Copper-Catalyzed $\alpha$-Amination of Aliphatic Aldehydes

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I. General Procedures

All reactions were carried out without exclusion of air or moisture unless otherwise stated. Copper salts, and peroxides such as tert-butyl hydroperoxide (~5.5 M in decane) (TBHP), di-tert-butyl peroxide, and tert-butyl benzoylperoxide were purchased from commercial suppliers, and used directly as received. Commercial solvents and reagents were used without further purification. Secondary amines 1b\textsuperscript{1-2}, enamine 1c\textsuperscript{3}, and aliphatic aldehydes 2 (f, h)\textsuperscript{4-5} were synthesized according to literatures.\textsuperscript{1-5} Reactions were monitored through thin layer chromatography [Merck 60 F254 precoated silica gel plate (0.2 mm thickness)]. Subsequent to elution, spots were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible using basic solution of potassium permanganate or acidic solution of ceric molybdate as stain, followed by heating on a hot plate. Flash chromatography was performed using Merck silica gel 60 with distilled solvents. Infrared spectra were recorded on a Shimadzu IR Prestige-21 FT-IR. Liquid samples were examined as film between NaCl salt plates. HRMS spectra were recorded on a Waters Q-Tof Permier Spectrometer. \textsuperscript{1}H NMR and \textsuperscript{13}C NMR spectra were recorded using Bruker Avance 300, 400 and 500 MHz spectrometers. Chemical shifts for \textsuperscript{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from SiMe\textsubscript{4} (δ 0.0) and relative to the signal of chloroform-d (δ 7.260, singlet). Multiplicities were given as: s (singlet); brs (broad singlet); d (doublet); t (triplet); q (quartet); dd (doublets of doublet); ddd (doublets of doublets of doublet); td (triplet of doublet); m (multiplets); ddt (doublet of doublet of triplet) and etc. Coupling constants are reported as a $J$ value in Hz. Carbon nuclear magnetic resonance spectra (\textsuperscript{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from SiMe\textsubscript{4} (δ 0.0) and relative to the signal of chloroform-d (δ 77.00, triplet).

II. Optimization of the reaction conditions

Among the various solvents screened (Table 1, entries 2 and 11-14), acetonitrile emerged as the best medium for this reaction, rendering the desired product in 80% isolated yield (Table 1, entry 2). Other copper catalysts were also examined (Table 1, entries 15-20). CuBr and CuBr\textsubscript{2} also catalyzed the reaction to afford the desired product 3a in moderate yield under the same conditions (Table 1, entries 15, 17). Other copper salts such as CuCl, CuCl\textsubscript{2}, Cu(OAc)\textsubscript{2} and Cu(OTf)\textsubscript{2} showed low or even no catalytic activities for this reaction (Table 1, entries 16, 18-20).
Table 1: Optimization of the reaction conditions

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>Oxidant</th>
<th>Solvent</th>
<th>T (°C)</th>
<th>t (h)</th>
<th>Yield (%)</th>
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<tr>
<td>1</td>
<td>CuI</td>
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<tr>
<td>3</td>
<td>—</td>
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<td>24</td>
<td>0</td>
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<tr>
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<td>24</td>
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<tr>
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<td>50</td>
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<td>50</td>
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<td>MeCN</td>
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<tr>
<td>20</td>
<td>Cu(OTf)$_2$</td>
<td>tBuOOH</td>
<td>MeCN</td>
<td>40</td>
<td>24</td>
<td>trace</td>
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$^a$ Reaction conditions: Dibenzylamine (0.5 mmol, 1 equiv.), butyraldehyde (0.75 mmol), copper catalyst (0.4 equiv.), oxidant (1.1 equiv.), methanol (0.4 mL), solvent (2 mL). $^b$ Isolated yields based on dibenzylamine. $^c$ The reaction was performed using 0.2 equiv. CuI. $^d$ The reaction was performed using 0.2 mL MeOH. $^e$ Dibenzylamine (0.5 mmol), butyraldehyde (0.5 mmol). $^f$ Dibenzylamine (0.75 mmol), butyraldehyde (0.5 mmol).
III

General Experimental Procedure for Copper-Catalyzed $\alpha$-Amination of Aliphatic Aldehydes

Typical procedure for copper-catalyzed $\alpha$-amination of aliphatic aldehydes for the synthesis of $\alpha$-amino acetics using secondary amines with readily removable protecting groups as a nitrogen source (dibenzyamine $1a$ and butyraldehyde $2a$ as a model system): tert-butyl hydroperoxide (~5.5 M in decane) (0.1 mL, ~0.55 mmol) was added to a mixture of CuI (38 mg, 0.2 mmol), dibenzyamine $1a$ (98 mg, 0.5 mmol), and butyraldehyde $2a$ (54 mg, 0.75 mmol) in methanol (0.4 mL)/acetonitrile (2.0 mL) at room temperature. The mixture was stirred at 40 °C until dibenzyamine $1a$ was completely converted by TLC detection. The resulting reaction mixture was mixed with a small amount of silica gel and concentrated. The crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product $3a$ as a light yellowish oil (0.125 g, 80% yield).

$N,N$-dibenzyl-1,1-dimethoxybutan-2-amine ($3a$): $R_f = 0.70$ (hexane: ethyl acetate = 3:1); $^1H$ NMR (CDCl$_3$, 400 MHz) δ 7.38-7.36 (m, 4H), 7.30-7.26 (m, 4H), 7.22-7.18 (m, 2H), 4.37 (d, $J = 5.16$ Hz, 1H), 3.74 (dd, $J = 50.34$, 13.61 Hz, 4H), 3.35 (s, 3H), 3.32 (s, 3H), 2.65-2.60 (m, 1H), 1.61-1.44 (m, 2H), 0.93 (t, $J = 7.43$ Hz, 3H) ppm; $^{13}C$ NMR (CDCl$_3$, 100 MHz) δ 140.7 (C x 2), 129.0 (CH x 4), 128.0 (CH x 4), 126.6 (CH x 2), 107.4, 59.4, 54.8, 54.44 (CH$_2$ x 2), 54.38, 19.5, 12.0 ppm; FTIR (neat): $v = 3015, 1452, 1215, 754, 700, 494$ cm$^{-1}$; HRMS (ESI, m/z): calcd for C$_{20}$H$_{28}$NO$_2^+$ [M+H]$^+$ 314.2120, found: 314.2119.
**N,N-dibenzyl-1,1-dimethoxy-3-methylbutan-2-amine (3b):** The product was prepared by above general procedure and the same chemicals except employing dibenzylation (1a) (98 mg, 0.5 mmol), and 3-methylbutanal (2b) (64 mg, 0.75 mmol). After 24 h, the crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3b as a light yellowish oil (0.108 g, 66% yield); $R_f = 0.76$ (hexane: ethyl acetate = 3:1); $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.38-7.36 (m, 4H), 7.30-7.26 (m, 4H), 7.22-7.18 (m, 2H), 4.51 (d, $J = 4.28$ Hz, 1H), 3.77 (dd, $J = 102.13$, 13.70 Hz, 1H), 3.40 (s, 3H), 3.37 (s, 3H), 2.46-2.43 (m, 1H), 2.05-1.97 (m, 1H), 0.98 (t, $J = 6.82$ Hz, 3H), 0.83 (t, $J = 6.68$ Hz, 3H) ppm; $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 140.7 (C x 2), 129.2 (CH x 4), 128.0 (CH x 4), 126.6 (CH x 2), 107.1, 62.5, 55.5 (CH$_2$ x 2), 55.2, 54.8, 27.2, 20.9, 20.7 ppm; FTIR (neat): $\nu = 3015, 1215, 754, 700, 494$ cm$^{-1}$; HRMS (ESI, m/z): calcd for C$_{21}$H$_{30}$NO$_2$ $^+ [M+H]^+$ 328.2277, found: 328.2273.

**N,N-dibenzyl-1,1-dimethoxyoctan-2-amine (3c):** The product was prepared by above general procedure and the same chemicals except employing dibenzylation (1a) (98 mg, 0.5 mmol), and octanal (2c) (96 mg, 0.75 mmol). After 24 h, the crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3c as a light yellowish oil (0.129 g, 70% yield); $R_f = 0.77$ (hexane: ethyl acetate = 3:1); $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.37-7.35 (m, 4H), 7.30-7.26 (m, 4H), 7.23-7.18 (m, 2H), 4.37 (d, $J = 5.03$ Hz, 1H), 3.73 (dd, $J = 50.95$, 13.56 Hz, 4H), 3.35 (s, 3H), 3.32 (s, 3H), 2.73-2.68 (m, 1H), 1.59-1.36 (m, 3H), 1.25-1.17 (m, 5H), 1.15-1.08 (m, 2H), 0.87 (t, $J = 7.07$ Hz, 3H) ppm; $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 140.8 (C x 2), 129.0 (CH x 4), 128.0 (CH x 4), 126.6 (CH x 2), 107.5, 57.4, 54.8, 54.5 (CH$_2$ x 2), 54.3, 31.9, 29.4, 26.9, 26.5, 22.7, 14.1
ppm; **FTIR (neat):** \( \nu = 3015, 1215, 754, 700, 494 \text{ cm}^{-1} \); **HRMS (ESI, m/z):** calcd for C\textsubscript{24}H\textsubscript{36}NO\textsubscript{2}\textsuperscript{+} [M+H]\textsuperscript{+} 370.2746, found: 370.2743.

**N,N-dibenzyl-1,1-dimethoxypent-4-en-2-amine (3d):** The product was prepared by above general procedure and the same chemicals except employing dibenzylamine (1a) (98 mg, 0.5 mmol), and pent-4-enal (2d) (63 mg, 0.75 mmol). After 24 h, The crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3d as a light yellowish oil (0.102 g, 63% yield); \( R_f = 0.70 \) (hexane: ethyl acetate = 3:1); **\(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz)** \( \delta 7.38-7.37 \text{ (m, 4H)}, 7.30-7.26 \text{ (m, 4H)}, 7.21-7.18 \text{ (m, 2H)}, 5.89-5.79 \text{ (m, 1H)}, 5.06 \text{ (d, } J = 17.14 \text{ Hz, 1H)}, 5.01 \text{ (d, } J = 10.10 \text{ Hz, 1H)}, 4.38 \text{ (d, } J = 4.93 \text{ Hz, 1H)}, 3.75 \text{ (dd, } J = 25.44, 13.56 \text{ Hz, 4H)}, 3.35 \text{ (s, 3H)}, 3.28 \text{ (s, 3H)}, 2.89-2.84 \text{ (m, 1H)}, 2.44-2.26 \text{ (m, 2H) ppm; **\(^13\)C NMR (CDCl\textsubscript{3}, 100 MHz)** \( \delta 140.5 \text{ (C x 2)}, 137.8, 129.0 \text{ (CH x 4)}, 128.0 \text{ (CH x 4)}, 126.7 \text{ (CH x 2)}, 115.5, 107.0, 57.8, 54.7, 54.5, 54.4 \text{ (CH}\textsubscript{2} \text{ x 2)}, 31.2 \text{ ppm; **FTIR (neat):**} \( \nu = 3015, 1454, 1215, 754, 700, 494 \text{ cm}^{-1} \); **HRMS (ESI, m/z):** calcd for C\textsubscript{21}H\textsubscript{28}NO\textsubscript{2}\textsuperscript{+} [M+H]\textsuperscript{+} 326.2120, found: 326.2114.

**\(Z\)-N,N-dibenzyl-1,1-dimethoxyhept-4-en-2-amine (3e):** The product was prepared by above general procedure and the same chemicals except employing dibenzylamine (1a) (98 mg, 0.5 mmol), and (Z)-hept-4-enal (2e) (84 mg, 0.75 mmol). After 48 h, The crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3e as a light yellowish oil (0.115 g, 65% yield); \( R_f = 0.72 \) (hexane: ethyl acetate = 3:1); **\(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz)** \( \delta 7.38-7.36 \text{ (m, 4H)}, 7.30-7.26 \text{ (m, 4H)}, 7.24-7.18 \text{ (m, 2H)}, 5.44-5.36 \text{ (m, 2H)}, 4.38 \text{ (d, } J = 4.73
Hz, 1H), 3.79-3.71 (m, 4H), 3.36 (s, 3H), 3.28 (s, 3H), 2.86-2.81 (m, 1H), 2.44-2.37 (m, 1H), 2.28-2.20 (m, 1H), 2.08-2.01 (m, 2H), 0.93 (t, J = 7.53 Hz, 3H) ppm; $^{13}$C NMR (CDCl$_3$, 100 MHz) δ 140.6 (C x 2), 131.9, 129.0 (CH x 4), 128.0 (CH x 4), 127.6, 126.7 (CH x 2), 107.3, 58.0, 54.8, 54.6, 54.5 (CH$_2$ x 2), 24.2, 20.7, 14.2 ppm; FTIR (neat): v = 3017, 1454, 1215, 1072, 756, 699, 498 cm$^{-1}$; HRMS (ESI, m/z): calcd for C$_{23}$H$_{32}$NO$_2^+$ [M+H]$^+$ 354.2433, found: 354.2443.

$N,N$-dibenzyl-5-(tert-butyldiphenylsilyloxy)-1,1-dimethoxypentan-2-amine (3f): The product was prepared by above general procedure and the same chemicals except employing dibenzylamine (1a) (98 mg, 0.5 mmol), and 5-(tert-butyldiphenylsilyloxy)pentanal (2f) (0.255 g, 0.75 mmol). After 48 h, The crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3f as a light yellowish oil (0.230 g, 79% yield); $R_f$= 0.72 (hexane: ethyl acetate = 3:1); $^1$H NMR (CDCl$_3$, 400 MHz) δ 7.66-7.64 (m, 4H), 7.43-7.33 (m, 10H), 7.28-7.17 (m, 6H), 4.38 (d, J = 5.42 Hz, 1H), 3.72 (dd, J = 40.61, 13.45 Hz, 4H), 3.61-3.45 (m, 2H), 3.33 (s, 3H), 3.32 (s, 3H), 2.73-2.68 (m, 1H), 1.86-1.76 (m, 1H), 1.68-1.40 (m, 3H), 1.04 (s, 9H) ppm; $^{13}$C NMR (CDCl$_3$, 100 MHz) δ 140.6 (C x 2), 135.6 (CH x 4), 134.2 (C x 2), 129.4 (CH x 2), 129.1 (CH x 4), 128.0 (CH x 4), 127.5 (CH x 4), 126.7 (CH x 2), 107.4, 63.9, 57.4, 54.7, 54.5 (CH$_2$ x 2), 54.2, 30.3, 26.9 (CH$_3$ x 3), 22.8, 19.2 ppm; FTIR (neat): v = 3017, 1215, 1111, 758, 494 cm$^{-1}$; HRMS (ESI, m/z): calcd for C$_{37}$H$_{48}$NO$_3$Si$^+$ [M+H]$^+$ 582.3403, found: 582.3401.
\textbf{N,N-dibenzyl-1,1-dimethoxy-3-phenylpropan-2-amine (3g):} The product was prepared by above general procedure and the same chemicals except employing dibenzylamine (1a) (98 mg, 0.5 mmol), and 3-phenylpropanal (2g) (0.100 g, 0.75 mmol). After 48 h, The crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3g as a light yellowish oil (0.129 mg, 69% yield); $R_f$ = 0.72 (hexane: ethyl acetate = 3:1); $^1\text{H NMR (CDCl}_3, 400 \text{ MHz})$ $\delta$ 7.27-7.01 (m, 15H), 4.42 (d, $J = 4.38$ Hz, 1H), 3.74 (s, 4H), 3.37 (s, 3H), 3.30 (s, 3H), 3.08-3.03 (m, 1H), 2.88-2.86 (m, 2H) ppm; $^{13}\text{C NMR (CDCl}_3, 100 \text{ MHz})$ $\delta$ 140.8, 140.3 (C x 2), 129.6 (CH x 2), 128.7 (CH x 4), 128.0 (CH x 6), 126.5 (CH x 2), 125.3, 107.2, 59.6, 54.9, 54.8, 54.4 (CH$_2$ x 2), 32.6 ppm; FTIR (neat): $\nu$ = 3019, 1495, 1454, 1215, 1072, 756, 698, 496 cm$^{-1}$; HRMS (ESI, m/z): calcd for C$_{25}$H$_{30}$NO$_2^+$ [M+H]$^+$ 376.2277, found: 376.2278.

\begin{center}
\includegraphics[width=0.2\textwidth]{3g.png}
\end{center}

\textbf{Methyl 4-(dibenzylamino)-5,5-dimethoxypentanoate (3h):} The product was prepared by above general procedure and the same chemicals except employing dibenzylamine (1a) (98 mg, 0.5 mmol), and methyl 5-oxopentanoate (2h) (97 mg, 0.75 mmol). After 48 h, The crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3h as a light yellowish oil (0.124 g, 67% yield); $R_f$ = 0.54 (hexane: ethyl acetate = 3:1); $^1\text{H NMR (CDCl}_3, 400 \text{ MHz})$ $\delta$ 7.33-7.27 (m, 8H), 7.25-7.19 (m, 2H), 4.43 (d, $J = 5.53$ Hz, 1H), 3.72 (dd, $J = 63.86$, 13.41 Hz, 4H), 3.55 (s, 3H), 3.363-3.359 (m, 6H), 2.78-2.73 (m, 1H), 2.53-2.26 (m, 2H), 1.86-1.70 (m, 2H) ppm; $^{13}\text{C NMR (CDCl}_3, 100 \text{ MHz})$ $\delta$ 174.2, 140.2 (C x 2), 129.1 (CH x 4), 128.1 (CH x 4), 126.8 (CH x 2), 106.7, 56.6, 54.8, 54.2 (CH$_2$ x 2), 53.6, 51.3, 31.1, 21.9 ppm; FTIR (neat): $\nu$ = 3017, 1454, 1215, 758, 494 cm$^{-1}$; HRMS (ESI, m/z): calcd for C$_{22}$H$_{30}$NO$_4^+$ [M+H]$^+$ 372.2175, found: 372.2178.
**N-allyl-N-benzyl-1,1-dimethoxybutan-2-amine (3i):** The product was prepared by above general procedure and the same chemicals except employing N-benzylprop-2-en-1-amine (1b) (74 mg, 0.5 mmol), and butyraldehyde (2a) (55 mg, 0.75 mmol). After 24 h, the crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3i as a light yellowish oil (92 mg, 69% yield); \( R_f = 0.72 \) (hexane: ethyl acetate = 3:1); \(^1H\) NMR (CDCl\(_3, 400\) MHz) \( \delta \) 7.36-7.34 (m, 2H), 7.30-7.26 (m, 2H), 7.24-7.18 (m, 1H), 5.85-5.75 (m, 1H), 5.15 (d, \( J = 17.21 \) Hz, 1H), 5.05 (d, \( J = 10.10 \) Hz, 1H), 4.31 (d, \( J = 5.35 \) Hz, 1H), 3.75 (dd, \( J = 14.03 \) Hz, 2H), 3.36 (s, 6H), 3.28-3.15 (m, 2H), 2.72-2.67 (m, 1H), 1.57-1.41 (m, 2H), 0.96 (t, \( J = 7.41 \) Hz, 3H) ppm; \(^{13}C\) NMR (CDCl\(_3, 100\) MHz) \( \delta \) 141.1, 138.1, 128.0 (CH x 2), 126.5, 116.1, 107.3, 106.5, 756, 494 cm\(^{-1}\); FTIR (neat): \( \nu = 3017, 1452, 1215, 1065, 756, 494 \) cm\(^{-1}\); HRMS (ESI, m/z): calcd for C\(_{16}\)H\(_{26}\)N\(_2\)O\(_2\)\(^+\) [M+H]\(^+\) 264.1964, found: 264.1965.

**N,N-dibenzyl-1,1-dimethoxyhexan-2-amine (3j):** The product was prepared by above general procedure and the same chemicals except employing N,N-dibenzylhex-1-en-1-amine (1c) (0.139 g, 0.5 mmol). After 20 h, the crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3j as a light yellowish oil (0.129 g, 76% yield); \( R_f = 0.70 \) (hexane: ethyl acetate = 3:1); \(^1H\) NMR (CDCl\(_3, 400\) MHz) \( \delta \) 7.37-7.35 (m, 4H), 7.30-7.27 (m, 4H), 7.23-7.18 (m, 2H), 4.37 (d, \( J = 5.01 \) Hz, 1H), 3.74 (dd, \( J = 50.64 \), 13.59 Hz, 4H), 3.35 (s, 3H), 3.31 (s, 3H), 2.73-2.69 (m, 1H), 1.59-1.39 (m, 3H), 1.26-1.10 (m, 3H), 0.85 (t, \( J = 7.31 \) Hz, 3H) ppm; \(^{13}C\) NMR (CDCl\(_3, 100\) MHz) \( \delta \) 140.8 (C x 2), 129.0 (CH...
x 4), 128.0 (CH x 4), 126.6 (CH x 2), 107.5, 57.4, 54.8, 54.5 (CH₂ x 2), 54.3, 29.2, 26.2, 22.7, 14.1 ppm; FTIR (neat): ν = 3015, 1452, 1215, 758, 494 cm⁻¹; HRMS (ESI, m/z): calcd for C₂₂H₃₂NO₂⁺ [M+H]⁺ 342.2433, found: 342.2436.

IV. Deuterium Labeling Experiment

$$\text{Bn}_2\text{NH} + \text{H}$$

1.5 equiv.

Cul (0.4 equiv.)

$${}^{t}\text{BuOOH (1.1 equiv.)}$$

CD₃OD/MeCN (1:4)

40 °C, 24 h

36% D atom

80% yield

(1)

Cul (0.4 equiv.)

$${}^{t}\text{BuOOBz (1.1 equiv.)}$$

CD₃OD/MeCN (1:4)

40 °C, 40 h

57% D atom

80% yield

(2)

H

CD₃OD/MeCN (1:4)

40 °C, 24 h

31% D atom

70% yield

(3)

Typical procedure for deuterium labeling experiment (eq. 2): tert-butyl benzoylperoxide (0.107 g, 0.55 mmol) was added to a mixture of Cul (38 mg, 0.2 mmol), dibenzylamine 1a (98 mg, 0.5 mmol), and butyraldehyde 2a (54 mg, 0.75 mmol) in CD₃OD (0.5 mL)/acetonitrile (2.0 mL) at room temperature. The mixture was stirred at 40 °C for 40 h. The resulting reaction mixture was mixed with a small amount of silica gel and concentrated. The crude product was purified by flash column chromatography (silica gel; ethyl acetate or diethyl ether/hexane = 1:100) to afford the desired product 3k as a light yellowish oil (0.144 g, 80% yield).
$N,N$-dibenzyl-1,1-dimethoxy-D6-butan-D1-2-amine (3k): $R_f = 0.70$ (hexane: ethyl acetate = 3:1); $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.38-7.36 (m, 4H), 7.30-7.26 (m, 4H), 7.23-7.18 (m, 2H), 4.38-4.37 (m, 1H), 3.74 (dd, $J = 49.61$, 13.60 Hz, 4H), 2.64-2.60 (m, 0.43H), 1.59-1.44 (m, 2H), 0.92 (t, $J = 7.43$ Hz, 3H) ppm; $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 140.7 (C x 2), 129.0 (CH x 4), 128.0 (CH x 4), 126.6 (CH x 2), 107.2, 59.4, 54.5 (CH$_2$ x 2), 19.54, 19.43, 12.03, 11.99 ppm; FTIR (neat): $\nu = 3015$, 1215, 754, 700, 494 cm$^{-1}$; HRMS (ESI, m/z): calcd for C$_{20}$H$_{33}$D$_5$NO$_4$ $^{+}$ [M+H]$^+$ 361.3115, found: 361.3109.
Reference:


