Supporting Information

Copper-catalyzed Oxidative Condensation of α-Oxocarboxylic Acids with Formamides: Synthesis of α-Ketoamides

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General Information

All Reactions were carried out under an atmosphere of nitrogen with the strict exclusion of moisture. The dry DMF were distilled from CaH₂ under nitrogen and stored over molecular sieves under nitrogen. Column chromatography was carried out on silica gel. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker Advance III-400 in solvents as indicate. Chemical shift are reported in ppm from CDCl₃ using TMS as internal standard. IR spectra were recorded on a Bruker Tensor 27 spectrometer and only major peaks are reported in cm⁻¹. HRMS were obtained on a Q-TOF micro spectrometer. Melting points were determined on a microscopic apparatus and were uncorrected.

Starting Materials

N,N-Diethylformamide, N-formylpiperidine, and N-formylmorpholine were purchased from Sigma-Aldrich and TCI. Phenylglyoxylic acid ¹a was purchased from Sigma-Aldrich. Other α-oxocarboxylic acids were prepared from the corresponding methyl ketones according to the reported procedure.¹
General Procedure for the Coupling of Formamides with α-Oxocarboxylic acids

\[ \text{ArOH} + \text{HNN}_R^\text{R}^2 \xrightarrow{\text{catalyst oxidant}} \text{ArO}_N^\text{R}^1 \text{R}^2 \]

A 10 mL oven-dried Schlenk-tube was charged with Cu(OAc)₂ (1.8 mg, 5 mol %). The tube was evacuated and backfilled with nitrogen (three times). α-Oxocarboxylic acids (1, 0.2 mmol, 1.0 equiv) and Di-tert-butyl peroxide (DTBP, 0.4 mmol, 2.0 equiv) in substituted formamides (2 mL) were added by syringe under nitrogen. The tube was then sealed and the mixture was stirred for 3 h at 110 °C. Upon completion of the reaction (monitored by TLC), the mixture was diluted with EtOAc, filtered through a pad of Celite, and the filtrate was washed with water, dried over Na₂SO₄. After the solvent was removed, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/10 to 1/5) to give the corresponding products 3 or 4 in yields listed in Table 2 and Table 3.
Characterization of Products 3

\[
\text{N,N-Dimethyl-2-oxo-2-phenyl-acetamide (3a):}^{2a,2d} \quad \text{A pale yellow oil, } R_f \ 0.3 \\
(\text{EtOAc/petroleum ether } = 1:5); \quad ^1\text{H NMR (400 MHz, CDCl}_3): \delta = 7.94-7.92 (d, J = 7.6 \text{ Hz, 2H}), 7.65-7.61 (t, J = 7.2 \text{ Hz, 1H}), 7.52-7.48 (t, J = 7.6 \text{ Hz, 2H}), 3.11 (s, 3H), 2.95 (s, 3H); ^{13}\text{C NMR (100 MHz, CDCl}_3): \delta = 191.7, 167.0, 134.7, 133.0, 129.6, 129.0, 37.0, 33.9 \text{ ppm.}
\]

\[
\text{N,N-Dimethyl-2-oxo-2-p-tolyl-acetamide (3b):}^{2f} \quad \text{A pale yellow solid, } R_f \ 0.3 \\
(\text{EtOAc/petroleum ether } = 1:5), \text{mp: } 46-48 \ ^\circ\text{C}; \quad ^1\text{H NMR (400 MHz, CDCl}_3): \delta = 7.83-7.81 (d, J = 7.2 \text{ Hz, 2H}), 7.30-7.28 (d, J = 7.6 \text{ Hz, 2H}), 3.10 (s, 3H), 2.94 (s, 3H), 2.42 (s, 3H); ^{13}\text{C NMR (100 MHz, CDCl}_3): \delta = 191.5, 167.2, 145.9, 130.6, 129.7, 37.0, 33.9, 21.9 \text{ ppm; IR (KBr): } \nu_{\max} \ 1651, 1406, 1254, 1144 \text{ cm}^{-1}.
\]

\[
\text{2-(4-Methoxy-phenyl)-N,N-dimethyl-2-oxo-acetamide (3c):}^{2b} \quad \text{A pale yellow solid, } R_f \ 0.1 \\
(\text{EtOAc/petroleum ether } = 1:5); \quad ^1\text{H NMR (400 MHz, CDCl}_3): \delta = 7.91-7.89 (d, J = 8.4 \text{ Hz, 2H}), 6.97-6.95 (d, J = 8.4 \text{ Hz, 2H}), 3.87 (s, 3H), 3.09 (s, 3H), 2.94 (s, 3H); ^{13}\text{C NMR (100 MHz, CDCl}_3): \delta = 190.5, 167.3, 164.8, 132.1, 126.1, 114.3, 55.6, 37.1, 33.9 \text{ ppm.}
\]

\[
\text{2-(4-Chloro-phenyl)-N,N-dimethyl-2-oxo-acetamide (3d):}^{2f} \quad \text{A pale yellow solid, } R_f \ 0.3 \\
(\text{EtOAc/petroleum ether } = 1:5), \text{mp: } 113-115 \ ^\circ\text{C}; \quad ^1\text{H NMR (400 MHz, CDCl}_3): \delta = 7.90-7.88 (d, J = 7.6 \text{ Hz, 2H}), 7.49-7.47 (d, J = 8.0 \text{ Hz, 2H}), 3.11 (s, 3H), 2.95 (s, 3H); ^{13}\text{C NMR (100 MHz, CDCl}_3): \delta = 190.3, 166.4, 141.3, 131.4, 131.0, 129.4, 37.0, 34.1
\]

S4
ppm; IR (KBr): $\nu_{\text{max}}$ 1641, 1406, 1246, 1147 cm$^{-1}$.

2-(4-Bromo-phenyl)-$N,N$-dimethyl-2-oxo-acetamide (3e): A pale yellow solid, $R_f$ 0.3 (EtOAc/petroleum ether = 1:5), mp: 68-70 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 7.82-7.80 (d, $J$ = 8.0 Hz, 2H), 7.66-7.64 (d, $J$ = 7.6 Hz, 2H), 3.11 (s, 3H), 2.95 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 190.5, 166.4, 132.4, 131.8, 131.0, 130.2, 37.0, 34.1 ppm; IR (KBr): $\nu_{\text{max}}$ 1641, 1400, 1246, 1145 cm$^{-1}$; HRMS (ESI) calcd for C$_{10}$H$_{10}$BrNNaO$_2$ [M+Na]$^+$ 277.9787, found 277.9794.

2-(4-Iodo-phenyl)-$N,N$-dimethyl-2-oxo-acetamide (3f): A pale yellow solid, $R_f$ 0.3 (EtOAc/petroleum ether = 1:5), mp: 96-98 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 7.88-7.87 (d, $J$ = 7.6 Hz, 2H), 7.65-7.63 (d, $J$ = 7.6 Hz, 2H), 3.10 (s, 3H), 2.95 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 190.8, 166.4, 138.3, 132.4, 130.8, 103.3, 37.0, 34.1 ppm; IR (KBr): $\nu_{\text{max}}$ 1637, 1396, 1247, 1144 cm$^{-1}$; HRMS (ESI) calcd for C$_{10}$H$_{10}$INNaO$_2$ [M+Na]$^+$ 325.9648, found 325.9658.

2-(4-Fluoro-phenyl)-$N,N$-dimethyl-2-oxo-acetamide (3g):$^{2f}$ A pale yellow solid, $R_f$ 0.3 (EtOAc/petroleum ether = 1:5), mp: 63-65 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 8.00-7.97 (t, $J$ = 6.0 Hz, 2H), 7.20-7.16 (t, $J$ = 8.0 Hz, 2H), 3.12 (s, 3H), 2.97 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 190.0, 166.7 (d, $J_{\text{C,F}}$ = 256.0 Hz), 166.6, 132.5 (d, $J_{\text{C,F}}$ = 9.8 Hz), 129.6, 116.3 (d, $J_{\text{C,F}}$ = 22.1 Hz), 37.1, 34.1 ppm; IR (KBr): $\nu_{\text{max}}$ 1648, 1409, 1239, 1147 cm$^{-1}$.

$N,N$-Dimethyl-2-(4-nitro-phenyl)-2-oxo-acetamide (3h):$^{2f}$ A pale yellow solid, $R_f$ 0.2 (EtOAc/petroleum ether = 1:2), mp: 136-138 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ =
8.36-8.34 (d, J = 8.0 Hz, 2H), 8.16-8.14 (d, J = 8.4 Hz, 2H), 3.15 (s, 3H), 3.00 (s, 3H); 

$^{13}$C NMR (100 MHz, CDCl$_3$): δ = 189.2, 165.6, 151.1, 137.5, 130.8, 124.1, 37.1, 34.3 ppm; IR (KBr): $\nu_{\text{max}}$ 1645, 1451, 1245, 1148 cm$^{-1}$.

![Diagram of N,N-Dimethyl-2-oxo-2-o-tolyl-acetamide (3i)]

$N,N$-Dimethyl-2-oxo-2-o-tolyl-acetamide (3i): A pale yellow solid, R$_f$ 0.25 (EtOAc/petroleum ether = 1:5), mp: 43-45 °C; 
$^1$H NMR (400 MHz, CDCl$_3$): δ = 7.68-7.66 (d, J = 8.0 Hz, 1H), 7.48-7.44 (t, J = 7.6 Hz, 1H), 7.31-7.28 (t, J = 7.2 Hz, 2H), 3.09 (s, 3H), 2.96 (s, 3H), 2.64 (s, 3H); 

$^{13}$C NMR (100 MHz, CDCl$_3$): δ = 193.7, 167.7, 141.4, 133.6, 132.6, 132.5, 131.4, 126.1, 37.0, 34.0, 21.7 ppm; IR (KBr): $\nu_{\text{max}}$ 1638, 1405, 1240, 1153 cm$^{-1}$.

![Diagram of 2-(2-Chloro-phenyl)-$N,N$-dimethyl-2-oxo-acetamide (3j)]

2-(2-Chloro-phenyl)-$N,N$-dimethyl-2-oxo-acetamide (3j): A pale yellow solid, R$_f$ 0.3 (EtOAc/petroleum ether = 1:5), mp: 76-78 °C; 

$^1$H NMR (400 MHz, CDCl$_3$): δ = 7.89-7.87 (d, J = 7.6 Hz, 1H), 7.52-7.48 (t, J = 7.6 Hz, 1H), 7.44-7.38 (m, 2H), 3.07 (s, 6H); 

$^{13}$C NMR (100 MHz, CDCl$_3$): δ = 190.1, 166.9, 134.3, 133.7, 133.4, 132.2, 130.7, 127.3, 37.0, 34.5 ppm; IR (KBr): $\nu_{\text{max}}$ 1644, 1412, 1275, 1151 cm$^{-1}$; HRMS (ESI) calcd for C$_{10}$H$_{10}$ClNNaO$_2$ [M+Na]$^+$ 234.0292, found 234.0304.

![Diagram of 2-(2-Fluoro-phenyl)-$N,N$-dimethyl-2-oxo-acetamide (3k)]

2-(2-Fluoro-phenyl)-$N,N$-dimethyl-2-oxo-acetamide (3k): A pale yellow solid, R$_f$ 0.2 (EtOAc/petroleum ether = 1:5), mp: 53-55 °C; 

$^1$H NMR (400 MHz, CDCl$_3$): δ = 7.99-7.95 (t, J = 7.6 Hz, 1H), 7.63-7.61 (m, 1H), 7.32-7.28 (t, J = 7.6 Hz, 1H), 7.18-7.13 (t, J = 9.6 Hz, 1H), 3.09 (s, 3H), 3.01 (s, 3H); 

$^{13}$C NMR (100 MHz, CDCl$_3$): δ = 188.2, 167.4, 162.6 (d, $J_{C,F} = 256.5$ Hz), 136.5 (d, $J_{C,F} = 9.3$ Hz), 131.0, 124.8 (d, $J_{C,F} = 3.5$ Hz), 122.2, 116.7 (d, $J_{C,F} = 21.6$ Hz), 36.8, 34.1 ppm; IR (KBr): $\nu_{\text{max}}$ 1659, 1409, 1289, 1150 cm$^{-1}$; HRMS (ESI) calcd for C$_{10}$H$_{10}$FNNaO$_2$ [M+Na]$^+$ 218.0588, found 218.0591.
**N,N-Dimethyl-2-naphthalen-2-yl-2-oxo-acetamide (3l):** A pale yellow solid, R_f 0.15 (EtOAc/petroleum ether = 1:5), mp: 112-114 °C; \(^1\)H NMR (400 MHz, CDCl₃): \(\delta = 8.43\) (s, 1H), 8.04-8.02 (d, \(J = 8.8 \) Hz, 1H), 7.98-7.93 (m, 2H), 7.90-7.88 (d, \(J = 8.0 \) Hz, 1H), 7.66-7.62 (t, \(J = 7.2 \) Hz, 1H), 7.59-7.55 (t, \(J = 7.2 \) Hz, 1H), 3.18 (s, 3H), 3.00 (s, 3H); \(^13\)C NMR (100 MHz, CDCl₃): \(\delta = 191.9, 167.1, 136.3, 133.0, 132.4, 130.4, 129.9, 129.4, 129.1, 127.9, 127.1, 123.6, 37.1, 34.1 \) ppm; IR (KBr): \(\nu_{max} 1641, 1458, 1274, 1145 \) cm\(^{-1}\); HRMS (ESI) calcd for C₁₄H₁₃NNaO₂ [M+Na]^+ 250.0838, found 250.0850.

**N,N-Dimethyl-2-naphthalen-1-yl-2-oxo-acetamide (3m):**\(^2\) A pale yellow oil, R_f 0.15 (EtOAc/petroleum ether = 1:5); \(^1\)H NMR (400 MHz, CDCl₃): \(\delta = 9.27-9.24\) (d, \(J = 8.4 \) Hz, 1H), 8.11-8.09 (d, \(J = 8.0 \) Hz, 1H), 8.00-7.98 (d, \(J = 7.2 \) Hz, 1H), 7.92-7.90 (d, \(J = 8.0 \) Hz, 1H), 7.72-7.68 (t, \(J = 7.6 \) Hz, 1H), 7.61-7.57 (t, \(J = 7.6 \) Hz, 1H), 7.56-7.52 (t, \(J = 7.6 \) Hz, 1H), 3.15 (s, 3H), 3.01 (s, 3H); \(^13\)C NMR (100 MHz, CDCl₃): \(\delta = 194.2, 167.6, 135.9, 134.3, 134.0, 130.9, 129.3, 128.7, 128.3, 126.9, 125.7, 124.5, 37.2, 34.1 \) ppm; IR (KBr): \(\nu_{max} 1647, 1404, 1270, 1069 \) cm\(^{-1}\).

**2-Furan-2-yl-N,N-dimethyl-2-oxo-acetamide (3n):**\(^2\) A yellow oil, R_f 0.1 (EtOAc/petroleum ether = 1:5); \(^1\)H NMR (400 MHz, CDCl₃): \(\delta = 7.70\) (s, 1H), 7.36 (d, \(J = 2.8 \) Hz, 1H), 6.60 (d, \(J = 1.6 \) Hz, 1H), 3.07 (s, 3H), 3.03 (s, 3H); \(^13\)C NMR (100 MHz, CDCl₃): \(\delta = 178.5, 165.4, 150.1, 148.7, 122.4, 112.8, 37.2, 34.5 \) ppm; IR (KBr): \(\nu_{max} 1637, 1460, 1259, 1150 \) cm\(^{-1}\).

**N,N-Dimethyl-2-oxo-2-thiophen-2-yl-acetamide (3o):** A pale yellow solid, R_f 0.15 (EtOAc/petroleum ether = 1:5), mp: 57-59 °C; \(^1\)H NMR (400 MHz, CDCl₃): \(\delta =
7.80-7.77 (m, 2H), 7.18-7.16 (t, J = 4.0 Hz, 1H), 3.08 (s, 3H), 3.03 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 183.5, 165.8, 140.3, 136.4, 136.1, 128.6, 37.3, 34.4 ppm; IR (KBr): $\nu_{\text{max}}$ 1648, 1407, 1246, 1142 cm$^{-1}$; HRMS (ESI) calcd for C$_8$H$_9$NNaO$_2$S [M+Na]$^+$ 206.0246, found 206.0253.

$N,N$-dimethyl-4-nitrobenzamide (3h$^+$): A pale yellow solid, $R_f$ 0.2 (EtOAc/petroleum ether = 1:5), $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 8.28-8.27 (d, $J$ = 7.6 Hz, 2H), 7.59-7.57 (d, $J$ = 7.6 Hz, 2H), 3.14 (s, 3H), 2.96 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 169.2, 148.2, 142.5, 128.0, 123.8, 39.3, 35.3 ppm.
Characterization of Products 4

\[
\begin{align*}
\text{N,N-Diethyl-2-oxo-2-phenyl-acetamide (4a):}^{2c} & \quad \text{A pale yellow oil, } R_f 0.3 \\
& \quad \text{(EtOAc/petroleum ether = 1:5); } ^1\text{H NMR (400 MHz, CDCl}_3): \delta = 7.93-7.92 (d, J = 7.6 \text{ Hz, 2H}), 7.64-7.61 (t, J = 7.2 \text{ Hz, 1H}), 7.51-7.48 (t, J = 7.6 \text{ Hz, 2H}), 3.58-3.53 (q, J = 7.2 \text{ Hz, 2H}), 3.25-3.20 (q, J = 6.8 \text{ Hz, 2H}), 1.29-1.26 (t, J = 7.2 \text{ Hz, 3H}), 1.16-1.12 (t, J = 7.2 \text{ Hz, 3H}); \quad ^13\text{C NMR (100 MHz, CDCl}_3): \delta = 191.6, 166.7, 134.5, 133.2, 129.6, 128.9, 42.0, 38.7, 14.1, 12.8 \text{ ppm.}
\end{align*}
\]

\[
\begin{align*}
\text{1-Phenyl-2-piperidin-1-yl-ethane-1,2-dione (4b):}^{2c, 2d} & \quad \text{A pale yellow solid, } R_f 0.4 \\
& \quad \text{(EtOAc/petroleum ether = 1:5), mp: 102-104 °C; } ^1\text{H NMR (400 MHz, CDCl}_3): \delta = 7.95-7.93 (d, J = 7.2 \text{ Hz, 2H}), 7.65-7.61 (t, J = 7.6 \text{ Hz, 1H}), 7.52-7.48 (t, J = 7.2 \text{ Hz, 2H}), 3.70 (s, 2H), 3.29-3.26 (t, J = 5.6 \text{ Hz, 2H}), 1.69-1.68 (m, 4H), 1.55-1.53 (m, 2H); \quad ^13\text{C NMR (100 MHz, CDCl}_3): \delta = 191.1, 165.4, 134.6, 133.2, 129.5, 128.9, 47.0, 42.1, 26.1, 25.4, 24.3 \text{ ppm; IR (KBr): } v_{\text{max}} 1645, 1446, 1216, 1137 \text{ cm}^{-1}.
\end{align*}
\]

\[
\begin{align*}
\text{1-Morpholin-4-yl-2-phenyl-ethane-1,2-dione (4c):}^{2c, 2d} & \quad \text{A pale yellow oil, } R_f 0.2 \\
& \quad \text{(EtOAc/petroleum ether = 1:2); } ^1\text{H NMR (400 MHz, CDCl}_3): \delta = 7.96-7.94 (d, J = 7.6 \text{ Hz, 2H}), 7.68-7.64 (t, J = 7.2 \text{ Hz, 1H}), 7.54-7.50 (t, J = 8.0 \text{ Hz, 2H}), 3.79 (s, 4H), 3.66-3.64 (t, J = 4.8 \text{ Hz, 2H}), 3.39-3.37 (t, J = 4.8 \text{ Hz, 2H}); \quad ^13\text{C NMR (100 MHz, CDCl}_3): \delta = 191.1, 165.4, 134.9, 133.0, 129.7, 129.1, 66.7, 46.2, 41.6 \text{ ppm.}
\end{align*}
\]

\[
\begin{align*}
\text{1-Morpholin-4-yl-2-p-tolyl-ethane-1,2-dione (4d):}^{2d} & \quad \text{A pale yellow solid, } R_f 0.15 \\
& \quad \text{(EtOAc/petroleum ether = 1:5); } ^1\text{H NMR (400 MHz, CDCl}_3): \delta = 7.85-7.83 (d, J = 7.6}
\]

S9
Hz, 2H), 7.32-7.30 (d, J = 8.0 Hz, 2H), 3.78 (s, 4H), 3.65-3.62 (t, J = 4.4 Hz, 2H), 3.37-3.35 (t, J = 4.8 Hz, 2H), 2.43 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 190.9, 165.6, 146.3, 130.6, 129.8, 129.7, 66.7, 66.6, 46.2, 41.5, 21.9 ppm.

1-(4-Chloro-phenyl)-2-morpholin-4-yl-ethane-1,2-dione (4e): A pale yellow solid, R$_f$ 0.2 (EtOAc/petroleum ether = 1:5); $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 7.92-7.90 (d, J = 8.4 Hz, 2H), 7.51-7.49 (d, J = 8.4 Hz, 2H), 3.79 (s, 4H), 3.67-3.65 (t, J = 4.8 Hz, 2H), 3.39-3.37 (t, J = 5.2 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 189.7, 164.9, 141.6, 131.4, 131.0, 129.5, 66.7, 66.6, 46.3, 41.7 ppm.
Investigation of the Reaction Mechanism

When the TEMPO was added to the reaction of DMF with phenylglyoxylic acid 2a under the standard condition, only a trace amount of the desired product 3a was obtained suggesting that free radical intermediate was involved in the reaction (eq 1). In addition, benzaldehyde was used instead of phenylglyoxylic acid under the standard conditions, only a trace amount of α-ketoamide 3a was detected (eq 2). The result indicated that aldehyde was not the intermediates in this reaction.
C^{13}\text{-Isotope Labeling Experiment}

\(N,N\text{-dimethylformamide (carbonyl-}^{13}\text{C, 99\%, cat. No. CLM-503-0)}\) were purchased from Cambridge Isotope Laboratories. The isotope reagent was used without further purification.
The Coupling of Phenylglyoxylic Acid with $^{13}$C-Labeling DMF

![Chemical reaction diagram]

Phenylglyoxylic acid (1, 0.1 mmol, 1.0 equiv) and Di-tert-butyl peroxide (DTBP, 0.2 mmol, 2.0 equiv) in $^{13}$C-labeling DMF (1 mL) were added by syringe under nitrogen. The tube was then sealed and the mixture was stirred for 3 h at 110 °C. Upon completion of the reaction, the mixture was diluted with EtOAc, filtered through a pad of Celite, and the filtrate was washed with water, dried over Na$_2$SO$_4$. After the solvent was removed, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/10 to 1/5) to give the corresponding products 3a in 72% yield.


The HRMS Spectra of 3a
The NMR Spectra of 3a

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Competing Kinetic Isotope Effect (KIE) Experiment

\[
\begin{align*}
\text{Ph} & \quad \text{O} & \quad \text{OH} & \quad + & \quad \text{H} & \quad \text{N} & \quad \text{CH}_3 \\
\text{D} & \quad \text{N} & \quad \text{CD}_3 & \quad \text{standard} & \quad \text{conditions} & \quad \text{Ph} & \quad \text{O} & \quad \text{N} & \quad \text{CH}_3 \\
\text{K}_\text{H}/\text{K}_\text{D} &= 2.5
\end{align*}
\]

Figure. \(^1\)H NMR spectra of the mixture of the product 3a and 3a'.

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References


$^{1}H$ NMR and $^{13}C$ NMR Spectra of the Products

**Bruker**

**NAME**
dah-DNP-2

**FREQUENCY**
8.43

**SPECTRUM**
spect

**PROBE**
5 mm PABB, DQF

**COUPLING**
62 KHz

**SOLOVET**
CDD13

**NS**
16

**DS**
8233.68 Hz

**P<sub>D</sub>**
8.12564 Hz

**AQ**
3.90661397 sec

**DF**
40.850 usec

**DM**
4.30 usec

**TT**
1.68084003 sec

**DD**
1

**CHANNEL 3**

**NM1**
10

**PI1**
12.80 usec

**P11**
3.00 dB

**SW1**
0.00144652 W

**SF**
400.13900014 MHz

**CM**
69

**BB**
20.00 Hz

**LR**
0.00 Hz

**PG**
0

**AC**
1.00

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