz) group present in amino acid derivatives or other molecules. To broaden the scope of the deprotection strategy, several other acid labile protecting groups such as tetrahydropyranyl, $-CO_2{}^tBu$ and –TBDMS were also addressed in our study. Since both phenolic –OH and alcoholic –OH get protected by Boc group at 70 °C under solvent free conditions[11a] we were interested to examine the behavior of these *O*-Boc protected compounds under the *N*-Boc deprotection reaction conditions. We noticed that only *O*-Boc protected phenol underwent deprotection to afford the desired product in 90% yield after 1.5 h (Table 4, entry 1), whereas alcoholic *O*-Boc compounds were completely inert under the same reaction conditions, even after 6 h (Table 4, entries 2, 3). The *S*-Boc protected benzyl thiol, *O*-TBDMS and *tert*-butyl ester were also inert against protic ionic

Table 3 Deprotection of *N*-Boc group of aliphatic amines[a]

Entry	Substrate	Product	Time (h)	Yield[b] (%)
1	 **3a**	 **4a**	1.5	96
2	 **3b**	 **4b**	1.5	92
3	 **3c**	 **4c**	4.0	98
4	 **3d**	 **4d**	5.5	80
5	 **3e**	 **4c**	7.0	74
6	PhNHBoc (**1a**) + $PhCH_2NHBoc$ (**3c**)	$PhNH_2$ (**2a**) + $PhCH_2NHBoc$ (**3c**)	1.0	90[c]

[a] Reaction temperature 80–82 °C. [b] Isolated yield. [c] Reaction temperature 70–72 °C.

Table 4 Deprotection of *O*-Boc and other protecting groups

Entry	Substrate	Product	Time (h)	Yield (%)
1	 **5a**	 **6a**	1.5	90
2	 **5b**	 **6b**	6.0	NR
3	 **5c**	 **6c**	6.0	NR
4	 **5d**	 **6d**	6.0	NR
5	 **5e**	 **6e**	6.0	NR
6	 **5f**	 **6f**	6.0	NR
7	 **5g**	 **6e**	4.0	93

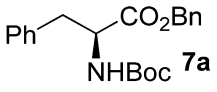
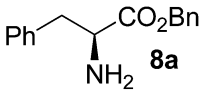
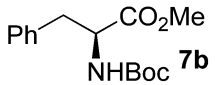
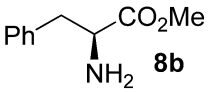
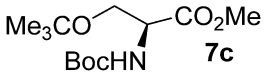
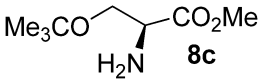
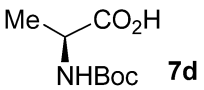
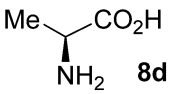
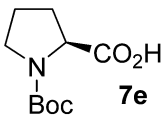
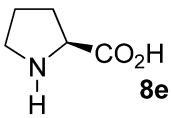
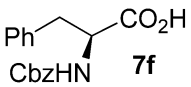
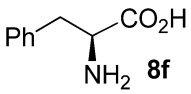
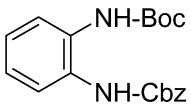
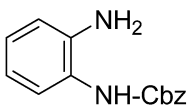
liquid under the similar reaction conditions (Table 4, entries 4–6) to afford the desired thiol, alcohol and acids. Interestingly the PIL catalyst was efficiently deprotected *O*-THP protecting group under the reaction conditions to afford corresponding aliphatic alcohol in 93% yield (Table 4, entry 7).

Having in mind the importance of *N*-Boc protection strategy in peptide synthesis, feasibility of *N*-Boc deprotected of different amino acids or their esters were examined keeping the reaction temperature at 80–82 °C. The *N*-Boc protected L-Phe-OBn, L-Phe-OMe and L-Ser(^{t}Bu)-OMe (Table 5, entries 1–3) all undergo smooth deprotection in water–dioxane to the corresponding amines. Racemization at the α-carbon of the amino acid derivatives were not observed in any cases. *N*-Boc protected amino acids such as Boc-Ala-OH or Boc-Pro-OH both underwent deprotection to free amino acids in 65% and 92% isolated yield respectively. In both the cases deprotection proceeds well in water (no need of dioxane) in shorter reaction time with respect to corresponding esters. It is noteworthy to mention that *N*-Cbz protected amino acid did not respond because no change was observed on treatment of Cbz-Phe-OH with 100 mol% of PIL (**II**) in water–dioxane (1 : 1) for 3 h at 80–82 °C. Thus, it is possible to deprotect *N*-Boc group in presence of an orthogonal *N*-Cbz group present in the same molecule (Table 5, entry 7).

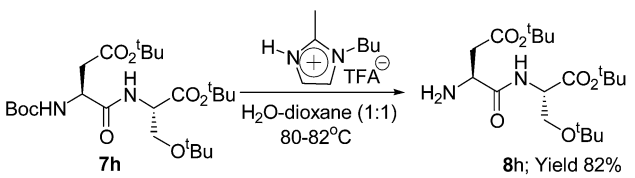
We are also delighted to observed that Boc protected N-terminal amino group was selectively deprotected when fully protected dipeptide BocAsp(O^{t}Bu)Ser(^{t}Bu)-O^{t}Bu (**7h**)[13] was treated with protic ionic liquid in water–dioxane (1 : 1) for 4 h at 80–82 °C to produced *N*-deprotected product **8h** in 82% yield (Scheme 1). In this reaction, peptide bond, $CO_2{}^t$Bu group of side chain as well as terminal position in aspartic acid residue and *tert*-butyl ether in serine residue were retained. This observation indicated that our method is highly selective for *N*-Boc group in a peptide in the presence of other common protecting groups.

The generally accepted mechanism for the deprotection of the Boc group under anhydrous acidic condition[14] involves the formation of carbon dioxide and isobutylene. In this case, the first step is the activation of Boc group by protonation and subsequent elimination of isobutylene produced unstable

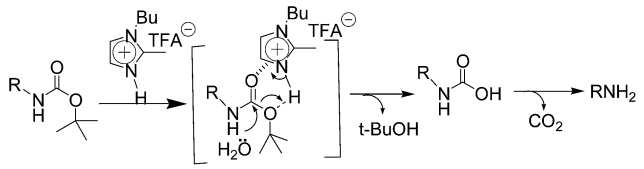
Table 5 Deprotection of *N*-Boc group present in amino acids and other derivatives[a]

Entry	Substrate	Product	Time (h)	Yield[b] (%)
1	 **7a**	 **8a**	3.5	72
2	 **7b**	 **8b**	3.0	70
3	 **7c**	 **8c**	4.5	75
4	 **7d**	 **8d**	1.0	65[c]
5	 **7e**	 **8e**	1.0	92[c]
6	 **7f**	 **8f**	3.0	NR
7	 **7g**	 **8g**	1.0	76

[a] Isolated yield. [b] Reaction temperature 80–82 °C. [c] Reaction proceed in water.



Scheme 1 Chemoselective deprotection of *N*-Boc group in a peptide.



Scheme 2 Probable mechanism of *N*-Boc deprotection reaction.

carbamic acid which releases carbon dioxide to afford parent amine. On the other hand, activation of carbonyls by imidazolium cation is well established. In our study we observed that the *N*-Boc deprotection was smoothly proceed when protic ionic liquid was employed and the cleavage reaction was not successful when imidazolium bromide or hydroxide was employed. To see whether proton alone could catalyzed the *N*-Boc deprotection we have carried out one control experiment with triethylammoniun trifluoroacetate by keeping the other conditions same (not shown in the tables) but this does not produce any deprotected product. Thus it is likely to involve dual activation of carbamate by imidazolium ion and proton attached in ionic liquid and then nucleophilic attack by water providing a tetra-coordinated species that can expels *tert*-BuOH and carbamic acid. The elimination of carbon dioxide from carbamic acid provides the parent amine (Scheme 2).

In conclusion we have demonstrated a simple, mild and chemoselective Boc-deprotection strategy for *N*-Boc amines using imidazolium based protic ionic liquid in water–dioxane mixture. The present approach proceeded well for aromatic, heteroaromatic, aliphatic *N*-Boc substrates as well as *N*-Boc amino acid/esters and *N*-Boc peptides. The acid sensitive and labile protecting groups such as methyl or *tert*-butyl ester, *tert*-butyl ether, TBDMS survived under the reaction conditions. Chemoselectivity of the reaction and many sensitive functional groups tolerance will make the protocol attractive and useful to the synthetic and medicinal chemists in academia and industry.

General procedure

To a stirred solution of *N*-Boc amine (1 mmol) in water–dioxane (1 : 1, 2 mL), protic ionic liquid **II** (1 equiv. or 10 mol%) was

added and then the mixture was heated at 70–72 °C or 80–82 °C depending upon the nature of substrate as indicated in the Tables 2–5 until the complete consumption of starting material (monitored by TLC). After completion of the reaction, dioxane was removed under reduced pressure and the mixture was diluted with water (5 mL). The mixture was extracted with diethyl ether or ethyl acetate (3 × 5 mL), washed with water, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was then passed through a short pad silica-gel to afforded parent amines. The aqueous part containing ionic liquid was recycled. The product was characterized by IR, ^1H and ^{13}C NMR spectral data and compare with authentic samples (ESI).

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