Cu\textsuperscript{II}-Catalyzed Decarboxylative Acylation of Acyl C–H of Formamides with α-Oxocarboxylic Acids Leading to α-Ketoamides

Dengke Li,\textsuperscript{a} Min Wang,*\textsuperscript{a} Jie Liu,\textsuperscript{a} Qiong Zhao,\textsuperscript{a} and Lei Wang*\textsuperscript{a,b}

\textsuperscript{a} Department of Chemistry, Huaibei Normal University, Huaibei, Anhui 235000, China
Tel: + 86-561-3802-069  Fax: + 86-561-3090-518
E-mail: leiwang@chnu.edu.cn, wangmin204@chnu.edu.cn

\textsuperscript{b} State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China

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1. General considerations

All the reactions of α-oxocarboxylic acids and formamides were carried out under an air atmosphere. $^1$H NMR and $^{13}$C NMR spectra were measured on a Bruker Avance NMR spectrometer (400 MHz or 100 MHz, respectively) with CDCl$_3$ as solvent and recorded in ppm relative to internal tetramethylsilane standard. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, $J$, are reported in Hertz (Hz). High resolution mass spectroscopy data of the product were collected on a Waters Micromass GCT instrument. Solvents and general chemicals were purchased from commercial suppliers and used without further purification.

2. Starting materials

For this study, all formamides (2a–2h) and α-oxocarboxylic acid (1a) were purchased from commercial sources. Other α-oxocarboxylic acids (1b–1n) can be prepared from oxidation of corresponding methyl ketones with SeO$_2$ according to the reported procedure.$^{[1]}$
3. Optimization of oxidant, additive, solvent and temperature (TS1) 

![Chemical structure diagram](image)

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* Reaction conditions: 1a (0.50 mmol), 2a (5.0 equiv), CuBr₂ (10 mol %), oxidant (2.0 equiv), additive (2.0 equiv), solvent (1.5 mL), air atmosphere, 110 °C, 18 h. † Isolated yields. ‡ H₂O₂ (30% in water). ‡ ‡ TBHP ( tert-butyl hydroperoxide, 70% in water). † † 100 °C. † † † 120 °C.
4. General procedure

Under air atmosphere, a sealable reaction tube with a Teflon-coated screw cap equipped with a magnetic stir bar was charged with 2-oxo-2-phenylacetic acid (1a, 0.50 mmol), N,N-dimethylformamide (2a, DMF, 2.5 mmol), CuBr$_2$ (0.05 mmol), di-tert-butyl peroxide (DTBP, 1.0 mmol), pivalic acid (PivOH, 1.0 mmol), and toluene (1.5 mL). The rubber septum was then replaced by a Teflon-coated screw cap, and the reaction vessel placed in an oil bath at 110 °C for 18 h. After the reaction was completed, it was cooled to room temperature and quenched with water and extracted with ethyl acetate and then dried with Na$_2$SO$_4$. The resulting solution was directly filtered through a pad of silica gel using a sintered glass funnel, and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (eluant: petroleum ether/ethyl acetate) to give the desired product N,N-dimethyl-2-oxo-2-phenylacetamide (3aa).
5. Characterization data for all products

\textbf{N,N-Dimethyl-2-oxo-2-phenylacetamide}

\begin{center}
\includegraphics[width=0.2\textwidth]{chemical_structure}
\end{center}

3aa:\[^{[2]}]\text{Yellow oil.}

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 7.96–7.94 (m, 2H), 7.66–7.62 (m, 1H), 7.53–7.49 (m, 2H), 3.12 (s, 3H), 2.96 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 191.74, 167.04, 134.65, 133.13, 129.63, 128.98, 37.01, 33.99.

\textbf{N,N-Diethyl-2-oxo-2-phenylacetanamide}

\begin{center}
\includegraphics[width=0.2\textwidth]{chemical_structure}
\end{center}

3ab:\[^{[3]}]\text{Yellow oil.}

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 7.95–7.93 (m, 2H), 7.65–7.61 (m, 1H), 7.52–7.49 (m, 2H), 3.56 (q, $J = 7.2$ Hz, 2H), 3.24 (q, $J = 7.2$ Hz, 2H), 1.29 (t, $J = 7.2$ Hz, 3H), 1.15 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 191.55, 166.74, 134.50, 133.32, 129.59, 128.92, 42.10, 38.81, 14.07, 12.80.

\textbf{N,N-Dibutyl-2-oxo-2-phenylacetamide}

\begin{center}
\includegraphics[width=0.2\textwidth]{chemical_structure}
\end{center}

3ac:\[^{[4]}]\text{Yellow oil.}

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 7.94–7.92 (m, 2H), 7.64–7.60 (m, 1H), 7.51–7.48 (m, 2H), 3.49 (t, $J = 8.0$ Hz, 2H), 3.14 (t, $J = 8.0$ Hz, 2H), 1.71–1.63 (m, 2H), 1.57–1.49 (m, 2H), 1.46–1.37 (m, 2H), 1.25–1.14 (m, 2H), 0.99 (t, $J = 8.0$ Hz, 3H), 0.81 (t, $J = 8.0$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 191.55, 167.07, 134.44, 133.38, 129.57, 128.89,
47.42, 44.03, 30.61, 29.43, 20.21, 19.73, 13.80, 13.49.

**1-Phenyl-2-(piperidin-1-yl)ethane-1,2-dione**

![Chemical structure](image)

3ad: Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.96$–$7.94$ (m, 2H), 7.65–7.62 (m, 1H), 7.53–7.49 (m, 2H), 3.71 (s, 2H), 3.29 (t, $J = 5.6$ Hz, 2H), 1.70 (s, 4H), 1.55 (s, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 191.91$, 165.45, 134.59, 133.31, 129.54, 128.97, 47.02, 42.15, 26.19, 25.43, 24.37.

**1-Morpholino-2-phenylethane-1,2-dione**

![Chemical structure](image)

3ae: Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.96$–$7.94$ (m, 2H), 7.66–7.63 (m, 1H), 7.53–7.49 (m, 2H), 3.78 (s, 4H), 3.65–3.63 (m, 2H), 3.38–3.36 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 191.13$, 165.45, 134.89, 133.09, 129.64, 129.07, 66.71, 66.63, 46.25, 41.62.

**N-Methyl-2-oxo-2-phenylacetamide**

![Chemical structure](image)

3af: Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 8.36$–$8.35$ (m, 2H), 7.65–7.61 (m, 1H), 7.51–7.47 (m, 2H), 7.11 (s, 1H), 2.98 (d, $J = 4.8$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 187.65$, 162.42, 134.34, 133.36, 131.20, 128.46, 25.98. IR (KBr, cm$^{-1}$): ($\nu_{C=O}$) 1665, 1595. HRMS (EI) ([M]$^+$) Calcd. for C$_9$H$_9$NO$_2$: 163.0633, Found: 163.0636.
**N-Ethyl-2-oxo-2-phenylacetamide**

![Chemical Structure](image)

### 3ag
Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 8.35$–$8.33$ (m, 2H), 7.64–7.61 (m, 1H), 7.50–7.46 (m, 2H), 7.11 (s, 1H), 3.48–3.41 (m, 2H), 1.26 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 187.92$, 161.69, 134.30, 133.41, 131.18, 128.44, 34.36, 14.45. IR (KBr, cm$^{-1}$): (ν$_{\text{C=O}}$) 1663, 1595. HRMS (EI) ([M]$^+$) Calcd. for C$_{10}$H$_{11}$NO$_2$: 177.0790, Found: 177.0786.

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**N-tert-Butyl-2-oxo-2-phenylacetamide**

![Chemical Structure](image)

### 3ah
Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 8.32$–$8.30$ (m, 2H), 7.63–7.59 (m, 1H), 7.49–7.45 (m, 2H), 6.93 (s, 1H), 1.47 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 188.57$, 161.14, 134.10, 133.43, 131.19, 128.35, 51.65, 28.38. IR (KBr, cm$^{-1}$): (ν$_{\text{C=O}}$) 1667, 1597. HRMS (EI) ([M]$^+$) Calcd. for C$_{12}$H$_{15}$NO$_2$: 205.1103, Found: 205.1099.

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**N,N-Dimethyl-2-oxo-2-p-tolylacetaminde**

![Chemical Structure](image)

### 3ba
Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.85$–$7.83$ (m, 2H), 7.31–7.29 (m, 2H), 3.11 (s, 3H), 2.95 (s, 3H), 2.43 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 191.50$, 167.28, 145.94, 130.69, 129.76, 129.71, 37.03, 33.96, 21.85.
2-(4-tert-Butylphenyl)-N,N-dimethyl-2-oxoacetamide

3ca: Yellow oil.

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta = 7.88\) (d, \(J = 8.4\) Hz, 2H), 7.53–7.51 (m, 2H), 3.12 (s, 3H), 2.96 (s, 3H), 1.34 (s, 9H); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta = 191.49, 167.29, 158.79, 130.57, 129.63, 126.00, 37.06, 35.34, 33.97, 30.97\). IR (KBr, cm\(^{-1}\)): \((\nu_{\text{C=O}}) 1678, 1650\).

HRMS (ESI) [M+H]\(^+\) Calcd. for C\(_{14}\)H\(_{20}\)NO\(_2\): 234.1494 Found: 234.1491.

2-(4-Methoxyphenyl)-N,N-dimethyl-2-oxoacetamide

3da: Yellow oil.

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta = 7.90\) (d, \(J = 8.8\) Hz, 2H), 6.98–6.95 (m, 2H), 3.86 (s, 3H), 3.09 (s, 3H), 2.94 (s, 3H); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta = 190.46, 167.38, 164.84, 132.08, 127.01, 114.31, 55.60, 37.05, 33.94\).

2-(4-Fluorophenyl)-N,N-dimethyl-2-oxoacetamide

3ea: White solid.

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta = 8.00–7.97\) (m, 2H), 7.20–7.16 (m, 2H), 3.11 (s, 3H), 2.97 (s, 3H); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta = 189.97, 166.66\) (d, \(J = 256.0\) Hz), 166.60, 132.47 (d, \(J = 10.0\) Hz), 129.67 (d, \(J = 3.0\) Hz), 116.29 (d, \(J = 22.0\) Hz), 37.02, 34.05.

2-(4-Chlorophenyl)-N,N-dimethyl-2-oxoacetamide
3fa:[7] Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.91$–7.89 (m, 2H), 7.50–7.48 (m, 2H), 3.12 (s, 3H), 2.97 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 190.26$, 166.47, 141.31, 131.53, 131.01, 129.37, 37.02, 34.08.

2-(4-Bromophenyl)-N,N-dimethyl-2-oxoacetamide

3ga: Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.82$–7.80 (m, 2H), 7.66–7.64 (m, 2H), 3.11 (s, 3H), 2.96 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 190.48$, 166.43, 132.37, 131.92, 131.04, 130.16, 37.02, 34.09. IR (KBr, cm$^{-1}$): (νC=O) 1679, 1646. HRMS (ESI) [M+H]$^+$ Calcd. for C$_{10}$H$_{11}$BrNO$_2$: 255.9973, Found: 255.9970.

2-(2-Chlorophenyl)-N,N-dimethyl-2-oxoacetamide

3ha: Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.89$–7.87 (m, 1H), 7.52–7.48 (m, 1H), 7.44–7.37 (m, 2H), 3.07 (s, 3H), 3.07 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 190.14$, 166.93, 134.27, 133.72, 133.45, 132.22, 130.76, 127.27, 37.04, 34.50. IR (KBr, cm$^{-1}$): (νC=O) 1679, 1650. HRMS (ESI) [M+H]$^+$ Calcd. for C$_{10}$H$_{11}$ClNO$_2$: 212.0478, Found: 212.0475.

2-(2,5-Dichlorophenyl)-N,N-dimethyl-2-oxoacetamide


**3ia:** Yellow solid.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.84$–7.83 (m, 1H), 7.47–7.44 (m, 1H), 7.38–7.36 (m, 1H), 3.08 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 188.67$, 166.18, 134.91, 133.99, 133.67, 131.85, 131.74, 131.69, 37.04, 34.60. IR (KBr, cm$^{-1}$): ($\nu$C=O) 1675, 1648. HRMS (ESI) [M+H]$^+$ Calcd. for C$_{10}$H$_{10}$Cl$_2$NO$_2$: 246.0089, Found: 246.0087.

**2-(3-Bromophenyl)-N,N-dimethyl-2-oxoacetamide**

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 8.09$ (s, 1H), 7.88 (d, $J = 8.0$ Hz, 1H), 7.77 (d, $J = 8.0$ Hz, 1H), 7.41–7.37 (m, 1H), 3.13 (s, 3H), 2.97 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 190.05$, 166.20, 137.48, 134.92, 132.38, 130.54, 128.27, 123.25, 37.03, 34.12. IR (KBr, cm$^{-1}$): ($\nu$C=O) 1683, 1650. HRMS (ESI) [M+H]$^+$ Calcd. for C$_{10}$H$_{11}$BrNO$_2$: 255.9973, Found: 255.9971.

**3ja:** Yellow solid.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.83$–7.81 (m, 1H), 7.64–7.62 (m, 1H), 7.45–7.37 (m, 2H), 3.09 (s, 3H), 3.07 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 190.79$, 166.27, 135.37, 134.06, 134.03, 132.57, 127.69, 121.48, 37.18, 34.61. IR (KBr, cm$^{-1}$): ($\nu$C=O) 1675, 1646. HRMS (ESI) [M+H]$^+$ Calcd. for C$_{10}$H$_{11}$BrNO$_2$: 255.9973, Found: 255.9975.

**3ka:** Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.83$–7.81 (m, 1H), 7.64–7.62 (m, 1H), 7.45–7.37 (m, 2H), 3.09 (s, 3H), 3.07 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 190.79$, 166.27, 135.37, 134.06, 134.03, 132.57, 127.69, 121.48, 37.18, 34.61. IR (KBr, cm$^{-1}$): ($\nu$C=O) 1675, 1646. HRMS (ESI) [M+H]$^+$ Calcd. for C$_{10}$H$_{11}$BrNO$_2$: 255.9973, Found: 255.9975.
**N,N-Dimethyl-2-(naphthalen-1-yl)-2-oxoacetamide**

![N,N-Dimethyl-2-(naphthalen-1-yl)-2-oxoacetamide](image)

3la: Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 9.27$–9.25 (m, 1H), 8.12–8.10 (m, 1H), 8.01–7.99 (m, 1H), 7.93–7.91 (m, 1H), 7.72–7.68 (m, 1H), 7.61–7.52 (m, 2H), 3.16 (s, 3H), 3.02 (s, 3H);

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta =$ 194.20, 167.68, 135.84, 134.29, 134.08, 130.98, 129.29, 128.72, 128.51, 126.97, 125.80, 124.54, 37.17, 34.15.

**2-(Furan-2-yl)-N,N-dimethyl-2-oxoacetamide**

![2-(Furan-2-yl)-N,N-dimethyl-2-oxoacetamide](image)

3ma: Yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta =$ 7.69 (s, 1H), 7.34 (br, s, 1H), 6.59–6.58 (m, 1H), 3.05 (s, 3H), 3.01 (s, 3H);

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta =$ 178.50, 165.41, 150.17, 148.69, 122.28, 112.82, 37.14, 34.45.

**N,N-Dimethyl-2-(4-nitrophenyl)-2-oxoacetamide**

![N,N-Dimethyl-2-(4-nitrophenyl)-2-oxoacetamide](image)

3na: Yellow solid.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta =$ 8.34–8.32 (m, 2H), 8.14–8.13 (m, 2H), 3.14 (s, 3H), 3.00 (s, 3H);

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta =$ 189.24, 165.60, 151.08, 137.57, 130.76, 124.05, 37.05, 34.28.
6. References


7. $^1$H and $^{13}$C NMR spectra of the products
Electronic Supplementary Material (ESI) for Chemical Communications
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