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ORIGINAL PAPER

A simple, rapid, and efficient *N*-Boc protection of amines under ultrasound irradiation and catalyst-free conditions

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Abstract A green and simple approach for the *N*-Boc protection on structurally diverse amines under ultrasound irradiation is described. Selective *N*-Boc protection was achieved in excellent isolated yield in a short reaction time at room temperature. Mild conditions, inexpensive and an easily available reagent, and absence of any auxiliary substances are the main advantages of this procedure.

Keywords Boc · Protection · Ultrasound · Green chemistry · Amines

Introduction

In modern synthetic chemistry, finding a suitable strategy for protection and deprotection of functional groups constitutes one of the most challenging tasks. Protection of the amine group is very important due to their presence in various biologically active compounds [1–3]. The *tert*-butoxycarbonyl (Boc) group has a widely useful functionality for the protection of amine among various protecting groups. The greatest attention is due to the extreme stability of *the N*-Boc group toward catalytic hydrogenolysis and the extreme resistance to basic and nucleophilic conditions [4, 5], and its labile nature under several chemical transformations [1].

Several methods have been described to introduce the Boc protecting group, using di-*tert*-butyl dicarbonate, $(Boc)_2O$, under a variety of conditions. *N*-Boc protection is frequently reported by base-catalysed reactions using DMAP [6], aq. NaOH [5], NaHMDS [7], or Lewis acids-catalysed reactions, such as $ZrCl_4$ [8], $LiClO_4$ [9], $HClO_4/SiO_2$ [10], $Cu(BF_4)_2{\cdot}\times H_2O$ [11], $Zn(ClO_4)_2{\cdot}6H_2O$ [12], yttria-zirconia [13], $La(NO_3)_3{\cdot}6H_2O$ [14], montmorillonite K-10 [15], amberlyst-15 [16], $H_3PW_{12}O_{40}$ [17], and sulfamic acid [18]. Many of these methods suffer from disadvantages such as acidity, high cost, toxicity, corrosiveness, and requirement of auxiliary substances in the isolation of the product [19].

In recent years, the emergence of the sustainable development of "green chemistry" has led to new solutions to existing problems in protecting group chemistry. Ionic liquids, as eco-friendly solvents, catalysts and reagents in green synthesis [20], such as 1-methylimidazolium tetrafluoroborate [(HMIm)BF_4] [21], 1,1,3,3-tetramethylguanidine acetate [TMG][Ac] [22], 1,3-disulfoimidazolium hydrogen sulfate [Dsim]HSO_4 [23], and 1-alkyl-3-methylimidazolium cation are employed for *N*-Boc protection [24]. Furthermore, water has attracted much attention as a green solvent. An environmentally benign approach is described where the *N*-Boc derivatives were prepared chemoselectively in water [25].

In our previous work, eco-sustainable methods for protection/deprotection of amines and alcohols with *t*-Boc group were carried by fusion [26], using heteropolyanion [27], and in water [28–30]. More recently, Dighe et al. [31] have reported a green approach for the synthesis of Boc-protected amines under microwaves without using of solvents and catalysts.

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In the last decade, the sonochemical approach has been widely used in various types of organic transformations [32]. This method is not only simple and efficient but also can assist in preserving green chemistry concepts [33]. Sonochemistry offers a more efficient and facile method for a large variety of syntheses in comparison to classical protocols, e.g., an *N*-sulfonylation reaction using Zn-Al-hydrotalcites solid base catalyst under ultrasound irradiation in ethanol has been developed [34].

In continuation of our interest to improve a facile process under green conditions not including catalysts and solvents, we report here the use of ultrasonic irradiation for selective *tert*-butoxycarbonylation of various amine derivatives.

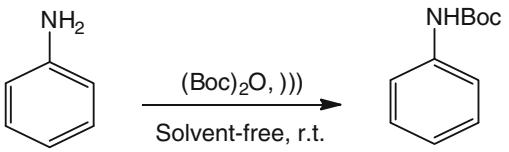
Results and discussion

Herein we studied the *N-tert*-butoxycarbonylation of structurally diverse amines using ultrasound irradiation. In an initial attempt, we reacted 1 mmol of aniline with 1.1 mmol of di-*tert*-butyl dicarbonate in the absence of any solvent or catalyst; after 5 min the reaction was completed with an excellent yield (Scheme 1).

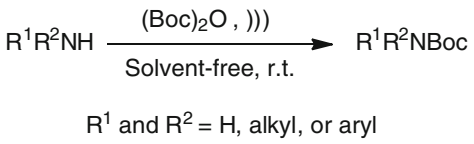
To find the effect of ultrasound, the same reaction was carried out under the same conditions in the absence of ultrasound irradiation. No reaction occurs after 5 h working time, this shows the essential role of ultrasound irradiation. This excellent result encourages us to extend this study to various structurally amines.

To optimize our protocol, we also applied our reaction conditions to a number of primary and secondary aromatic and aliphatic (cyclic and acyclic) amines (Scheme 2). In all cases we obtained the *N*-Boc products in short reaction times with quantitative yields (Table 1, entries 1–11). No competitive side reactions lending formation of isocyanate [35], urea [6], and *N*,*N*-di-Boc derivatives [36] were detected by TLC of the crude products (entries 1–8).

Scheme 1



Scheme 2



In order to explore the generality of this method, we also attempted the protection of *β*-aminoalcohol under the same reaction conditions (Table 1, entries 12–15). A notable chemoselectivity of the protection has been established where the amine functional was only protected without competitive formation of *O*-Boc or oxazolidinone derivatives [6].

The mildness of this procedure was next illustrated by a range of α-aminoesters (entries 16–20), the reactions worked very well, a methyl ester group was resisted and the optically pure *NH*-Boc derivative was confirmed by optical rotation and comparison with the literature [37, 38], where the configuration of the chiral center is not affected under reaction conditions.

To increase the scope of this reaction, we attempted the *N-tert*-butoxycarbonylation to sulfamides synthesized from α-aminoesters [39–41]. In view of their importance, they were used as substrates (Table 1, entries 21–25) and were tested in this method to verify electron-withdrawing effect of substituents on the formation of pure *NH*-Boc products. All *N*-protected sulfamides were obtained in excellent yields and the reaction preserves stereochemical integrity of *N*-Boc amino acids [42].

The presented results demonstrate the specific ultrasonic effect on *N-tert*-butoxycarbonylation giving pure product with quantitative yields in a few minutes.

The ultrasonic energy applying without any base or acid catalyst to the reaction generates the acoustic cavitation mechanical effect when sonic waves propagate through the medium. In solids, both longitudinal and transverse waves can be transmitted whereas in liquids only longitudinal waves can be transmitted [43–45]. Vibrations of molecules generate compressions and rarefactions which give rise to the phenomenon of bubble formation and collapse in the reaction mixture [amine and reactant $(Boc)_2O$] and facilitate the nucleophilic attack of the amino functional on the carbonyl group. During cavitation, the chemical bonds break, and carbon dioxide and the *tert*-butanol were eliminated to afford the *N*-Boc amine (Scheme 3).

In conclusion, the ultrasound irradiation allowed for the highly chemoselective, simple, efficient, environmentally benign *N*-Boc protection of various aliphatic and aromatic amine derivatives under solvent free conditions, in short reaction times and excellent isolated yields without formation of isocyanate, urea, *N*,*N*-di-*t*-Boc, or *O*-*t*-Boc as side products. In contrast to conventional energy sources of traditional methods suffering from harsh conditions, this easier manipulation delineates the scope of this technique and offers potential in different applications in organic transformations.

Table 1 *N-tert*-Butoxycarbonylation of amines under ultrasound irradiation

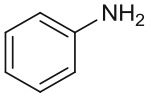
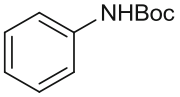
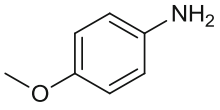
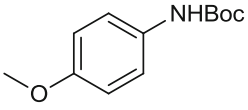
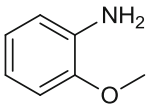
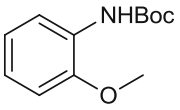
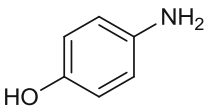
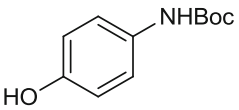
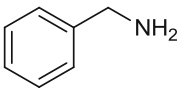
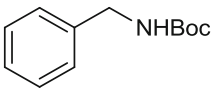
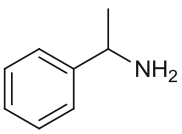
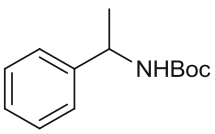
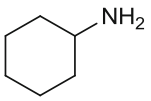
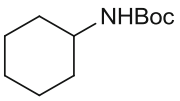
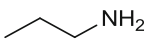
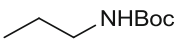
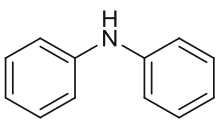
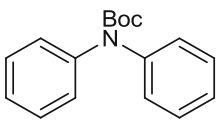
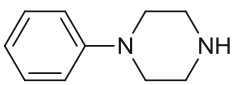
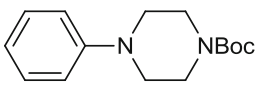
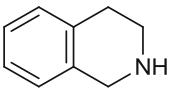
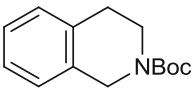
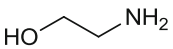
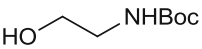
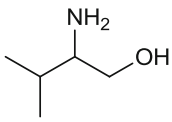
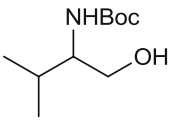
Entry	Substrate	Product	Time /min	M.p. /°C
1			5	132 (132 [14])
2			3	92-94 (94-96 [14])
3			3	34-36
4			3	146 (146 [14])
5			5	56-58 (52-54 [14])
6			4	80-82
7			2	64-66 (65-67 [42])
8			2	30-32
9			6	29-30
10			4	71-73 (70-71 [42])
11			4	29-30
12			2	32-33
13			2	Oil

Table 1 continued

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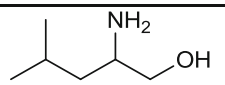
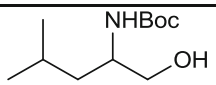
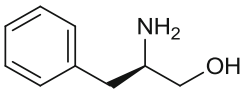
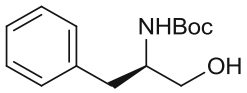
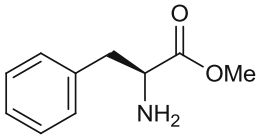
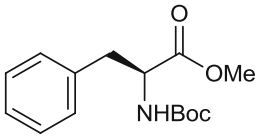
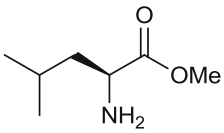
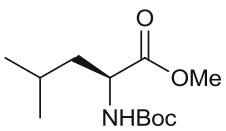
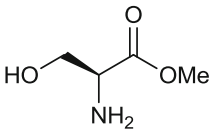
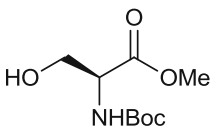
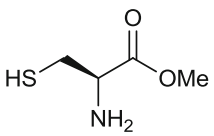
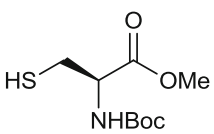
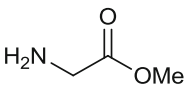
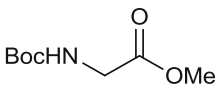
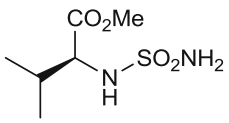
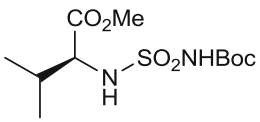
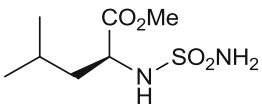
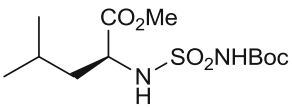
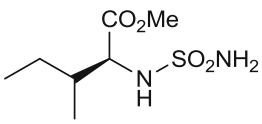
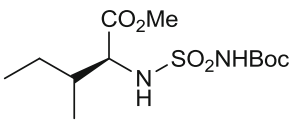
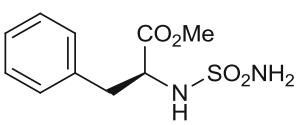
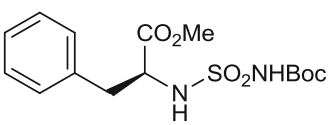
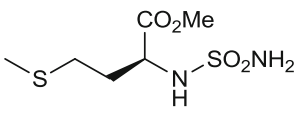
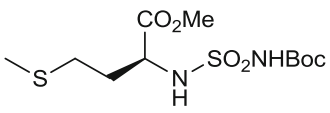
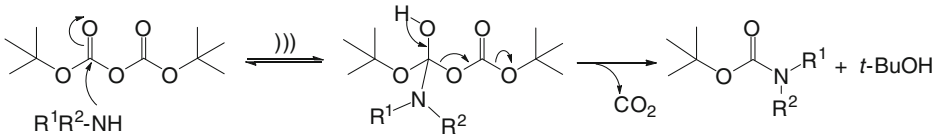
14			2	104-106
15			2	96-98 (96-97 [42])
16			2	40-41 (39-41 [43])
17			2	Oil [43]
18			2	33-35
19			2	29-30
20			2	109
21			6	89-90 [27]
22			6	67-68 [27]
23			6	75-77
24			7	131-132 [27]
25			7	78-80

Scheme 3



Experimental

All commercial chemicals were used without further purification. Sonication was performed in a FUNGILAB ultrasonic bath with a frequency of 40 kHz and a power of 250 W. All reactions were monitored by thin layer chromatography (TLC) on silica Merck 60 F254 percolated aluminum plates. ^{1}H and ^{13}C NMR spectra were recorded in a 250 or 300 MHz Bruker spectrometer. Chemical shifts are reported in δ units (ppm) with tetramethylsilane (TMS) as a reference. All coupling constants (J) are reported in Hertz. Multiplicity is indicated as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Optical rotations were measured on a JUSCO DIP-370 digital polarimeter.

General procedure for tert-butoxycarbonylation of amines

Amine (1 mmol) and di-*tert*-butyl dicarbonate [$(Boc)_2O$, 1.1 mmol] were placed in a glass tube under neat conditions and were sonicated for a suitable time (as indicated in Table 1). All reactions were performed in a water bath at room temperature. After completion of the reaction (as indicated by TLC), 5 cm^3 of diethyl ether was added to the mixture, the resulting *tert*-butanol was freely soluble in diethyl ether and the *N*-Boc product was crystallized. Purification of the product was accomplished by recrystallization from diethyl ether.

N-(tert-Butoxycarbonyl)-2-methoxyaniline
(**3**, $C_{12}H_{17}NO_3$)
White solid, m.p.: 34–36 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.50$ (s, 9H), 3.75 (s, 3H), 6.56 (s, 1H, NH), 6.81–7.26 (m, 4H, Ar) ppm; ^{13}C NMR (74 MHz, $CDCl_3$): $\delta = 28.4$ (3 CH_3), 55.4 (CH_3), 80.1 (C), 114.1 (CH), 116.1 (CH), 120.6 (CH), 131.5 (CH), 139.8 (C), 153.4 (C), 155.6 (C = O).

N-(tert-Butoxycarbonyl)-2-phenylethylamine
(**6**, $C_{13}H_{18}NO_2$)
White solid, m.p.: 80–82 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.44$ (d, $J = 6.1$ Hz, 3H), 1.53 (s, 9H), 2.18 (m, 1H), 4.79 (s, 1H, NH), 7.11–7.53 (m, 5H, Ar) ppm; ^{13}C NMR (74 MHz, $CDCl_3$): $\delta = 23.5$ (CH_3), 28.7 (3 CH_3), 47.5 (*CH), 80.1 (C), 126.5 (CH), 127.9 (2 CH), 128.7 (2 CH), 142.4 (C), 157.6 (C = O) ppm.

N-(tert-Butoxycarbonyl)propylamine (**8**, $C_8H_{17}NO_2$)
White solid, m.p.: 30–32 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.80$ (t, $J = 6.0$ Hz, 3H), 1.47 (s, 9H), 1.59 (m, 2H), 2.96 (t, $J = 5.8$ Hz, 2H, CH_2-NH), 4.55 (s, 1H, NH) ppm; ^{13}C NMR (74 MHz, $CDCl_3$): $\delta = 11.5$ (CH_3), 24.8 (CH_2), 27.7 (3 CH_3), 46.3 (CH_2), 80.1 (C), 156.9 (C = O) ppm.

N-(tert-Butoxycarbonyl)diphenylamine (**9**, $C_{17}H_{19}NO_2$)
White solid, m.p.: 29–30 °C; ^{1}H NMR (300 MHz, $CDCl_3$) $\delta = 1.49$ (s, 9H), 6.82–7.31 (m, 10H, 2 Ph) ppm; ^{13}C NMR (74 MHz, $CDCl_3$): $\delta = 27.2$ (3 CH_3), 83.0 (C), 117.5 (4 CH), 120.6 (2 CH), 129.1 (4 CH), 143.1 (2C), 146.7 (C = O) ppm.

N-(tert-Butoxycarbonyl)-1,2,3,4-tetrahydroisoquinoline
(**11**, $C_{14}H_{19}NO_2$)
White solid, m.p.: 29–30 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.54$ (s, 9H), 2.85 (t, $J = 7.8$ Hz, 2H), 3.66(t, $J = 7.8$ Hz, 2H), 4.59 (s, 2H), 7.12-7.28 (m, 4H, Ar) ppm; ^{13}C NMR (74 MHz, $CDCl_3$): $\delta = 28.4$ (3 CH_3), 31.7 (CH_2), 49.9 (CH_2), 53.9 (CH_2), 81.0 (C), 128.3 (CH), 129.1 (CH), 131.1 (CH), 134.7 (CH), 145.6 (C), 147.9 (C), 156.0 (C = O) ppm.

N-(tert-Butoxycarbonyl)-2-hydroxyethylamine
(**12**, $C_7H_{15}NO_3$)
White solid, m.p.: 32–33 °C; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 1.45$ (s, 9H, *N-t*-Bu), 3.25 (q, $J = 5.2$ Hz, 2H, CH_2-NH), 3.65 (t, $J = 5.1$ Hz, 2H, CH_2-OH), 5.25 (s, 1H, NH) ppm; ^{13}C NMR (62 MHz, $CDCl_3$): $\delta = 28.2$ (3 CH_3), 43.6 (CH_2-NH), 62.5 (CH_2-OH), 80.0 (C), 157.3 (C = O) ppm.

(S)(-)-N-(tert-Butoxycarbonyl)valinol (**13**, $C_{10}H_{21}NO_3$)
Oil; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.92$ (d, $J = 8.5$ Hz, 3H), 0.94 (d, $J = 8.5$ Hz, 3H), 1.44 (s, 9H), 1.86 (m, 1H, CH), 3.39 (m, 2H), 4.96 (m, 1H, *CH), 5.35 (s, 1H, NH) ppm; ^{13}C NMR ($CDCl_3$,74 MHz): $\delta = 18.3$ (CH_3), 19.36 (CH_3), 28.2 (3 CH_3), 57.8 (CH), 60.2 (CH_2), 63.4 (*CH), 79.1(C), 156.6 (C = O) ppm.

(S)(-)-N-(tert-Butoxycarbonyl)leucinol (**14**, $C_{11}H_{23}NO_3$)
White solid, m.p.: 104–106 °C; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 0.92$ (d, $J = 7.5$ Hz, 6H), 1.27 (m, 2H), 1.5 (s, 9H), 1.65 (dd, $J = 5.7$, 7.0 Hz, 2H, CH_2-OH), 1.90 (m, 1H), 4.25 (m, 1H, *CH), 4.80 (d, $J = 8.3$ Hz, 1H, NH) ppm; ^{13}C NMR (62 MHz, $CDCl_3$): $\delta = 21.4$ (2 CH_3), 23.7

(CH), 27.9 (3 CH_3), 42.0 (CH_2), 45.3 (*CH), 64.2 (CH_2), 83.7 (C), 151.8 (C = O) ppm.

(S)(-)-N-(tert-Butoxycarbonyl)serine methyl ester (**18**, $C_9H_{16}NO_5$)
White solid, m.p.: 33–35 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.47 (s, 9H), 3.68 (s, 3H, OCH_3), 4.43 (dd, J = 7.8, 6.0 Hz, 2H, CH_2), 4.61 (m, 1H, *CH), 5.10 (d, J = 8.0 Hz, 1H, NH) ppm; ^{13}C NMR (62 MHz, $CDCl_3$): δ = 28.8 (3 CH_3), 54.0 (CH_3), 61.0 (CH_2), 69.3 (*CH), 79.5 (C), 157.0 (C = O), 172.0 (C = O) ppm.

(S)(-)-N-(tert-Butoxycarbonyl)cysteine methyl ester (**19**, $C_9H_{16}NO_5S$)
White solid, m.p.: 29–30 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.47 (s, 9H), 3.23 (dd, J = 8.0, 6.8 Hz, 2H, CH_2), 3.71 (s, 3H, OCH_3), 4.81 (m, 1H, *CH), 5.50 (d, J = 8.0 Hz, 1H, NH) ppm; ^{13}C NMR (62 MHz, $CDCl_3$): δ = 28.8 (3 CH_3), 40.0 (CH_2), 54.0 (CH_3), 60.3 (*CH), 80.5 (C), 157.2 (C = O), 171.8 (C = O) ppm.

N-(tert-Butoxycarbonyl)glycine methyl ester (**20**, $C_8H_{15}NO_4$)
White solid, m.p.: 109 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.49 (s, 9H), 3.75 (d, J = 6.0 Hz, 2H, CH_2), 3.71 (s, 3H, OCH_3), 6.10 (t, J = 8.0 Hz, 1H, NH) ppm; ^{13}C NMR (62 MHz, $CDCl_3$): δ = 28.5 (3 CH_3), 49.0 (CH_2), 52.9 (CH_3), 80.5 (C), 157.2 (C = O), 171.8 (C = O) ppm.

(S)(-)-N-(tert-Butoxycarbonylaminosulfonyl)isoleucine methyl ester (**23**, $C_{12}H_{24}N_2O_6S$)
White solid, m.p.: 75–77 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 0.90 (t, J = 6.2 Hz, 3H, CH_3), 0.96 (d, J = 5.8 Hz, 3H, CH_3), 1.55 (s, 9H), 1.71 (m, 2H, CH_2), 1.87 (m, 1H, CH), 3.70 (s, 3H, OCH_3), 4,25 (m, 1H, *CH), 5.80 (d, J = 8.9 Hz, 1H, NH-*C), 7.60 (s,1H, NH-Boc) ppm; ^{13}C NMR (62 MHz, $CDCl_3$): δ = 20.4 (CH_3), 21.7 (CH_3), 24.6 (CH_2), 28.1 (3 CH_3), 42.8 (CH), 53.1 (CH_3), 55.0 (*CH), 80.7 (C), 148.8 (C = O), 171.7 (C = O) ppm.

(S)(-)-N-(tert-Butoxycarbonylaminosulfonyl)methionine methyl ester (**25**, $C_{11}H_{22}N_2O_6S_2$)
White solid, m.p.: 78–80 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.48 (s, 9H), 2.18 (s, 3H, CH_3), 2.27 (m, 2H, CH_2), 2.67 (t, J = 6.5 Hz, 2H, CH_2), 3.38 (m, 1H, *CH), 3.70 (s, 3H, OCH_3), 6.30 (d, J = 8.4 Hz, 1H, NH-*C), 7.80 (s, 1H, NH-Boc) ppm; ^{13}C NMR (62 MHz, $CDCl_3$): δ = 23.8 (CH_3), 28.7 (3 CH_3), 29.6 (CH_2), 30.1 (CH_2), 53.2 (CH_3), 51.8 (*CH), 80.1 (C), 158.2 (C = O), 171.5 (C = O) ppm.

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