Supporting Information

Fe-Catalyzed Oxidative C-H/N-H Coupling between Aldehydes and Simple Amides

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**General information**

All reactions were isolated from oxygen by a nitrogen atmosphere. All glassware was oven dried at 110 °C for hours and cooled down under vacuum. DCE was purified by distillation with CaH₂. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp.60-90 °C). Gas chromatographic analyses were preformed on Varian GC 2000 gas chromatography instrument with a FID detector and dibenzo[b,d]furan was added as internal standard. GC-MS spectra were recorded on a Varian GC-MS 3900-2100T. 

**1H and **13C** NMR data were recorded with Varian Mercury (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. All chemical shifts are reported relative to tetramethylsilane and d-solvent peaks (77.00 ppm, chloroform), respectively.**
General procedure for the oxidative coupling of amides with aldehydes

Amide (0.5 mmol) was added to a Schlenk tube equipped with a magnetic stir bar. Then, FeBr$_2$ (5.4 mg, 0.025 mmol) was added to the Schlenk tube in an argon-filled glove box. DCE (2.0 mL) was then injected into the tube by syringe. After stirring for 5 min, aldehyde (1.50 mmol) and TBHP (128.7 mg, 1.0 mmol, a 70% aqueous solution) were consecutively injected into the reaction tube. The reaction was then heated to 60 °C and stirred for 16 h. Upon completion, the reaction was quenched with water and extracted with ethyl ether (3 × 10 mL). The organic layers were then combined. The pure product was obtained by flash column chromatography on silica gel (petroleum/ethyl acetate= 10:1).

Procedure for eq.3

1a (74.6 mg, 0.5 mmol) was added to a Schlenk tube equipped with a magnetic stir bar. Then, FeBr$_2$ (5.4 mg, 0.025 mmol) was added to the Schlenk tube in an argon-filled glove box. DCE (2.0 mL) was then injected into the tube by syringe. After stirring for 5 min, 2a (159.2 mg, 1.5 mmol) and TBHP (128.7 mg, 1.0 mmol, a 70% aqueous solution) were consecutively injected into the reaction tube. After stirring for 5 min, TEMPO (156.1 mg, 1.0 mmol) was added to the reaction tube in N$_2$ atmosphere. The reaction was then heated to 60 °C and stirred for 16 h. Upon completion, the reaction was quenched with water and extracted with ethyl ether (3 × 10 mL). The organic layers were then combined. The pure product was obtained by flash column chromatography on silica gel (petroleum/ethyl acetate= 200:1). The yield of the isolated product was 78% based on TEMPO.
**N-Acetyl-N-benzylbenzamide (3aa).** Compound 3aa was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 75%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.57 – 7.54 (m, 3H), 7.45 – 7.42 (m, 2H), 7.28 – 7.24 (m, 5H), 5.00 (s, 2H), 2.16 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 174.3, 173.2, 137.2, 135.7, 132.4, 128.7, 128.5, 128.3, 127.7, 127.4, 49.2, 26.4 ppm. HRMS (ESI) calcd for C$_{16}$H$_{15}$NO$_2$ [M+H]$^+$: 254.1181; found: 254.1172.

**N-Acetyl-N-benzyl-4-methylbenzamide (3ab).** Compound 3ab was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and 4-methylbenzaldehyde (180.0 mg, 1.5 mmol). Isolated yield = 64%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.47 (d, $J$ = 8.1, 2H), 7.31 – 7.18 (m, 6H), 5.00 (s, 2H), 2.39 (s, 3H), 2.12 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 174.3, 173.1, 143.4, 137.3, 132.8, 129.4, 128.6, 128.4, 127.8, 127.3, 49.3, 26.3, 21.6 ppm. HRMS (APCI) calcd for C$_{17}$H$_{17}$NO$_2$ [M$^+$]: 267.1259 found: 267.1259.

**N-Acetyl-N-benzyl-3-methylbenzamide (3ac).** Compound 3ac was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and 3-methylbenzaldehyde (180.0 mg, 1.5 mmol). Isolated yield = 73%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.36 – 7.22 (m, 9H), 5.00 (s, 2H), 2.35 (s, 3H), 2.12 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 174.6, 173.3, 138.8, 137.4, 135.7, 133.3, 129.0, 128.6, 128.5, 127.8, 127.4, 125.5, 49.3, 26.4, 21.3 ppm. HRMS (APCI) calcd for C$_{17}$H$_{17}$NO$_2$ [M$^+$]:
N-Acetyl-N-benzyl-4-bromobenzamide (3ad). Compound 3ad was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and 4-bromobenzaldehyde (277.5 mg, 1.5 mmol). Isolated yield = 80%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.55 (d, $J$=8.5, 2H), 7.41 (d, $J$=8.4, 2H), 7.31 – 7.17 (m, 5H), 4.98 (s, 2H), 2.19 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.3, 173.0, 136.9, 134.5, 132.0, 129.7, 128.6, 127.5, 127.2, 49.2, 26.2 ppm. HRMS (APCI) calcd for C$_{16}$H$_{14}$BrNO$_2$ [M]$^+$: 331.0211; found: 331.0213.

N-Acetyl-N-benzyl-2-bromobenzamide (3ae). Compound 3ae was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and 2-bromobenzaldehyde (277.5 mg, 1.5 mmol). Isolated yield = 73%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.59 – 7.57 (m, 1H), 7.28 – 7.22 (m, 5H), 7.08 – 7.03 (m, 3H), 4.89 (s, 2H), 2.43 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.0, 171.5, 137.9, 136.9, 132.9, 131.3, 128.5, 128.5(1), 127.5, 127.4, 127.3, 118.7, 48.3, 26.5 ppm. HRMS (APCI) calcd for C$_{16}$H$_{14}$BrNO$_2$ [M]$^+$: 331.0209; found: 331.0208.

N-Acetyl-N-benzyl-4-chlorobenzamide (3af). Compound 3af was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and 4-chlorobenzaldehyde (210.8 mg, 1.5 mmol). Isolated yield = 61%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.49 (d, $J$= 8.5 Hz, 2H), 7.39 (d, $J$ = 8.5 Hz, 2H), 7.33 – 7.14 (m, 5H), 4.98 (s, 2H), 2.19 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.2,
N-Acetyl-N-benzyl-2-chlorobenzamide (3ag). Compound 3ag was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and 2-chlorobenzaldehyde (210.8 mg, 1.5 mmol). Isolated yield = 43%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.41 –7.31 (m, 2H), 7.27 – 7.21 (m, 4H), 7.11 – 7.07 (m, 3H), 4.90 (s, 2H), 2.41 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.0, 170.8, 136.9, 135.9, 131.3, 130.1, 129.8, 128.5, 127.4, 127.2, 127.0, 48.3, 26.4 ppm. HRMS (APCI) calcd for C$_{16}$H$_{14}$ClNO$_2$ [M]$^+$: 287.0715; found: 287.0713.

N-Acetyl-N-benzyl-4-iodobenzamide (3ah). Compound 3ah was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and 4-iodobenzaldehyde (348.0 mg, 1.5 mmol). Isolated yield = 50%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.77 (d, $J$ = 8.5 Hz, 2H), 7.33 – 7.15 (m, 7H), 4.97 (s, 2H), 2.19 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.5, 173.0, 138.0, 136.9, 135.0, 129.6, 128.6, 127.5, 99.6, 49.2, 26.3 ppm. HRMS (APCI) calcd for C$_{16}$H$_{14}$INO$_2$ [M]$^+$: 379.0070; found: 379.0069.

N-Acetyl-N-benzyl-4-fluorobenzamide (3ai). Compound 3ai was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and 4-fluorobenzaldehyde (186.2 mg, 1.5 mmol). Isolated yield = 67%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.60 – 7.56 (m, 2H), 7.30 – 7.20 (m,
$^1$H NMR (400 MHz, CDCl$_3$) $\delta =$ 7.10 (dd, $J = 11.9$, 5.1 Hz, 2H), 4.99 (s, 2H), 2.18 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta =$ 173.2, 173.0, 166.3, 163.8, 137.0, 131.7(8), 131.7(5), 131.0, 130.9, 128.6, 127.6, 127.5, 116.1, 115.9, 49.3, 26.2 ppm. HRMS (APCI) calcd for C$_{16}$H$_{14}$FNO$_2$ [M]$^+$: 271.1014; found: 271.1013.

$N$-Acetyl-N-benzyl-2-fluorobenzamide (3aj). Compound 3aj was prepared following the general procedure starting from $N$-benzylacetamide (74.6 mg, 0.5 mmol) and 2-fluorobenzaldehyde (186.2 mg, 1.5 mmol). Isolated yield = 46%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta =$ 7.47 – 7.45 (m, 1H), 7.39 – 7.35 (m, 1H), 7.29 – 7.09 (m, 7H), 4.98 (s, 2H), 2.34 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta =$ 173.1, 169.2, 159.7, 157.2, 133.0, 129.7, 129.6(9), 128.5, 127.4, 127.1, 124.6(3), 124.6(0), 116.0, 115.8, 48.6, 25.8 ppm. HRMS (APCI) calcd for C$_{16}$H$_{14}$FNO$_2$ [M]$^+$: 271.1010; found: 271.1009.

$N$-Acetyl-N-benzyl-4-(trifluoromethyl)benzamide (3ak). Compound 3ak was prepared following the general procedure starting from $N$-benzylacetamide (74.6 mg, 0.5 mmol) and 4-(trifluoromethyl)benzaldehyde (261.2 mg, 1.5 mmol). Isolated yield = 52%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta =$ 7.61-7.52 (m, 4H), 7.22 – 7.09 (m, 5H), 4.91 (s, 2H), 2.17 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta =$ 173.0, 172.8, 139.0, 136.7, 128.6, 128.2, 127.5, 127.2, 125.6, 49.0, 26.2. ppm. HRMS (APCI) calcd for C$_{17}$H$_{14}$F$_3$NO$_2$ [M]$^+$: 321.0982; found: 321.0982.

$N$-Acetyl-N-benzyl-4-methoxybenzamide (3al). Compound 3al was prepared following the general procedure starting from $N$-benzylacetamide (74.6 mg, 0.5 mmol) and 4-methoxybenzaldehyde (204.2 mg,
1.5 mmol). Isolated yield = 26%. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta = 7.59 - 7.57\) (m, 2H), 7.28 - 7.25 (m, 5H), 6.91 (d, \(J = 8.8\) Hz, 2H), 5.00 (s, 2H), 3.86 (s, 3H), 2.11 (s, 3H) ppm; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta = 173.8, 173.0, 163.3, 137.4, 131.1, 128.5, 127.9, 127.7, 127.4, 114.1, 55.5, 49.4, 26.1\) ppm. HRMS (APCI) calcd for C\(_{17}\)H\(_{17}\)NO\(_3\) [M\(^+\)]: 283.1207; found: 283.1208.

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\begin{array}{c}
\text{N-Acetyl-N-benzylhexanamide (3am). Compound 3am was prepared following the general procedure starting from N-benzylacetamide (74.6 mg, 0.5 mmol) and hexanaldehyde (150.2 mg, 1.5 mmol). Isolated yield = 47%. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta = 7.37 - 7.28\) (m, 3H), 7.16 (d, \(J = 4.0\) Hz, 2H), 4.99 (s, 2H), 2.69 (t, \(J = 7.4\) Hz, 2H), 2.46 (s, 3H), 1.67 - 1.63 (m, 3H), 1.31 - 1.28 (m, 5H), 0.89 (t, \(J = 8.0\) Hz, 3H) ppm; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta = 176.5, 173.6, 137.1, 128.8, 127.4, 126.1, 47.2, 37.8, 31.2, 26.8, 24.5, 22.4, 13.9\) ppm. HRMS (APCI) calcd for C\(_{15}\)H\(_{21}\)NO\(_2\) [M\(^+\)]: 176.0708; found: 176.0712.
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\begin{array}{c}
\text{N-Acetyl-N-phenylbenzamide (3ba). Compound 3ba was prepared following the general procedure starting from N-phenylacetamide (67.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 75%. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta = 7.64\) (d, \(J = 8.0\) Hz, 2H), 7.43 - 7.20 (m, 6H), 7.18 (d, \(J = 8.0\) Hz, 2H) 2.46 (s, 3H) ppm; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta = 173.5, 172.8, 139.1, 134.8, 132.1, 129.4, 129.2, 128.6, 128.3, 128.1, 25.7\) ppm. HRMS (ESI) calcd for C\(_{15}\)H\(_{13}\)NO\(_2\) [M+H\(^+\)]: 240.1025; found: 240.1018.
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\begin{array}{c}
\text{N-Acetyl-N-(o-tolyl)benzamide (3ca). Compound 3ca was prepared following the general procedure}
\end{array}
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starting from N-(o-tolyl)acetamide (74.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 87%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.64 (d, $J$ = 8.0 Hz, 2H), 7.41 – 7.11 (m, 7H), 2.36 (s, 3H), 2.30 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.4, 172.5, 138.1, 135.7, 135.0, 131.7, 131.2, 129.1, 128.7, 128.4, 128.0, 127.0, 25.4, 17.9 ppm. HRMS (APCI) calcd for C$_{16}$H$_{15}$NO$_2$ [M]$^+$: 253.1106; found: 253.1108.

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\begin{array}{c}
\text{N-Acetyl-N-(p-tolyl)benzamide (3da).} \\
\text{Compound 3da was prepared following the general procedure starting from N-(p-tolyl)acetamide (74.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 77%.} \\
\text{$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.66 (d, $J$ = 8.0 Hz, 2H), 7.41 – 7.11 (m, 7H), 2.36 (s, 3H), 2.30 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.7, 173.0, 138.1, 136.5, 135.0, 132.0, 130.1, 129.2, 128.2(7), 128.2(6), 25.6, 21.1 ppm. HRMS (APCI) calcd for C$_{16}$H$_{15}$NO$_2$ [M]$^+$: 253.1105; found: 253.1108.} \\
\end{array}
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\begin{array}{c}
\text{N-Acetyl-N-(4-methoxyphenyl)benzamide (3ea).} \\
\text{Compound 3ea was prepared following the general procedure starting from N-(4-methoxyphenyl)acetamide (82.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 68%.} \\
\text{$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.60 – 7.59 (m, 2H), 7.38 – 7.28 (m, 3H), 7.09 – 7.05 (m, 2H), 6.84 – 6.82 (m, 2H), 3.73 (s, 3H), 2.39 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.6, 172.8, 158.9, 134.8, 131.8, 131.5, 129.4, 128.9, 128.1, 114.5, 55.2, 25.4 ppm. HRMS (APCI) calcd for C$_{16}$H$_{15}$NO$_3$ [M]$^+$: 269.1051; found: 269.1052.} \\
\end{array}
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**N-Acetyl-N-mesitylbenzamide (3fa).** Compound 3fa was prepared following the general procedure starting from N-mesitylacetamide (88.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 99%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.65 (d, $J$ = 8.0 Hz, 2H), 7.48 – 7.37 (m, 3H), 6.96 (s, 2H), 2.31 – 2.25 (m, 12H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 173.7, 172.1, 138.8, 135.7, 135.6, 134.9, 131.7, 129.8, 128.3, 128.1, 25.3, 21.1, 18.3 ppm. HRMS (APCI) calcd for C$_{18}$H$_{19}$NO$_2$ [M]$^+$: 281.1412; found: 281.1416.

**N-Acetyl-N-methylbenzamide (3ga).** Compound 3ga was prepared following the general procedure starting from N-methylacetamide (36.5 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 71%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.64 – 7.47 (m, 5H), 3.22 (s, 3H), 2.34 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 174.2, 173.6, 135.2, 132.4, 128.8, 128.4, 34.4, 25.9 ppm. HRMS (APCI) calcd for C$_{10}$H$_{11}$NO$_2$ [M]$^+$: 177.0788; found: 177.0790.

**N-Acetyl-N-phenethylbenzamide (3ha).** Compound 3ha was prepared following the general procedure starting from N-phenethylacetamide (81.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 78%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.51 – 7.37 (m, 5H), 7.25 – 7.18 (m, 3H), 7.09 (d, $J$ = 8.0 Hz, 2H), 4.02 – 4.00 (m, 2H), 2.93 – 2.88 (m, 2H), 2.14 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$)
\[ \delta = 174.1, 173.1, 138.0, 135.4, 128.9, 128.5, 128.3, 128.0, 126.3, 47.7, 34.9, 26.1 \text{ ppm.} \]

HRMS (APCI) calcd for C_{17}H_{17}NO_{2} [M]^+ : 267.1264; found: 267.1264.

\[ \text{N-Acetyl-N-butylbenzamide (3ia).} \]

Compound 3ia was prepared following the general procedure starting from N-butylacetamide (65.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 81%. \[^1\text{H} \text{NMR (400 MHz, CDCl}_3\text{)} \delta = 7.62 - 7.53 (m, 3H), 7.48 - 7.44 (m, 2H), 3.77 - 3.74 (m, 2H), 2.15 (s, 3H), 1.58 - 1.54 (m, 2H), 1.27 - 1.25 (m, 2H), 0.85 (t, } J = 7.3 \text{ Hz, 3H) ppm; } \[^13\text{C} \text{NMR (100 MHz, CDCl}_3\text{)} \delta = 174.6, 173.2, 135.8, 132.4, 128.8, 128.4, 46.2, 31.1, 26.2, 20.1, 13.6 \text{ ppm.} \]

HRMS (APCI) calcd for C_{13}H_{17}NO_{2} [M]^+ : 219.1263; found: 219.1264.

\[ \text{1-Benzoylazepan-2-one (3ja).} \]

Compound 3ja was prepared following the general procedure starting from azepan-2-one (56.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 67%. \[^1\text{H} \text{NMR (400 MHz, CDCl}_3\text{)} \delta = 7.56 - 7.53 (m, 2H), 7.46 - 7.37 (m, 3H), 4.00 - 3.97 (m, 2H), 2.71 - 2.68 (m, 2H), 1.85 - 1.83 (m, 6H) \text{ ppm; } \[^13\text{C} \text{NMR (100 MHz, CDCl}_3\text{)} \delta = 177.5, 174.1, 136.5, 131.3, 128.1, 127.6, 45.1, 38.8, 29.5, 29.1, 23.7 \text{ ppm.} \]

HRMS (APCI) calcd for C_{13}H_{15}NO_{2} [M]^+ : 217.1098; found: 217.1103.
**N-Phenyl-N-propionylbenzamide (3ka).** Compound 3ka was prepared following the general procedure starting from N-phenylpropionamide (74.6 mg, 0.5 mmol) and benzaldehyde (159.2 mg, 1.5 mmol). Isolated yield = 47%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 7.65 – 7.63 (m, 2H), 7.44 – 7.31 (m, 6H), 7.20-7.18 (m, 2H), 2.71 (q, $J$ = 7.4 Hz, 2H), 1.23 (t, $J$ = 7.4 Hz, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 177.4, 173.0, 139.1, 135.1, 131.9, 129.4, 129.0, 128.7, 128.3, 128.1, 31.0, 9.5 ppm. HRMS (APCI) calcd for C$_{16}$H$_{15}$NO$_2$ [M]$^+$: 253.1101; found: 253.1103.

**2,2,6,6-Tetramethylpiperidin-1-yl benzoate (4).** Isolated yield = 78%. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ = 8.10 – 8.08 (m, 2H), 7.59 – 7.57 (m, 1H), 7.50 – 7.46 (m, 2H), 1.97 – 1.46 (m, 6H), 1.29 (s, 6H), 1.14 (s, 6H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ = 166.3, 132.8, 129.6, 129.5, 128.4, 60.3, 39.0, 31.9, 20.8, 16.9 ppm.
Copies of product $^1$H and $^{13}$C NMR
\[ \text{3af} \]
3ai
3al

\[
\begin{align*}
\text{N} & \quad \text{O} \\
\text{Me} & \quad \text{OMe}
\end{align*}
\]

\[
\begin{align*}
\text{1.88} & \quad \text{1.88} \\
\text{5.11} & \quad \text{5.11} \\
\text{2.26} & \quad \text{2.26} \\
\text{2.23} & \quad \text{2.23}
\end{align*}
\]

\[
\begin{align*}
\text{173.010} & \quad \text{173.010} \\
\text{137.396} & \quad \text{137.396} \\
\text{128.496} & \quad \text{128.496} \\
\text{127.886} & \quad \text{127.886} \\
\text{114.099} & \quad \text{114.099} \\
\text{80.033} & \quad \text{80.033} \\
\text{40.448} & \quad \text{40.448} \\
\text{20.119} & \quad \text{20.119}
\end{align*}
\]
3ia

\[
\text{NO}_3
\]

\[
\text{NO}_3
\]