Iron(III)triflate as a highly efficient, recyclable and green catalyst for the *N*-Boc protection of amines

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Iron (III) triflate was used as an efficient catalyst for *N-t*-butoxycarbonylation of amines with di-*t*-butyl dicarbonate under solvent-free conditions at room temperature. Various aliphatic, aromatic, heterocyclic amines and aminols were protected as their corresponding mono-carbamates in excellent yields and short reaction times. Only two of the 23 monocarbamates were new. No competitive side reactions such as isocyanate, urea, nor *N,N*-di-Boc formation were observed. The reported method is mild and has the advantages of low cost, chemoselectivity and, because no solvent is used and the catalyst can be recycled, it is classifiable as a green procedure.

Keywords: amines, Boc_2O, *N*-Boc protection, iron triflate

Protection and deprotection of any polyfunctional molecule often needs a selective and efficient protecting reagent together with mild reaction conditions.[1] Owing to the desire to develop a mild, selective, and efficient protecting group, especially for amines, Boc protection has become one of the most useful steps due to its stability towards basic and nucleophilic reactions.[2]

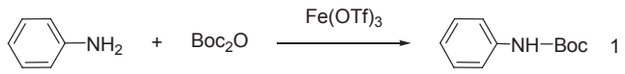
A variety of reagents and procedures have been applied to introduce this group using Boc_2O and either a nucleophilic basic catalyst such as DMAP,[3] NH_2OH,[4] or K_2CO_3,[5] or a Lewis acid catalyst such as I_2,[6] yttria-zirconia,[7] $ZrCl_4$,[8] $Zn(ClO_4)_2{\cdot}6H_2O$,[9] $LiClO_4$,[10] montmorillonite K10 or KSF,[11] $La(NO_3)_3$,[12] saccharin sulfonic acid (SaSA),[13] sulfonic acid-functionalised ordered nanoporous Na^+-montmorillonite (SANM),[14] succinimide sulfonic acid (SuSA),[15] [TMG][Ac],[16] sulfonic acid-functionalised silica,[17] $H_3PW_{12}O_{40}$,[18] thioglycoluril,[19] thiourea,[20] sulfamic acid,[21] Nano ZnO,[22] nanocrystalline TiO_2-$HClO_4$,[23] poly(4-vinylpyridine),[24] N-sulfonic acid poly(4-vinylpyridinium)chloride,[25] poly(N-vinylimidazole),[26] ionic liquid($[C_6(mpy)_2][C\,^oCl_4]^{2-}$),[27] and Amberlyst-15.[28] Some of these methodologies, although effective, have several drawbacks such as long reaction times, formation of side-products,[29–31] potential hazards (*e.g.* the high toxicity of DMAP and reagents), excess reagents in the case of Lewis acid-catalysed reactions,[32] and the problem of catalyst recovery, catalyst preparation and limited applicabilities (*e.g.* the use of H_2SO_4 at 500 °C to prepare yttria-zirconia; $ZrCl_4$ is highly moisture sensitive and liberates HCl fumes; perchlorate reagents are strong oxidants and explosive in nature).

Iron triflate is a better potential Lewis acid catalyst, since it is highly soluble in water and does not decompose under aqueous conditions. The nonhygroscopic nature, high catalytic activity, short reaction periods, facile separation and reusability of this catalyst are other advantages over other conventional Lewis acids. Recently, iron triflate has been used to catalyse organic reactions under solvent-free conditions, *e.g.* synthesis of quinolones,[33] and synthesis of indenoquinolinones,[34] and here, we report an efficient, mild and green protocol for the *N*-Boc protection of amines under similar conditions.

Results and discussion

In our initial experiments, aniline (1 mmol) was chosen as a model substrate for optimisation of the best reaction conditions under solvent-free conditions with Boc_2O (1 mmol). The mixture of aniline and Boc_2O was stirred in the presence of $Fe(OTf)_3$ (0.1–5 mol%) at room temperature for certain times. As shown in Table 1, the reaction was conducted in the presence of 0.1 mol%, 0.5 mol%, 1 mol%, 2 mol% and 5 mol% of $Fe(OTf)_3$ and the product *t*-butyl phenylcarbamate (1) was isolated in 88, 98, 97, 98 and 96% yield, respectively. Although employment of 0.5 mol% of the catalyst also proceeded well, long reaction times were typical to achieve comparable yields to those obtained with 1 mmol% of catalysts. Therefore, 1 mol% of catalyst was a sufficient amount to obtain the best yield without adversely affecting the product yields.

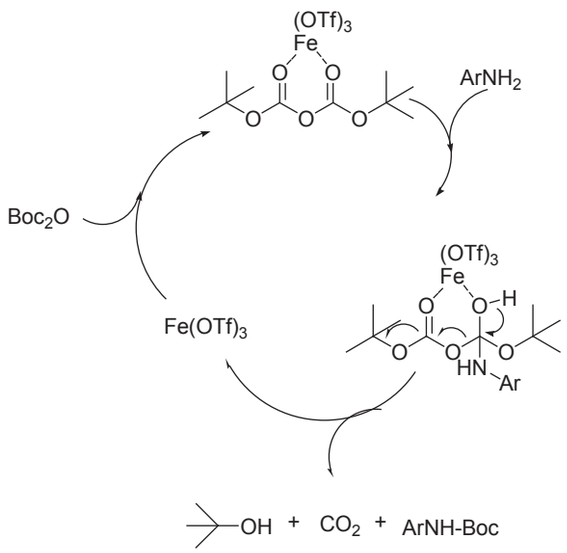
Table 1 Optimisation of the reaction conditions for the iron triflate-catalysed *N-t*-butoxycarbonylation of 1 equiv. aniline with 1 equiv. Boc_2O



Entry	Catalyst/mol%	Time/min	Yield/%[a]
1	none	24 h	21
2	0.1	60	88
3	0.5	30	98
4	1	8	97
5	2	8	98
6	5	8	96

[a]Isolated yields.

After optimisation of the reaction conditions, a variety of aliphatic, aromatic and heterocyclic amines and aminols were subjected to *N*-Boc protection with Boc_2O under the selected



Scheme 1

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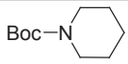
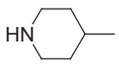
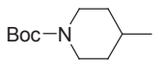
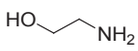
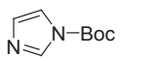
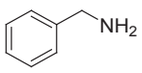
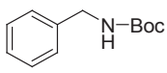
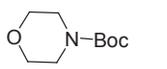
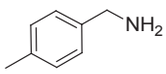
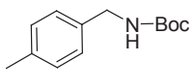
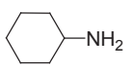
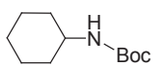
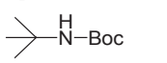
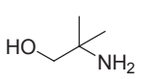
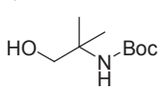
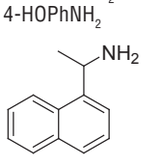
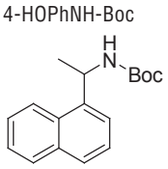
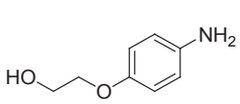
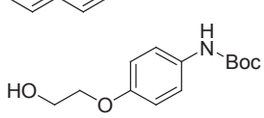
Table 2 *N*-Boc protection of amines by Boc_2O in the presence of 1 mol% $Fe(OTf)_3$ under solvent-free conditions

Entry	Amines	Product	Time/min	Yield/%[a]
1			5	96
2			5	97
3			2	99
4			7	96
5			2	99
6			5	94
7			2	98
8			1	97
9	Et_2NH	Et_2*N*-Boc	3	96
10			2	96
11			4	99
12	$PhNH_2$	PhNH-Boc	10	92
13	4-$MePhNH_2$	4-MePhNH-Boc	4	99
14	4-$MeOPhNH_2$	4-MeOPhNH-Boc	3	99
15	4-NO_2PhNH_2	4-NO_2PhNH-Boc	7	98
16	4-$ClPhNH_2$	4-ClPhNH-Boc	8	97
17	4-$BrPhNH_2$	4-BrPhNH-Boc	6	96
18	2-$HOPhNH_2$	2-HOPhNH-Boc	5	99
19	3-$ClPhNH_2$	3-ClPhNH-Boc	8	94
20	3-$MeOPhNH_2$	3-MeOPhNH-Boc	3	99
21	4-$HOPhNH_2$	4-HOPhNH-Boc	5	98
22			3	99
23			5	97

[a]Isolated yields.

conditions. The results are summarised in Table 2. The results showed that the reactions of amines including primary and secondary amines with Boc_2O proceeded well and gave high yields. Furthermore, no byproducts such as isocyanates, urea or *N,N*-di-Boc derivatives were observed. When present, alcohol and phenol groups remained intact under the reaction conditions. Therefore, this method can be useful for the chemoselective *N*-Boc protection of amines containing alcohol and phenol groups (Table 2, entries 3, 11, 18 and 21). A plausible mechanistic pathway is outlined in Scheme 1. The initial step is the formation of a complex between the iron atom and the two C=O groups of Boc_2O, which facilitates attack by the amine at one of the C=O groups. Collapse of the tetrahedral intermediate so formed yields the *N*-*t*-butoxycarbonylated product, *t*-butanol, CO_2 and regenerates the iron catalyst.

Table 3 Recyclability of the catalyst[a] for the synthesis of *t*-butyl phenylcarbamate (**1**)

Entry	Cycle	Recovery rate of catalyst/%	Time/min	Yield/%[b]
1	Cycle 1	98	10	98[b]
2	Cycle 2	96	10	95
3	Cycle 3	98	10	94
4	Cycle 4	97	10	94

[a]The catalyst was new
[b]Isolated yields.

From the green chemistry point of view, efficient recovery and reuse of the catalyst are highly desirable, thus the recovery and reusability of $Fe(OTf)_3$ were investigated. $Fe(OTf)_3$ has two important features, solubility and stability in water. After the reaction was completed, the reaction mixture was completely dissolved in ethyl acetate, and then washed with water. The catalyst was recovered from the aqueous layer *via* evaporation

of the water and dried at 150 °C. The recovered $Fe(OTf)_3$ was reused directly for at least three consecutive cycles and the results are listed in Table 3.

In summary, we have described a simple, efficient and environmentally benign method for chemoselective *N*-Boc protection for amines using a novel Lewis acid and an easily recoverable catalyst $Fe(OTf)_3$ under solvent-free conditions. Our protocol gave the products in high yields in a reduced reaction time and reduced amount of catalyst. Further investigations on the application of this catalyst are underway in our laboratory.

Experimental

All reagents were purchased from commercial sources and used without further purification. Melting points were determined on a RY-1 hot stage microscope and are uncorrected. IR spectra were determined as KBr pellets on a Thermo Nicolet 6700 spectrophotometer. NMR spectra were obtained on a Bruker Avance DPX-300 MHz spectrometer (^{1}H NMR at 300 MHz, ^{13}C NMR at 75 MHz) in $CDCl_3$, chemical shifts (δ) were given in part per million (ppm) relative to TMS as an internal standard. The HRMS spectra were obtained on a Thermo Finnigan spectrometer, model MAT 95XP. All reactions were monitored by TLC on silica gel GF-254 glass plates (E.Merck) and viewed under UV light at 254 nm.

Synthesis of $Fe(OTf)_3$

Triflic acid (4.52 g, 30 mmol) was gradually added to cool water, and then Fe_2O_3 powder (2.4 g, 15 mmol) was added. The mixture was heated at 95 °C until the pH of the water became neutral. After completion, the reaction mixture was filtered and the filtrate was evaporated and the pale white powder obtained was dried.

N-Boc protection of amines; typical procedure

$Fe(OTf)_3$ (1 mol%) was added to a magnetically stirred mixture of an amine (1 mmol) and Boc_2O (1 mmol) at room temperature. The mixture was stirred until completion of the reaction (TLC), then diluted with EtOAc and washed with water. The organic layer was dried over anhydrous $MgSO_4$, then the solvent was distillated off under vacuum to yield the highly pure *N*-Boc derivatives.

Most of the products are known, except for entries 22 and 23 in Table 2. All compounds were characterised by IR, ^{1}H NMR, ^{13}C NMR, the new ones also by HRMS.

t-Butyl piperidine-1-carboxylate (**1**): Colourless oil, (lit.[6] colourless oil); IR: 2990, 2840, 1678, 1520, 1450, 1360, 1272, 1238, 1170, 1050, 898 cm^{-1}; ^{1}H NMR δ 3.26–3.32 (m, 4H), 1.42 (s, 9H), 1.36–1.56 (m, 6H); ^{13}C NMR δ 24.3, 25.5, 28.2, 46.6, 76.7, 157.4.

t-Butyl 4-methylpiperidine-1-carboxylate (**2**): Colourless oil, (lit.[6] colourless oil); IR: 2990, 2984, 1680, 1451, 1407, 1358, 1266, 1245, 1164, 1119, 1079, 857 cm^{-1}; ^{1}H NMR δ 3.38 (m, 4H), 2.32 (m, 4H), 2.18 (s, 3H), 1.43 (s, 9H); ^{13}C NMR δ 28.3, 43.4, 45.9, 54.7, 79.5, 154.6.

t-Butyl 2-hydroxyethylcarbamate (**3**): Colourless oil, (lit.[6] colourless oil); IR: 3357, 2976, 2933, 1690, 1525, 1366, 1279, 1251, 1171, 1068, 864 cm^{-1}; ^{1}H NMR δ 5.16 (s, 1H), 3.64 (m, 2H), 3.25 (m, 2H), 1.44 (s, 9H); ^{13}C NMR δ 27.9, 42.8, 62.0, 79.5, 156.4.

t-Butyl 1H-imidazole-1-carboxylate (**4**): White solid, m.p. 45–46 °C (MeOH) (lit.[8] 42–45 °C); IR: 3125, 2975, 1747, 1464, 1379, 1315, 1291, 1241, 1150, 1089, 995, 833, 765, 644 cm^{-1}; ^{1}H NMR δ 8.02(s, 1H), 7.32 (s, 1H), 7.02 (s, 1H), 1.64 (s, 9H); ^{13}C NMR δ 27.7, 84.9, 117.2, 129.8, 136.4, 147.5.

t-Butyl benzylcarbamate (**5**): Colourless oil, (lit.[6] colourless oil); IR: 3338, 2978, 2925, 1691, 1440, 1230, 1158, 876 cm^{-1}; ^{1}H NMR δ 1.50 (s, 9H), 4.24 (d, J = 5.84 Hz, 2H), 4.85 (br s, 1H), 7.17–7.26 (m, 5H); ^{13}C NMR δ 28.3, 44.6, 127.2, 127.3, 128.5, 138.9, 155.8.

t-Butyl morpholine-4-carboxylate (**6**): Yellow oil, (lit.[10] yellow oil); IR: 2995, 1683, 1448, 1413, 1360, 1277, 1246, 1166, 1123, 1083, 860 cm^{-1}; ^{1}H NMR δ 3.52 (m, 4H), 3.36 (m, 4H), 1.51 (s, 9H); ^{13}C NMR δ 28.2, 43.7, 67.2, 79.5, 154.7.

t-Butyl-4-methylbenzylcarbamate (**7**): White solid, m.p. 69–71 °C (MeOH) (lit.[27] 72–73 °C); IR: 3395, 2980, 2920, 1680, 1600, 1508, 1360, 1320, 1295, 1260, 1172, 1000, 858, 761, 694 cm^{-1}; ^{1}H NMR δ 7.20 (m, 2H), 7.16 (m, 2H), 4.88 (s, 1H), 4.30 (m, 2H), 2.36 (s, 3H), 1.50 (s, 9H); ^{13}C NMR δ 21.1, 28.3, 44.5, 79.4, 127.5, 129.3, 135.8, 136.9, 155.8.

t-Butyl cyclohexylcarbamate (**8**): White solid, m.p. 63–65 °C (MeOH) (lit.[6] 65–67 °C); IR:3364, 2973, 2934, 2854, 1681, 1520 1448, 1366, 1315, 1251, 1233, 1168 cm^{-1}; ^{1}H NMR δ 4.42 (s, 1H), 3.44 (s, 1H), 1.90–1.93 (m, 2H), 1.57–1.71(m, 3H), 1.44 (s, 9H), 1.26–1.34 (m, 2H), 1.04–1.15 (m, 3H); ^{13}C NMR δ 22.0, 27.2, 28.3, 32.7, 48.2, 80.1, 155.7.

t-Butyl diethylcarbamate (**9**): Colourless oil, (lit.[26] colourless oil); IR: 2900, 2860, 1678, 1520, 1448, 1362, 1317, 1272 1160, 1020, 900 cm^{-1}; ^{1}H NMR δ 3.24 (m, 4H), 1.48 (s, 9H), 1.12 (m, 6H); ^{13}C NMR δ 13.8, 28.4, 41.3, 78.9, 155.3.

t-Butyl tert-butylcarbamate (**10**) Yellow oil, (lit.[10] yellow oil); IR: 3420, 2950, 1799, 1762, 1680, 1451, 1367, 1207, 1113, 1066 cm^{-1}; ^{1}H NMR δ 3.48 (s, 1H), 2.17 (s, 9H), 1.56 (s, 9H); ^{13}C NMR δ 27.3, 30.0, 49.9, 85.1, 147.1.

t-Butyl 1-hydroxy-2-methylpropan-2-ylcarbamate (**11**) White solid, m.p. 48–50 °C (MeOH) (lit.[26] 50–52 °C); IR: 3490, 3300, 2994, 2930, 1696, 1680, 1540, 1520, 1362, 1300, 1250, 1170, 1080, 1040 cm^{-1}; ^{1}H NMR δ 6.12 (s, 1H), 4.68 (s, 1H), 3.24 (s, 2H), 1.32(s, 9H), 1.02 (s, 6H). ^{13}C NMR δ 24.0, 29.1, 54.4, 67.8, 76.6, 152.7.

t-Butyl phenylcarbamate (**12**): Yellow oil, (lit.[6] yellow oil); IR: 3308, 1686, 1526, 1438, 1366, 1313, 1240, 1148, 1053, 908, 824, 745, 691 cm^{-1}; ^{1}H NMR δ 7.25–7.37(m, 4H), 7.01(m, 1H), 6.55(s, 1H), 1.51(s, 9H); ^{13}C NMR δ 28.3, 118.5, 123.0, 128.9, 138.3, 152.8.

t-Butyl p-tolylcarbamate (**13**): White solid, m.p. 84–86 °C (MeOH) (lit.[6] 86–87 °C); IR: 3333, 1696, 1596, 1523, 1365, 1314, 1233, 1153, 1049, 815, 771, 668 cm^{-1}; ^{1}H NMR δ 7.07–7.25(m, 4H), 6.43(s, 1H), 2.29(s, 3H), 1.51(s, 9H); ^{13}C NMR δ 20.7, 28.3, 118.7, 129.4, 132.5, 135.7, 152.9.

t-Butyl 4-methoxyphenylcarbamate (**14**): White solid, m.p. 92–93 °C (MeOH) (lit.[6] 94–95 °C); IR: 3364, 2987, 1692, 1519, 1368, 1234, 1157, 1025, 823, 771, 724, 624 cm^{-1}; ^{1}H NMR δ 7.27(m, 2H), 6.83(m, 2H), 6.45(s, 1H), 3.77(s, 3H), 1.50(s, 9H); ^{13}C NMR δ 28.3, 55.5, 80.3, 114.3, 120.8, 131.4, 153.3, 155.8.

t-Butyl 4-nitrophenylcarbamate (**15**): White solid, m.p. 110–112 °C (MeOH) (lit.[6] 112–113 °C). IR: 3372, 2988, 2965, 1720, 1586, 1521, 1496, 1462, 1365, 1305, 1281, 1106, 840, 763, 721, 621 cm^{-1}; ^{1}H NMR δ 8.02(m, 2H), 6.64(m, 2H), 4.28(s, 1H), 1.54(s, 9H); ^{13}C NMR δ 29.1, 51.6, 113.0, 125.3, 126.3, 137.7, 152.8.

t-Butyl 4-chlorophenylcarbamate (**16**): White solid, m.p. 101–102 °C (MeOH) (lit.[15] 102–104 °C); IR: 3352, 2982, 1804, 1728, 1596, 1525, 1368, 1156, 1067, 844, 773, 682 cm^{-1}; ^{1}H NMR δ: 7.31(m, 2H), 7.22(m, 2H), 6.73(s, 1H), 1.50(s, 9H); ^{13}C NMR δ: 28.3, 80.8, 119.8, 127.9, 128.9, 137.1, 152.8.

t-Butyl 4-bromophenylcarbamate (**17**): White solid, m.p. 100–102 °C (MeOH) (lit.[15] 100–103 °C); IR: 33370, 2983, 1695, 1588, 1513, 1392, 1233, 1154, 814, 767, 634 cm^{-1}; ^{1}H NMR δ: 7.39(m, 2H), 7.27(m, 2H), 6.51(s, 1H), 1.51(s, 9H); ^{13}C NMR δ: 28.1, 79.4, 120.6, 129.3, 130.8, 139.5, 153.8.

t-Butyl 2-hydroxyphenylcarbamate (**18**): Brown solid, m.p. 140–141 °C (MeOH) (lit.[15] 140–143 °C); IR: 3423, 3286, 3300, 2982, 2926, 1689, 1622, 1612, 1523, 1455, 1444, 1325, 1151, 1054, 749 cm^{-1}; ^{1}H NMR δ 8.16 (s, 1H), 7.05–7.12 (m, 2H), 7.02 (m, 1H), 6.87 (m, 1H), 6.70 (s, 1H), 1.56 (s, 9H); ^{13}C NMR δ 28.7, 81.4, 117.8, 121.2, 121.7, 125.8, 126.1, 147.7, 154.4.

t-Butyl 3-chlorophenylcarbamate (**19**): Brown solid, m.p. 63–64 °C (MeOH)(lit.[27] m.p. 66–68 °C); IR: 3368, 3306, 2987, 2938, 1695, 1592, 1519, 1400, 1268, 1237, 1178 cm^{-1}; ^{1}H NMR δ 7.47 (s, 1H), 7.11–7.19 (m, 2H), 7.01 (m, 1H), 6.53 (s, 1H), 1.52 (s, 9H); ^{13}C NMR δ 28.2, 85.1, 116.4, 122.7, 128.7, 134.5, 138.8, 146.7, 152.6.

t-Butyl 3-methoxyphenylcarbamate (**20**): Brown solid, m.p. 49–52 °C (MeOH) (lit.[14] 52–54 °C); IR: 3320, 2990, 2920, 1690, 1600, 1530, 1450, 1420, 1362, 1285, 1240, 1160, 1045, 1032, 960, 870, 842 cm^{-1}; ^{1}H NMR δ 7.21 (m, 1H), 7.12 (s, 1H), 6.86 (m, 1H), 6.67 (s, 1H), 6.61 (m, 1H), 3.86 (s, 3H), 1.53 (s, 9H); ^{13}C NMR δ 28.3, 55.3, 80.7, 104.1, 108.9, 110.7, 130.0, 139.8, 152.7, 160.3.

t-Butyl 4-hydroxyphenylcarbamate (**21**): White solid, m.p. 144–145 °C (MeOH) (lit.[15] 146 °C); IR: 3360, 2982, 1697, 1519, 1437, 1369, 1228, 1056, 904, 830, 627 cm^{-1}; ^{1}H NMR δ 7.17(m, 2H), 6.76(m, 2H), 6.51(s, 1H), 4.95(s, 1H), 1.55(s, 9H); ^{13}C NMR δ 28.4, 84.2, 116.5, 122.3, 136.5, 147.1, 154.2.

t-Butyl 1-(naphthalen-1-yl)ethylcarbamate (**22**): Yellow oil; IR: 3378, 2980, 2921, 1672, 1598, 1493, 1362, 1288, 1172, 842, 761, 688 cm^{-1}; ^1H NMR δ 7.02–7.71(m, 7H), 4.92(s, 1H), 4.17(m, 1H), 1.91(m, 3H), 1.51(s, 9H);^{13}C NMR δ 22.4, 28.1, 51.04, 79.4, 122.2, 123.7, 124.6, 124.4, 126.7, 127.8, 128.6, 134.5, 134.6, 154.3; HRMS calcd for $C_{17}H_{22}NO_2$ $[M+H]^+$: 272.1651, found: 272.1653.

t-Butyl 4-(2-hydroxyethoxy)phenylcarbamate (**23**): White solid, m.p. 108–110 °C; IR: 3482, 3320, 2982, 1696, 1590, 1515, 1496, 1368, 1277, 1106, 848, 676 cm^{-1}; ^1H NMR δ 7.24(m, 2H), 6.82(m, 2H), 6.42(s, 1H), 4.42(s, 1H), 4.02(m, 2H), 3.77(m, 2H), 1.51(s, 9H); ^{13}C NMR δ 28.3, 70.4, 59.8, 80.1, 114.7, 120.6, 131.5, 153.2, 157.6; HRMS calcd for $C_{13}H_{20}NO_4$ $[M+H]^+$: 254.1392, found: 254.1395.

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