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Magnesium Bistrifluoromethanesulfonimide Catalyzed Three-component Synthesis of Protected Homoallylic Amines

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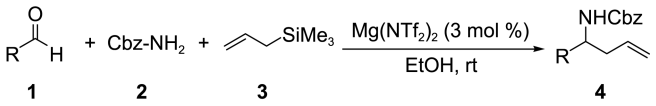
A mild and efficient procedure has been developed for the one-pot, three-component reaction of aldehydes, benzyl carbamate and allyltrimethylsilane in the presence of 3 mol % of magnesium bistrifluoromethanesulfonimide at room temperature to afford the corresponding protected homoallylic amines in high yields.

Key Words : Magnesium bistrifluoromethanesulfonimide, Three-component reaction, Protected homoallylic amines

Introduction

Homoallyl amines and their derivatives are useful intermediates in natural product synthesis and also precursors to β-amino acids and β-lactams.[1] Diverse homoallylamines possess a particular skeleton in which chemical units might be involved in the construction of heterocyclic rings of different size. In addition, making asymmetric synthesis of homoallylic amines, they could offer real facilities to assemble chiral heterocycles in a straight way. On the other hand, being biogenic amines, the homoallyl amines represent very attractive biological targets.[2] Moreover, acylated homoallylic amines are important synthons for many synthetic applications.[3] Homoallylic amines are commonly prepared by the allylation of aldimines prepared from aldehydes and amines in advance 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 merit, some of the methods employed earlier for this one-pot conversion are associated with certain 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 to 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] cycloisomerization of 1,6-dienes[17] and aminolysis of lactones with amines.[18] Previously, we have reported the use of $Eu(NTf_2)_3$ as an efficient catalyst for the synthesis of bis(indol-3-yl)methanes.[19] Now we choose $Mg(NTf_2)_2$ as a catalyst because it 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 herein 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).



Scheme 1. Synthesis of Cbz-protected homoallylic amines catalyzed by $Mg(NTf_2)_2$.

Results and Discussion

In order to establish the optimum condition 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 $Mg(NTf_2)_2$ could accelerate the reaction, but the reaction afforded the the corresponding product in a low yield and with a slow rate. The increase in the amount of $Mg(NTf_2)_2$ not only

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

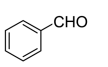
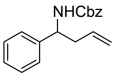
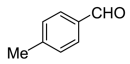
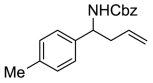
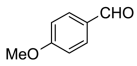
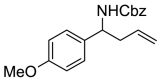
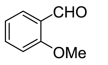
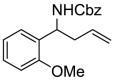
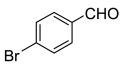
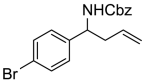
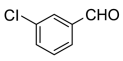
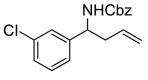
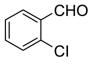
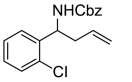
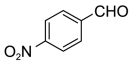
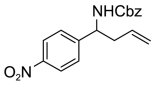
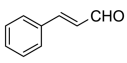
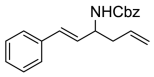
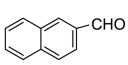
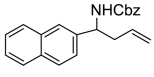
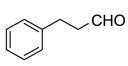
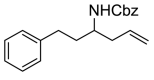
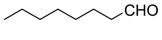
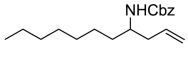
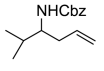
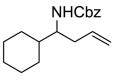
[a]The reactions were were performed at room temperature in EtOH. [b]Isolated yield.

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.

Table 3. Synthesis of Cbz-protected homoallylic amines catalyzed 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.

enhances the product yield, but also reduces the reaction time. Rate enhancement was observed when 10 mol % of $Mg(NTf_2)_2$ was used, but the yield was relatively lower. Considering the the amount of the catalyst, reaction time and yield, 3 mol % of $Mg(NTf_2)_2$ was found to be the most effective.

Then, the effect of different solvent on the reaction was 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 came out as the solvent of choice, which not only afforded the product in excellent yield, but also with higher reaction rate. The higher catalytic activity of $Mg(NTf_2)_2$ in ethanol may be due to the increased solubility and the Lewis acidity of it.

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 ethanol at room temperature and the results are summarized 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 in 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-naphthalaldehyde (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 observed. However, ketones did not yield the products under the present reaction conditions.

Conclusion

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 low toxic organic solvent and a catalytic amount and commercially available catalyst.

Experimental Section

General. Melting points were determined on an XT4A electrothermal apparatus equipped with a microscope and are uncorrected. ^{1}H NMR spectra were recorded on a Bruker Avance 400 spectrometer in $CDCl_3$ with TMS as an internal standard. ^{13}C NMR spectra were recorded on NMR spectrometer AC100 in $CDCl_3$. 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.

General Procedure for the Synthesis of Protected Homoallylic Amines. A mixture of 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 completed (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):** mp 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), 4.90-5.12 (m, 5H), 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, cm^{-1}) ν 3051, 1712, 1427, 1260, 745. Anal. Calcd for $C_{18}H_{19}NO_2$: C, 76.87; H, 6.76; N, 4.98. Found: C, 76.80; H, 6.85; N, 4.91.

***N*-Benzyloxycarbonyl-1-(4-methylphenyl)but-3-enylamine (4b):** mp 62-63 °C (Lit.[20] 62-64 °C); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.37 (s, 3H), 2.46-2.53 (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, cm^{-1}) ν 3357, 1690, 1522, 1455, 1266. 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):** mp 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, cm^{-1}) ν 3053, 1717, 1501, 1429, 1264, 735. Anal. calcd for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.21; H, 6.83; N, 4.58.

***N*-Benzyloxycarbonyl-1-(2-methoxyphenyl)but-3-enylamine (4d):** mp 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, cm^{-1}) ν 3330, 1688, 1602, 1537, 1490. Anal. calcd for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.20; H, 6.85; N, 4.59.

***N*-Benzyloxycarbonyl-1-(4-bromophenyl)but-3-enylamine (4e):** mp 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, cm^{-1}) ν 3051, 1711, 1599, 1416, 1261, 745. Anal. calcd for $C_{18}H_{18}BrNO_2$: C, 60.01; H, 5.03; N, 3.89. Found: C, 60.07; H, 5.10; N, 3.80.

***N*-Benzyloxycarbonyl-1-(3-chlorophenyl)but-3-enylamine (4f):** mp 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), 4.88-5.25 (m, 5H), 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, cm^{-1}) ν 3051, 1717, 1505, 1260, 737, 708. Anal. calcd for $C_{18}H_{18}ClNO_2$: C, 68.46; H, 5.74; N, 4.43. Found: C, 68.49; H, 5.66; N, 4.50.

***N*-Benzyloxycarbonyl-1-(2-chlorophenyl)but-3-enylamine (4g):** mp 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, cm^{-1}) ν 3320, 1689, 1544, 1433. Anal. Calcd for $C_{18}H_{18}ClNO_2$: C, 68.46; H, 5.74; N, 4.43. Found: C, 68.51; H, 5.67; N, 4.49.

***N*-Benzyloxycarbonyl-1-(4-nitrophenyl)but-3-enylamine (4h):** mp 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, cm^{-1}) ν 3044, 2300, 1711, 1516, 1263, 737. Anal. Calcd for $C_{18}H_{18}N_2O_4$: C, 66.25; H, 5.56; N, 8.58. Found: C, 66.19; H, 5.50; N, 8.67.

***N*-Benzyloxycarbonyl-1-(2-phenylethenyl)but-3-enylamine (4i):** mp 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), 4.83 (brs, 1H), 5.11-5.23 (m, 4H), 5.70-5.85 (m, 1H), 6.12 (dd, J = 6.0, 15.9 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, cm^{-1}) ν 3053, 3028, 2917, 1716, 1638, 1602, 1505, 1318, 1264, 968, 746, 693. 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):** mp 67-68 °C (Lit.[20] 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, cm^{-1}) ν 3360, 1683, 1520, 1466, 1256. 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):** mp 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, cm^{-1}) ν 3066, 1690, 1533, 1458, 1240, 1041, 735, 696. Anal. Calcd for $C_{20}H_{23}NO_2$: C, 77.67; H, 7.44; N, 4.53. Found: C, 77.76; H, 7.52; N, 4.44.

***N*-Benzyloxycarbonyl-1-heptylbut-3-enylamine (4l):** mp 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, cm^{-1}) ν 3057, 1713, 1500, 1436, 1264, 736. 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, 4.69.

***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, cm^{-1}) ν 3077, 1695, 1537, 1246, 736. 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, 5.57.

***N*-Benzyloxycarbonyl-1-cyclohexylbut-3-enylamine (4n):** mp 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, cm^{-1}) ν 3436, 2930, 2853, 1724, 1506, 1446, 1342, 1214. Anal. Calcd for $C_{18}H_{25}NO_2$: C, 75.26; H, 8.71; N, 4.88. Found: C, 75.36; H, 8.67; N, 4.81.

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