Magnesium bistrifluoromethanesulfonimide catalysed three-component synthesis of protected homoallylic amines

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A one-pot, three-component reaction of an aldehyde, benzyl carbamate and allyltrimethylsilane in the presence of 3 mol% of magnesium bistrifluoromethanesulfonimide at room temperature has been shown to afford the corresponding protected homoallylic amine in high yield.

Keywords: magnesium bistrifluoromethanesulfonimide, three-component reaction, protected homoallylic amines

Homoallylic amines and their derivatives are useful intermediates in natural product synthesis[1,2] and acylated homoallylic amines have many synthetic applications.[3] Homoallylic amines are commonly prepared by the allylation of aldimines which had previously been prepared from aldehydes and amines using allylic nucleophiles such as allyl silanes and allyl organometallics.[4–7] To avoid the prior synthesis of aldimines, a direct one-pot, three-component reaction has been reported for the synthesis of homoallyl amines in the presence of a catalyst such as $BF_3{\cdot}OEt_2$,[8] phosphomolybdic acid,[9] $Cu(OTf)_2$,[10] $Bi(OTf)_3$,[11] $Sc(OTf)_3$[12] and I_2.[13] Although each of the above methods has its own merits, some of these earlier methods employed for this one-pot conversion have drawbacks such as the use of toxic organic solvents, long reaction times and unsatisfactory yields. Therefore, the development of convenient approaches with mild reaction conditions and high yields for the preparation of homoallylic amines is still desirable.

In recent years, metal bistrifluoromethanesulfonimides [$M(NTf_2)_n$] have been successfully used for the acetylation of phenols and alcohols,[14] [2+2] cycloadditions of siloxy alkynes with carbonyl compounds,[15] Friedel-Crafts acylation reactions,[16] cycloisomerisation of 1,6-dienes[17] and aminolysis of lactones with amines.[18] Previously, we have reported the use of $Eu(NTf_2)_3$[19] and magnesium bistrifluoromethanesulfonimide [$Mg(NTf_2)_2$][20] as efficient catalysts in organic synthesis. $Mg(NTf_2)_2$ is commercially available, cheaper and not sensitive to air, and therefore better suited for catalytic use. Following our interest in the catalytic uses of $M(NTf_2)_n$, we report a facile and efficient procedure for the synthesis of protected homoallylic amines in the presence of 3 mol% of $Mg(NTf_2)_2$ at room temperature (Scheme 1).

In order to establish optimum conditions for the synthesis of homoallylic amines, different amount of $Mg(NTf_2)_2$ and various solvents were examined. Using the reaction of benzaldehyde, benzyl carbamate, and allyltrimethylsilane as a model, initially we investigated the effect of the amount of $Mg(NTf_2)_2$ on the reaction. As shown in Table 1, in the absence of this catalyst, only a trace amount of the desired product was produced. However, even 0.5 mol% of $Mg(NTf_2)_2$ accelerated the reaction, although the corresponding product was obtained in a low yield and at a slow rate. Increasing the amount of $Mg(NTf_2)_2$ not only enhanced the product yield, but also reduced the reaction time. Rate enhancement was observed when 10 mol% of $Mg(NTf_2)_2$ was used, but the yield was relatively low. Considering the amount of the catalyst, reaction time and yield, 3 mol% of $Mg(NTf_2)_2$ was found to be the most effective.

Table 1 Effect of different amount of $Mg(NTf_2)_2$ on the reaction of benzaldehyde, benzyl carbamate and allyltrimethylsilane[a]

Entry	$Mg(NTf_2)_2$/mol%	Time/min	Yield[b]/%
1	0	180	Trace
2	0.5	60	67
3	1	25	85
4	3	10	95
5	5	8	95
6	7	8	94
7	10	5	90

[a] The reactions were were performed at room temperature in EtOH.
[b] Isolated yield.

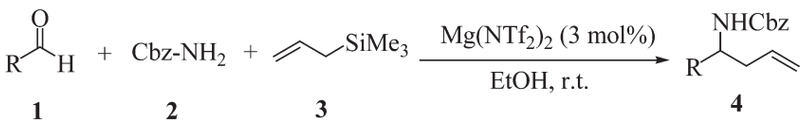
The effect of different solvents on the reaction was then studied and the results are listed in Table 2. As shown in Table 2, moderate yields were obtained in dichloromethane, acetonitrile and toluene. *N*,*N*-Dimethylformamide and dimethylsulfoxide afforded lower yields. After screening different solvents, ethanol was the solvent of choice. It not only afforded the product in excellent yield, but also with a higher reaction rate.

To demonstrate the generality of this method, other aldehydes were also used to react with benzyl carbamate and allyltrimethylsilane in the presence of $Mg(NTf_2)_2$ (3 mol%) in

Table 2 Effect of different solvent on the reaction of benzaldehyde, benzyl carbamate and allyltrimethylsilane[a]

Entry	Solvent	Time/min	Yield[b]/%
1	CH_2Cl_2	20	85
2	Toluene	20	82
3	MeCN	15	88
4	EtOH	10	95
5	DMF	45	68
6	DMSO	45	56

[a] The reactions were carried out in different solvents at room temperature with 3 mol% of $Mg(NTf_2)_2$.
[b] Isolated yield.



1 **2** **3** **4**

Scheme 1

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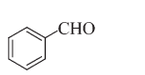
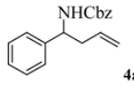
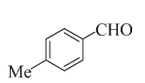
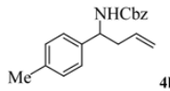
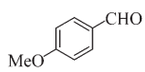
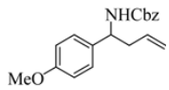
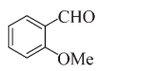
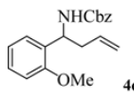
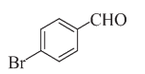
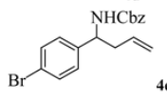
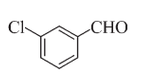
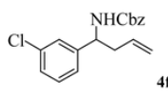
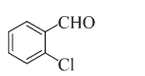
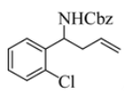
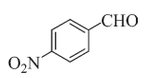
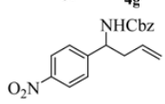
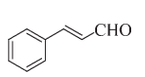
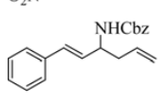
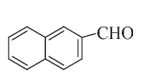
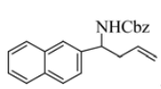
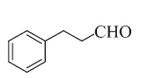
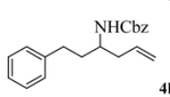
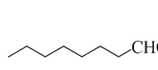
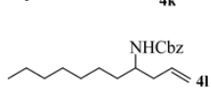
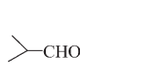
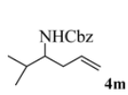
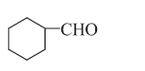
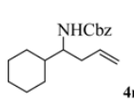
Table 3 Synthesis of Cbz-protected homoallylic amines catalysed by $Mg(NTf_2)_2$[a]

Entry	Aldehyde	Product	Time /min	Yield[b] /%
1		 **4a**	10	95
2		 **4b**	10	94
3		 **4c**	15	94
4		 **4d**	10	96
5		 **4e**	20	89
6		 **4f**	15	96
7		 **4g**	20	88
8		 **4h**	25	84
9		 **4i**	10	97
10		 **4j**	15	90
11		 **4k**	20	86
12		 **4l**	20	89
13		 **4m**	20	85
14		 **4n**	20	89

[a] The reactions were conducted in the presence of 3 mol% of $Mg(NTf_2)_2$ at room temperature in EtOH.
[b] Isolated yield.

ethanol at room temperature. The results are summarised in Table 3. As shown in Table 3, in all cases, aromatic and aliphatic aldehydes afforded the desired products smoothly. Aromatic aldehydes containing both electron-donating and electron-withdrawing groups on the aromatic ring worked well (Table 3, entries 1–8). The three-component reactions derived from an *α*,*β*-unsaturated aldehyde such as cinnamaldehyde (Table 3, entry 9) and a sterically-hindered aldehyde such as 2-naphthaldehyde (Table 3, entry 10) also afforded the corresponding homoallyllic amines in high yields. Additionally, the method was suitable for the conversion of aliphatic aldehydes (Table 3, entries 11-14), but relatively longer times were required. However, ketones did not yield any products under the present reaction conditions.

The structures of all products were proved on the basis of their spectral (^{1}H and ^{13}C NMR) data. For example, the ^{1}H NMR spectrum of $CH{=}CH_2$ in compound **4a** exhibited a doublet (J = 10.2 Hz) at 4.98 ppm, two doublets (J = 17.2, 1.1 Hz) at 5.12 ppm and a multiplet at 5.55–5.70 ppm. The formation of allylic amines was not detected from ^{1}H NMR and ^{13}C NMR spectra. Isomerisation of the double bond in products had not occured.

In conclusion, $Mg(NTf_2)_2$ was found to be an efficient catalyst for the one-pot, three-component reaction of aldehydes, benzyl carbamate and allyltrimethylsilane at room temperature to afford the corresponding protected homoallylic amines. The advantages of this protocol include mild reaction conditions, easy workup, high yields, and the use of non-toxic organic solvent and a catalytic amount and commercially available catalyst.

Experimental

Melting points were determined on an XT4A electrothermal apparatus equipped with a microscope and are uncorrected. NMR spectra were recorded on a Bruker Avance 400 spectrometer in $CDCl_3$ with TMS as an internal standard. IR spectra were recorded on a Nicolet FTIR-750 spectrometer. Elemental analyses were performed on a Perkin Elmer 240-C instrument. All solvents were dried by standard procedures. The benzaldehyde was distilled prior to use. All other reagents were commercially available products and were used without further purification.

Synthesis of protected homoallylic amines; general procedure
A mixture of the aldehyde **1** (1 mmol), benzyl carbamate **2** (1 mmol), allyltrimethylsilane **3** (1.2 mmol) and $Mg(NTf_2)_2$ (3 mol%) in absolute ethanol (2 mL) was stirred at room temperature until the reaction was complete (monitored by TLC). The reaction mixture was quenched with saturated NH_4Cl solution and the aqueous layer was extracted twice with EtOAc. The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated under vacuum. The crude product was purified by column chromatography on silica gel to give the desired product **4**.

N-Benzyloxycarbonyl-1-phenylbut-3-enylamine (**4a**): M.p. 68–69 °C (lit.[12] 67–68 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.31–2.50 (m, 2H), 4.60–4.83 (m, 1H), 4.90–4.96 (m, 3H), 4.98 (d, J = 10.2 Hz, 1H), 5.12 (dd, J = 17.2, 1.1 Hz, 1H), 5.55–5.70 (m, 1H), 7.11–7.20 (m, 5H), 7.20–7.31 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 41.2, 54.6, 66.8, 118.6, 126.6, 127.3, 128.2, 128.4, 128.7, 133, 136.1, 142.2, 155.9. IR (KBr) ν: 3051, 1712, 1427, 1260, 745 cm^{-1}. Anal. Calcd for $C_{18}H_{19}NO_2$: C, 76.87, H, 6.76; N, 4.98. Found: C, 76.80; H, 6.80; N, 4.91%.

N-Benzyloxycarbonyl-1-(4-methylphenyl)but-3-enylamine (**4b**): M.p. 62–63 °C (lit.[21] 62–64 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.37 (s, 3H), 2.46–2.56 (m, 2H), 4.60–4.71 (m, 1H), 4.98–5.15 (m, 5H), 5.60–5.65 (m, 1H), 7.01–7.18 (m, 4H), 7.29–7.45 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 21.6, 40.6, 54.8, 67.5, 100.2, 118.3, 126.8, 128.4, 128.8, 129.0, 134.2, 136.6, 137.1, 156.8. IR (KBr) ν: 3357, 1690, 1522, 1455, 1266 cm^{-1}. Anal. Calcd for $C_{19}H_{21}NO_2$: C, 77.29; H, 7.12; N, 4.75. Found: C, 77.36; H, 7.07; N, 4.83%.

N-Benzyloxycarbonyl-1-(4-methoxyphenyl)but-3-enylamine (**4c**): M.p. 71–72 °C (lit.[12] 70–71 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.50–2.58 (m, 2H), 3.76 (s, 3H), 4.71–4.80 (m, 1H), 5.04–5.21 (m, 5H), 5.55–5.76 (m, 1H), 6.83 (d, J = 8.6 Hz, 2H), 7.17 (d, J = 7.8 Hz, 2H), 7.31–7.40 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 40.9, 53.5, 55.0, 86.6, 113.8, 118.5, 127.3, 128.0, 128.3, 133.6, 136.4, 155.6, 158.9. IR (KBr) ν: 3053, 1717, 1501, 1429, 1264, 735 cm^{-1}. Anal. Calcd for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.21; H, 6.73; N, 4.58%.

N-Benzyloxycarbonyl-1-(2-methoxyphenyl)but-3-enylamine (**4d**): M.p. 90–91 °C (lit.[3] 90–91 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.52–2.59 (m, 2H), 3.86 (s, 3H), 4.92–5.17 (m, 5H), 5.64–5.71 (m, 2H), 6.88–6.93 (m, 2H), 7.18–7.33 (m, 7H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 40.1, 52.9, 55.8, 66.7, 111.0, 117.1, 120.7, 128.1, 128.3, 128.4,

128.5, 129.8, 134.4, 136.8, 155.8, 157.2. IR (KBr) ν: 3330, 1688, 1602, 1537, 1490 cm^{-1}. Anal. Calcd for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.24; H, 6.70; N, 4.57%.

N-Benzyloxycarbonyl-1-(4-bromophenyl)but-3-enylamine (**4e**): M.p. 86–87 °C (lit.[12] 87–88 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.40–2.58 (m, 2H), 4.66–4.89 (m, 1H), 4.91–5.24 (m, 5H), 5.50–5.68 (m, 1H), 7.16 (d, *J* = 7.6 Hz, 2H), 7.22–7.45 (m, 5H), 7.40 (d, *J* = 8.2 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 40.6, 53.9, 66.6, 118.9, 121.7, 127.9, 128.2, 128.5, 131.0, 133.2, 136.2, 141.4, 155.5. IR (KBr) ν: 3051, 1711, 1599, 1416, 1261, 745 cm^{-1}. Anal. Calcd for $C_{18}H_{18}BrNO_2$: C, 60.01; H, 5.03; N, 3.89. Found: C, 60.05; H, 5.10; N, 3.80%.

N-Benzyloxycarbonyl-1-(3-chlorophenyl)but-3-enylamine (**4f**): M.p. 62–63 °C (lit.[12] 62–63 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.30–2.51 (m, 2H), 4.61–4.79 (m, 1H), 4.88–5.18 (m, 3H), 5.21 (d, *J* = 10.2 Hz, 1H), 5.25 (dd, *J* = 17.2, 1.4 Hz, 1H), 5.56–5.66 (m, 1H), 7.03 (d, *J* = 6.3 Hz, 1H), 7.11–7.48 (m, 8H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 40.5, 53.7, 66.2, 119.3, 124.9, 126.0, 127.1, 128.0, 128.8, 129.3, 133.6, 134.8, 136.6, 155.1. IR (KBr) ν: 3051, 1717, 1505, 1260, 737, 708 cm^{-1}. Anal. Calcd for $C_{18}H_{18}ClNO_2$: C, 68.46; H, 5.74; N, 4.43. Found: C, 68.51; H, 5.66; N, 4.52.

N-Benzyloxycarbonyl-1-(2-chlorophenyl)but-3-enylamine (**4g**): M.p. 65–66 °C (lit.[3] 64–65 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.33–2.58 (m, 2H), 5.05–5.24 (m, 6H), 5.62–5.71 (m, 1H), 7.14–7.38 (m, 9H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 39.0, 52.1, 67.3, 118.9, 127.3, 127.5, 128.3, 128.5, 128.6, 130.5, 132.7, 133.5, 136.9, 139.4, 155.8. IR (KBr) ν: 3320, 1689, 1544, 1433cm^{-1}. Anal. Calcd for $C_{18}H_{18}ClNO_2$: C, 68.46; H, 5.74; N, 4.43. Found: C, 68.54; H, 5.69; N, 4.49%.

N-Benzyloxycarbonyl-1-(4-nitrophenyl)but-3-enylamine (**4h**): M.p. 86–88 °C (lit.[12] 88–89 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.33–2.55 (m, 2H), 4.70–4.88 (m, 1H), 4.94–5.29 (m, 5H), 5.40–5.65 (m, 1H), 6.97–7.34 (m, 5H), 7.37 (d, *J* = 7.6 Hz, 2H), 8.16 (d, *J* = 7.8 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 40.3, 54.7, 67.1, 119.6, 123.4, 127.3, 128.2, 128.3, 128.5, 132.9, 147.0, 149.6, 155.1. IR (KBr) ν: 3044, 2300, 1711, 1516, 1263, 737 cm^{-1}. Anal. Calcd for $C_{18}H_{18}N_2O_4$: C, 66.25; H, 5.56; N, 8.58. Found: C, 66.21; H, 5.50; N, 8.63%.

N-Benzyloxycarbonyl-1-(2-phenylethenyl)but-3-enylamine (**4i**): M.p. 71–72 °C (lit.[3] 72–74 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.40–2.48 (m, 2H), 4.41–4.57 (m, 1H), 4.80–4.85 (m, 1H), 5.11–5.15 (m, 2H), 5.18 (d, *J* = 10.2 Hz, 1H), 5.23 (dd, *J* = 17.2, 1.1 Hz, 1H), 5.70–5.85 (m, 1H), 6.12 (dd, *J* = 15.9, 6.0 Hz, 1H), 6.63 (d, *J* = 15.9 Hz, 1H), 7.24–7.30 (m, 10H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 40.1, 52.6, 66.2, 118.9, 126.4, 127.2, 128.0, 128.4, 128.5, 128.7, 129.4, 130.9, 133.3, 136.9, 136.9,155.8. IR (KBr) ν: 3053, 3028, 2917, 1716, 1638, 1602, 1505, 1318, 1264, 968, 746, 693 cm^{-1}. Anal. Calcd for $C_{20}H_{21}NO_2$: C, 78.18; H, 6.84; N, 4.56. Found: C, 78.11; H, 6.80; N, 4.67%.

N-Benzyloxycarbonyl-1-(2-naphtyl)but-3-enylamine (**4j**): M.p. 67–68 °C (lit.[21] 65–67 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.57–2.65 (m, 2H), 4.76–4.84 (m, 1H), 4.93–5.18 (m, 5H), 5.60–5.69 (m, 1H), 7.25–7.53 (m, 7H), 7.69–7.80 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 40.5, 54.8, 66.9, 118.6, 124.1, 125.4, 125.7, 126.6, 127.4, 127.8, 128.3, 128.8, 132.2, 132.6, 133.1, 135.4, 139.5, 155.4. IR (KBr) ν: 3360, 1683, 1520, 1466, 1256 cm^{-1}. Anal. Calcd for $C_{22}H_{21}NO_2$: C, 79.76; H, 6.34; N, 4.23. Found: C, 79.71; H, 6.30; N, 4.31%.

N-Benzyloxycarbonyl-1-(2-phenylethyl)but-3-enylamine (**4k**): M.p. 50–52 °C (lit.[12] 50–51 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.58–1.69 (m, 1H), 1.72–1.80 (m, 1H), 2.11–2.26 (m, 2H), 2.44–2.67 (m, 2H), 3.60–3.78 (m, 1H), 4.58 (d, *J* = 7.8 Hz, 1H), 4.99–5.12 (m, 4H), 5.64–5.79 (m, 1H), 7.09–7.17 (m, 3H), 7.11–7.27 (m, 7H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 32.0, 36.4, 39.6, 50.9, 66.1, 118.3, 125.9, 128.1, 128.3, 128.4, 128.5, 133.5, 136.1, 141.4, 156.7. IR (KBr) ν: 3066, 1690, 1533, 1458, 1240, 1041, 735, 696 cm^{-1}. Anal. Calcd for $C_{20}H_{23}NO_2$: C, 77.67; H, 7.44; N, 4.53. Found: C, 77.74; H, 7.49; N, 4.50%.

N-Benzyloxycarbonyl-1-heptylbut-3-enylamine (**4l**): M.p. 50–51 °C (lit.[12] 51–52 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.86 (t, *J* = 6.3 Hz, 3H), 1.20–1.45 (m, 12H), 2.02–2.30 (m, 2H), 3.51–3.80 (m, 1H), 4.41–4.60 (m, 1H), 5.01–5.16 (m, 4H), 5.70–5.85 (m, 1H), 7.29–7.41 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 14.6, 22.2, 25.4, 29.0, 29.5, 31.7, 34.9, 39.4, 50.5, 66.6, 117.8, 128.4, 128.6, 134.3, 136.9, 156.1. IR (KBr) ν: 3057, 1713, 1500, 1436, 1264, 736 cm^{-1}. Anal. Calcd for $C_{19}H_{29}NO_2$: C, 75.21; H, 9.63; N, 4.62. Found: C, 75.16; H, 9.55; N, 4.66%.

N-Benzyloxycarbonyl-1-isopropylbut-3-enylamine (**4m**): Oil.[12] ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.85 (d, *J* = 6.8 Hz, 3H), 0.96 (d, *J* = 6.8 Hz, 3H), 1.66–1.79 (m, 1H), 2.06–2.13 (m, 1H), 2.20–2.35 (m, 1H), 3.50–3.64 (m, 1H), 4.55–4.70 (m, 1H), 5.01–5.18 (m, 4H), 5.73–5.80 (m, 1H), 7.29–7.42 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 17.9, 19.0, 31.8, 36.5, 55.9, 66.3, 117.8, 128.1, 128.3, 128.5, 134.4, 136.9, 156.6. IR (neat) ν: 3077, 1695, 1537, 1246, 736 cm^{-1}. Anal. Calcd for $C_{15}H_{21}NO_2$: C, 72.84; H, 8.56; N, 5.66. Found: C, 72.95; H, 8.50; N, 5.61%.

N-Benzyloxycarbonyl-1-cyclohexylbut-3-enylamine (**4n**): M.p. 64–66 °C (lit.[3] 64–65 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.98–1.27 (m, 6H), 1.56–1.78 (m, 5H), 2.18–2.30 (m, 2H), 3.56–3.68 (m, 1H), 4.48–4.63(m, 1H), 5.01–5.11 (m, 4H), 5.70–5.79 (m, 1H), 7.28–7.40 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 26.1, 26.2, 26.4, 28.8, 36.5, 39.4, 41.1, 55.0, 66.8, 117.7, 128.5, 128.4, 134.3, 136.6,156.3. IR (KBr) ν: 3436, 2930, 2853, 1724, 1506, 1446, 1342, 1214 cm^{-1}. Anal. Calcd for $C_{18}H_{25}NO_2$: C, 75.26; H, 8.71; N, 4.88. Found: C, 75.32; H, 8.67; N, 4.81%.

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