Supporting information

A Novel Approach for the One-Pot Preparation of α-Ketoamides by anode Oxidation
Zhenlei Zhang, Jihu Su, Zhenggen Zha* and Zhiyong Wang*
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**General Remarks:** Unless otherwise indicated, all commercial reagents and solvents were used without additional purification. $^1$H-NMR and $^{13}$C-NMR were recorded on a Bruker AVIII-400 spectrometer. Chemical shifts (in ppm) were referenced to tetramethylsilane ($\delta = 0$ ppm) in CDCl$_3$ as an internal standard. $^{13}$C-NMR spectra were obtained by the same NMR spectrometer and were calibrated with CDCl$_3$ ($\delta = 77.00$ ppm).

1. **Experimental Section.**

**Instruments:** The instrument for electrolysis is dual display potentiostat (CJS-292) (made in China). The anode electrode and cathode electrode all are Pt (1.5×1.5 cm$^2$). A saturated calomel electrode (SCE) was used as the reference electrode.

**Representative procedures for synthesis of α-Ketoamides:** An undivided cell was equipped with a magnet stirrer, platinum electrode as the working electrode and counter electrode. In the electrolytic cell a solution of acetophenone (0.5 mmol), amines (2 mmol), n-Bu$_4$NI (1 mmol), O$_2$ (balloon) and EtOH (10 ml) was allowed to stir and electrolyze at a constant current of 20 mA for three hours until the quantity of the electricity 4.5 F/mol was passed at room temperature. Upon completion of the reaction, the solvent was removed with the aid of a rotary evaporator. The residue was purified by column chromatography on silica gel, and the product was dried under high vacuum for at least 0.5h before it was weighed and characterized by NMR spectroscopy.

**Representative procedures for Synthesis of α-Oxobenzeneacetamides:** An undivided cell was equipped with a magnet stirrer, platinum electrode as the working electrode and counter electrode. In the electrolytic cell a solution of acetophenone (0.5 mmol), NH$_4$OAc (2 mmol), t-BuNH$_2$ (2 mmol), n-Bu$_4$NI (1 mmol), O$_2$ (balloon) and EtOH (10 ml) was allowed to stir and electrolyze at a constant current of 40 mA for five hours until the quantity of the electricity 15 F/mol was passed at room temperature. Upon completion of the reaction, the solvent was removed with the aid of a rotary evaporator. The residue was purified by column chromatography on silica gel, and the product was dried under high vacuum for at least 0.5h before it was weighed and characterized by NMR spectroscopy.

**Representative procedures for Synthesis of 2-oxo-2-phenylacetaldehyde:** An undivided cell was equipped with a magnet stirrer, platinum electrode as the working electrode and counter electrode. In the electrolytic cell a solution of acetophenone (0.5 mmol), t-BuNH$_2$ (2 mmol), n-Bu$_4$NI (1 mmol), O$_2$ (balloon) and EtOH (10 ml) was allowed to stir and electrolyze at a constant current of 20 mA for three hours until the quantity of the electricity 4.5 F/mol was passed at room temperature. Upon completion of the reaction, the solvent was removed with the aid of a rotary evaporator. The residue was purified by column chromatography on silica gel, and the product was dried under high vacuum for at least 0.5h before it was weighed and characterized by NMR spectroscopy.

**Experimental details for the capture of radical:** An undivided cell was equipped with a magnet stirrer, two platinum electrodes both as the working electrode and the counter electrode respectively. In the electrolytic cell a solution of acetophenone (0.5 mmol), n-BuNH$_2$ (2 mmol), n-Bu$_4$NI (1 mmol), O$_2$ (balloon) and EtOH (10 ml) was allowed to stir and electrolyze at a constant current of 20 mA for one hour. 0.05 ml solution was taken out into a small tube, mixed well with 0.01 ml DMPO. Then this mixture was analyzed by EPR. The EPR measurements were performed with a Braker Elexsys X-band.
(9.7 GHz) E580 EPR spectrometer at room temperature. The X-band EPR measurement was conducted at room temperature and the result was shown in Fig. S1.

**Figure S1.** The overall 9.7 GHz X-band cw-EPR spectra (a) of the radicals in Scheme 3 trapped by DMPO, and their simulations (b, c, d, e). With EPR simulation, the complicate spectral overlapping in spectrum a is de-convoluted: spectrum b, c and d are assigned to the radical signals originating from the DMPO-CH$_2$COPh (7'), DMPO-OOCH$_2$COPh (8'), and DMPO-OH (9') complexes respectively. The overlapping of the spectrum b, c and d with a intensity ratio of 1:5:15 leads to both the complicate spectra e, corresponding to the experimental result a.

The intermediate radical 7 trapped by DMPO was characterized by the hyperfine coupling (hfc). Constants for the nitrogen ($A_N = 23$ G) and the $\beta$-proton ($A_H = 8$ G) in pyrroline (Spectrum b in Figure 1). The data showed that the radical 7 was centered at an alkoxyl group. The active intermediate 8 was trapped by DMPO as a superoxide radical $^\cdot$O$_2^-$, characterized by the hfc's of nitrogen ($A_N = 14.4$G) and the $\beta$-proton ($A_H = 8.5$ G) in pyrroline (Spectrum c in Figure. 1). 9 can be readily trapped by DMPO as
in the known Fenton’s reaction (Spectrum d in Figure 1). The hyperfine constant for the nitrogen and proton in d are $A_{1N} = 14.6$ and $A_{1H} = 14.6$ respectively.
2. Optimization of Reaction Conditions

Table S1: Optimization of reaction conditions.\textsuperscript{a}

\[ \text{Entry} \quad \text{Electrolyte} \quad \text{Solvent} \quad \text{Yield (\%)} \textsuperscript{b} \]

<table>
<thead>
<tr>
<th>Entry</th>
<th>Electrolyte</th>
<th>Solvent</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>KI</td>
<td>EtOH</td>
<td>40</td>
</tr>
<tr>
<td>2</td>
<td>n-Bu(_4)\text{NI}</td>
<td>EtOH</td>
<td>80</td>
</tr>
<tr>
<td>3</td>
<td>NaI</td>
<td>EtOH</td>
<td>43</td>
</tr>
<tr>
<td>4</td>
<td>n-Bu(_4)\text{NBr}</td>
<td>EtOH</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>LiClO(_4)</td>
<td>EtOH</td>
<td>0</td>
</tr>
<tr>
<td>6\textsuperscript{b}</td>
<td>n-Bu(_4)\text{NI}</td>
<td>EtOH</td>
<td>&lt;5</td>
</tr>
<tr>
<td>7</td>
<td>n-Bu(_4)\text{NI}</td>
<td>MeOH</td>
<td>50</td>
</tr>
<tr>
<td>8</td>
<td>n-Bu(_4)\text{NI}</td>
<td>THF</td>
<td>0</td>
</tr>
<tr>
<td>9</td>
<td>n-Bu(_4)\text{NI}</td>
<td>CH(_2)\text{Cl}_2</td>
<td>0</td>
</tr>
<tr>
<td>10</td>
<td>n-Bu(_4)\text{NI}</td>
<td>DMSO</td>
<td>&lt;5</td>
</tr>
<tr>
<td>11\textsuperscript{c}</td>
<td>n-Bu(_4)\text{NI}</td>
<td>EtOH</td>
<td>0</td>
</tr>
<tr>
<td>12\textsuperscript{d}</td>
<td>n-Bu(_4)\text{NI}</td>
<td>EtOH</td>
<td>0</td>
</tr>
<tr>
<td>13\textsuperscript{e}</td>
<td>n-Bu(_4)\text{NI}</td>
<td>EtOH</td>
<td>75</td>
</tr>
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\textsuperscript{a} Reaction conditions: 1a (0.5 mmol), 2a (2 mmol), electrolyte (1 mmol), solvent (10 ml), O\(_2\) (balloon), platinum sheet as anode and cathode, at room temperature. \textsuperscript{b} 2 equiv of H\(_2\)O was added. \textsuperscript{c} N\(_2\) instead O\(_2\). \textsuperscript{d} 1 mmol of TEMPO was added. \textsuperscript{e} n-Bu\(_4\)\text{NI} (0.1 mmol), n-Bu\(_4\)\text{NBr} (1 mmol).
Table S2. Optimization of Reaction Conditions for α-Oxobenzeneacetamide\textsuperscript{a}

\[
\begin{array}{cccc}
\text{entry} & \text{amine} & \text{N source} & \text{yield\%}\textsuperscript{b} \\
1 & & \text{NH}_2\text{OAc} & 25 \\
2 & NH_2 (1b) & \text{NH}_2\text{OAc} & 75 \\
3 & & \text{NH}_2\text{OAc} & 55 \\
4 & & \text{NH}_2\text{OAc} & 47 \\
5 & 1b & \text{HCOO}\text{NH}_2 & 63 \\
6 & 1b & (\text{NH}_4)_2\text{CO}_3 & 42 \\
7 & 1b & \text{NH}_4\text{HCO}_3 & 46 \\
8 & 1b & \text{NH}_2\text{Cl} & 52 \\
9 & 1b & \text{NH}_2\text{H}_2\text{O} & 23 \\
\end{array}
\]

\textsuperscript{a} Reaction conditions: 1a (0.5mmol), amine (2 mmol), KI (1mmol), N source (2 mmol), MeOH (10 ml), O\textsubscript{2} (balloon), platinum sheet as anode and cathode, at a constant of 40 mA for 5 hours in an undivided cell.\textsuperscript{b} Isolated yields.
Table S3. Optimization of the synthesis 2-oxo-2-phenylacetaldehyde

<table>
<thead>
<tr>
<th>Entry</th>
<th>Amine</th>
<th>Yield$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>none</td>
<td>0%</td>
</tr>
<tr>
<td>2</td>
<td>2-methylpropan-2-amine</td>
<td>64%</td>
</tr>
<tr>
<td>3</td>
<td>KOH</td>
<td>0%</td>
</tr>
<tr>
<td>4</td>
<td>piperidine</td>
<td>trace</td>
</tr>
<tr>
<td>5</td>
<td>2,2,6,6-tetramethylpiperidine</td>
<td>34%</td>
</tr>
<tr>
<td>6</td>
<td>diisopropylamine</td>
<td>23%</td>
</tr>
<tr>
<td>7</td>
<td>triethylamine</td>
<td>10%</td>
</tr>
<tr>
<td>8$^c$</td>
<td>2-methylpropan-2-amine</td>
<td>56%</td>
</tr>
<tr>
<td>9$^d$</td>
<td>2-methylpropan-2-amine</td>
<td>trace</td>
</tr>
</tbody>
</table>

$^a$ Reaction conditions: 1a (0.5 mmol), amine (2 mmol), KI (1 mmol), MeOH (10 ml), O$_2$ (balloon), platinum sheet as anode and cathode, at a constant of 20 mA for 3 hours. $^b$ Isolated yield. $^c$ n-Bu$_4$NI as electrolyte, EtOH as solvent. $^d$ Under the air condition.
3. Characterization data of all products.

**General Remarks:** $^1$H NMR and $^{13}$C NMR were recorded on a Bruker AC-300 FT ($^1$H: 400 MHz, $^{13}$C: 100 MHz) using TMS as internal reference. The chemical shifts ($\delta$) and coupling constants ($J$) were expressed in ppm and Hz respectively.

**3a** N-butyl-2-oxo-2-phenylacetamide

\[
\begin{array}{c}
\text{O} \\
\text{H} \\
\text{N} \\
\text{CH}_3 \\
\text{CH}_2 \\
\end{array}
\]

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 8.33$ (t, $J = 1.6$ Hz, 2 H), 7.64-7.59 (m, 1 H), 7.49-7.45 (m, 2 H), 7.09 (b, 1 H), 3.42-3.37 (m, 2 H), 1.63-1.56 (m, 2 H), 1.45-1.35 (m, 2 H), 0.96 (t, $J = 7.4$ Hz, 3 H);

$^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 187.9, 161.7, 134.3, 133.4, 131.2, 128.4, 39.1, 31.3, 20.063, 13.6$.

**3b** 2-(benzo[d][1, 3]dioxol-5-yl)-N-butyl-2-oxoacetamide

\[
\begin{array}{c}
\text{O} \\
\text{H} \\
\text{N} \\
\text{CH}_3 \\
\text{CH}_2 \\
\end{array}
\]

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 8.16$ (dd, $J_1 = 8$ Hz, $J_2 = 2$ Hz, 1 H), 7.78 (d, $J = 1.6$ Hz, 1 H), 7.17 (b, 1 H), 6.86 (d, $J = 8$ Hz, 1 H), 6.04 (s, 2 H), 3.39-3.34 (m, 2 H), 1.61-1.54 (m, 2 H), 1.44-1.37 (m, 2 H), 0.96-0.89 (m, 3 H);

$^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 185.5, 162.1, 153.0, 147.9, 129.0, 127.9, 110.2, 108.0, 101.9, 39.1, 31.3, 20.0, 13.6$.

**3c** N-butyl-2-(2-chlorophenyl)-2-oxoacetamide

\[
\begin{array}{c}
\text{Cl} \\
\text{O} \\
\text{H} \\
\text{N} \\
\text{CH}_3 \\
\text{CH}_2 \\
\end{array}
\]

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 7.68-7.66$ (m, 1 H) 7.46-7.44 (m, 2 H), 7.37-7.32 (m, 1 H), 7.01 (b, 1 H), 3.41-3.36 (m, 2 H), 1.63-1.56 (m, 2 H), 1.45-1.36 (m, 2 H), 0.97-0.92 (m, 3 H);

$^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 190.3, 160.7, 134.1, 133.0, 132.9, 131.2, 130.4, 126.5, 39.4, 31.2, 20.0, 13.6$.

**3d** N-butyl-2-oxo-2-m-tolylacetamide

\[
\begin{array}{c}
\text{O} \\
\text{H} \\
\text{N} \\
\text{CH}_3 \\
\text{CH}_2 \\
\end{array}
\]
1HNMR (CDCl₃, 400 MHz, ppm): δ = 8.14-8.12 (m, 2 H), 7.43-7.41 (m, 1 H), 7.37-7.26 (m, 1 H), 7.09 (s, 1 H), 3.41-3.36 (m, 2 H), 2.40 (s, 3 H), 1.63-1.55 (m, 2 H), 1.47-1.36 (m, 2 H), 0.95 (t, J = 7.2 Hz, 3 H); 13CNMR (CDCl₃, 100 MHz, ppm): δ = 188.1, 161.9, 138.2, 135.1, 133.4, 131.5, 128.4, 128.3, 39.1, 31.3, 21.3, 20.0, 13.6.

3e N-butyl-2-oxo-2-p-tolylacetamide

![Structure of 3e](image)

1HNMR (CDCl₃, 400 MHz, ppm): δ = 8.26-8.24 (m, 2 H), 7.27-7.25 (m, 2 H), 7.15 (b, 1 H), 3.40-3.35 (m, 2 H), 2.41 (s, 3 H), 1.62-1.54 (m, 2 H), 1.47-1.35 (m, 2 H), 0.96-0.94 (m, 3 H); 13CNMR (CDCl₃, 100 MHz, ppm): δ = 187.4, 162.0, 145.5, 131.3, 130.9, 129.2, 39.1, 31.3, 21.8, 20.0, 13.7.

3f 2-(biphenyl-4-yl)-N-butyl-2-oxoacetamide

![Structure of 3f](image)

1HNMR (CDCl₃, 400MHz, ppm): δ = 8.45-8.42 (m, 2 H), 7.70-7.61 (m, 4 H), 7.48-7.36 (m, 3 H), 7.15 (b, 1 H), 3.43-3.38 (m, 2 H), 1.64-1.57 (m, 2 H), 1.46-1.37 (m, 2 H), 0.98-0.96 (m, 3 H); 13CNMR (CDCl₃, 100 MHz, ppm): δ = 187.3, 161.9, 146.9, 139.7, 132.1, 131.8, 128.9, 128.4, 127.3, 127.0, 39.1, 31.3, 20.0, 13.7.

3g N-butyl-2-(4-methoxyphenyl)-2-oxoacetamide

![Structure of 3g](image)

1HNMR (CDCl₃, 400 MHz, ppm): δ = 8.44-8.40 (m, 2 H), 7.12 (b, 1 H), 6.96-6.92 (m, 2 H), 3.88 (s, 3 H), 3.40-3.35 (m, 2 H), 1.62-1.55 (m, 2 H), 1.45-1.36 (m, 2 H), 1.00-0.93 (m, 3 H); 13CNMR (CDCl₃, 100 MHz, ppm): δ = 185.8, 164.6, 162.2, 133.9, 129.3, 113.8, 55.5, 39.1, 31.3, 20.0, 13.7.

3h N-butyl-2-(4-fluorophenyl)-2-oxoacetamide

![Structure of 3h](image)

1HNMR (CDCl₃, 400 MHz, ppm): δ = 8.39-8.35 (m, 2 H), 7.12-7.04 (m, 2 H), 3.34-3.29 (m, 2 H), 1.57-1.48 (m, 2 H), 1.38-1.28 (m, 2 H), 093-0.85 (m, 3 H); 13CNMR (CDCl₃, 100 MHz, ppm): δ = 184.9, 166.8, 164.37, 160.5, 133.3, 133.2, 128.8, 128.8, 114.8, 114.6, 38.1, 30.3, 19.0, 12.6.

3j 2-oxo-N, 2-diphenylacetamide
HNMR (CDCl$_3$, 400 MHz, ppm): $\delta$ = 8.94 (b, 1 H), 8.43-8.39 (m, 2 H), 7.71-7.63 (m, 3 H), 7.53-7.40 (m, 2 H), 7.38-7.36 (m, 2 H), 7.22-7.17 (m, 1 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta$ = 187.4, 158.8, 136.6, 134.6, 133.1, 131.4, 129.2, 128.5, 125.3, 119.9.

3k 2-oxo-2-phenyl-N-p-tolylacetamide

HNMR (CDCl$_3$, 400 MHz, ppm): $\delta$ = 8.82 (b, 1 H), 8.35-8.32 (m, 2 H), 7.59-7.55 (m, 1 H), 7.52-7.49 (m, 1 H), 7.45-7.40 (m, 2 H), 2.27 (s, 3 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta$ = 186.5, 157.7, 134.0, 133.5, 133.0, 132.1, 130.4, 128.7, 127.5, 118.8, 19.9.

3l N-(4-methoxyphenyl)-2-oxo-2-phenylacetamide

HNMR (CDCl$_3$, 400 MHz, ppm): $\delta$ = 8.86 (b, 1 H), 8.43-8.40 (m, 2 H), 7.67-7.60 (m, 3 H), 7.53-7.48 (m, 2 H), 6.94-6.90 (m, 2 H), 3.82 (s, 3 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta$ = 187.6, 158.6, 157.1, 134.5, 133.2, 131.4, 129.8, 128.5, 121.5, 114.4, 55.5.

3m N-(3-nitrophenyl)-2-oxo-2-phenylacetamide

HNMR (CDCl$_3$, 400 MHz, ppm): $\delta$ = 9.21 (b, 1 H), 8.68-8.66 (m, 1 H), 8.45-8.42 (m, 2 H), 8.07-8.01 (m, 2 H), 7.72-7.67 (m, 1 H), 7.60-7.51 (m, 3 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta$ = 186.3, 158.9, 148.7, 137.7, 135.0, 132.6, 131.5, 130.1, 128.7, 125.4, 119.8, 114.8.

3n N-(2-methylbenzyl)-2-oxo-2-phenylacetamide

HNMR (CDCl$_3$, 400 MHz, ppm): $\delta$ = 8.37-8.34 (m, 2 H), 7.64-7.60 (m, 1 H), 7.49-7.46 (m, 2 H), 7.34 (s, 1 H), 7.23 (t, J = 8 Hz, 2 H), 7.16 (d, J = 4 Hz, 2 H), 4.52 (d, J = 2.8 Hz, 2 H), 2.34 (s, 3 H);
$^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 187.5, 161.4, 137.6, 134.4, 134.0, 133.3, 131.2, 129.5, 128.5, 127.9, 43.2, 21.1.$

3o 2-oxo-2-phenyl-N-(4-(trifluoromethyl)phenyl)acetamide

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 8.37-8.34$ (m, 2 H), 7.71-7.61 (m, 1 H), 7.53-7.46 (m, 2 H), 7.41 (b, 1 H), 7.32-7.28 (m, 2 H), 7.06-7.01 (m, 2 H), 4.53 (d, $J = 2.8$ Hz, 2 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 187.4, 163.6, 161.5, 161.1, 134.5, 133.2, 132.9, 132.9, 131.2, 130.9, 129.6, 129.6, 128.7, 128.5, 115.8, 115.6, 42.7.$

3p N-(3-methylbenzyl)-2-oxo-2-phenylacetamide

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 8.37-8.35$ (m, 2 H), 7.64-7.60 (m, 1 H), 7.49-7.46 (m, 2 H), 7.38 (b, 1 H), 7.26-7.22 (m, 1 H), 7.13-7.10 (m, 3 H), 4.53 (d, $J = 2.8$ Hz, 2 H), 2.34 (s, 3 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 187.5, 161.5, 138.6, 137.0, 134.4, 133.3, 131.2, 128.7, 128.6, 128.5, 128.5, 128.1, 124.9, 43.4, 21.3.$

3q N-benzyl-2-oxo-2-phenylacetamide

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 8.38-8.35$ (m, 2 H), 7.62-7.61 (m, 1 H), 7.53-7.47 (m, 2 H), 7.39-7.28 (m, 6 H), 4.58 (d, $J = 2.8$ Hz, 2 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 187.5, 161.5, 137.1, 134.4, 133.3, 131.2, 128.8, 128.5, 127.9, 127.8, 43.5.$

3r N-(benzo[d] [1, 3] dioxol-5-ylmethyl)-2-oxo-2-phenylacetamide

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 8.36-8.34$ (m, 2 H), 7.64-7.60 (m, 1 H), 7.50-7.46 (m, 2 H), 7.33 (b, 1 H), 6.81-6.76 (m, 3 H), 5.95 (s, 3 H), 4.46 (d, $J = 2.8$ Hz, 2 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 187.5, 161.4, 148.0, 147.2, 134.4, 133.3, 131.2, 130.9, 128.5, 121.3, 108.5, 108.4, 101.1, 43.3.$

3s (R)-2-oxo-2-phenyl-N-(1-phenylethyl)acetamide

S12
$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 8.33$ (d, $J = 0.6$ Hz, 2 H), 7.63-7.59 (m, 1 H), 7.48-7.44 (m, 2 H), 7.39-7.34 (m, 4 H), 7.32-7.25 (m, 1 H), 5.22-5.15 (m, 1 H), 1.60 (d, $J = 3.6$ Hz, 3 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 187.6, 160.7, 142.2, 134.4, 133.3, 131.2, 128.8, 128.4, 127.7, 126.1, 49.1, 21.7$.

3t N,N-dibenzyl-2-oxo-2-phenylacetamide

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 8.01-7.98$ (m, 2 H), 7.64-7.62 (m, 1 H), 7.53-7.48 (m, 2 H), 7.39-7.29 (m, 8 H), 7.25-7.22 (m, 2 H), 4.62 (s, 2 H), 4.28 (s, 2 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 190.7, 166.9, 135.4, 134.2, 132.8, 129.2, 128.3, 128.1, 127.7, 127.6, 127.4, 49.5, 45.5$.

3u 1-morpholino-2-phenylethane-1, 2-dione

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 7.97-7.94$ (m, 2 H), 7.67-7.63 (m, 1 H), 7.54-7.50 (m, 2 H), 3.80-3.77 (m, 4 H), 3.66-3.64 (m, 2 H), 3.39-3.36 (m, 2 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 191.1, 165.4, 134.9, 133.2, 129.6, 129.1, 66.7, 66.6, 46.2, 41.6$.

3v 1-phenyl-2-(piperidin-1-yl) ethane-1, 2-dione

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 7.96-7.93$ (m, 2 H), 7.66-7.62 (m, 1 H), 7.53-7.49 (m, 2 H), 3.71-3.69 (m, 2 H), 3.30-3.27 (m, 2 H), 1.76-1.62 (m, 4 H), 1.57-1.53 (m, 2 H); $^{13}$CNMR (CDCl$_3$, 100 MHz, ppm): $\delta = 191.9, 165.4, 134.6, 133.27, 129.5, 129.0, 47.0, 42.1, 26.2, 25.4, 24.3$.

3w N,N-diethyl-2-oxo-2-phenylacetamide

$^1$HNMR (CDCl$_3$, 400 MHz, ppm): $\delta = 7.95-7.92$ (m, 2 H), 7.65-7.61 (m, 1 H), 7.52-7.48 (m, 2 H), 3.56 (dd, $J_1 = 14.4$ Hz, $J_2 = 7.2$ Hz, 2H), 3.24 (dd, $J_1 = 14.4$ Hz, $J_2 = 7.2$ Hz, 2 H), 1.29 (t, $J = 7.2$ Hz, 3
H), 1.15 (t, J = 7.2 Hz, 3 H); ¹³C NMR (CDCl₃, 100 MHz, ppm): δ = 191.6, 166.7, 134.5, 133.2, 129.6, 128.9, 42.1, 38.7, 14.1, 12.8.

3x N-hexyl-2-oxo-2-phenylacetamide

¹H NMR (CDCl₃, 400 MHz, ppm): δ = 8.35-8.33 (m, 2 H), 7.64-7.59 (m, 1 H), 7.49-7.45 (m, 2 H), 7.08 (b, 1 H), 3.41-3.36 (m, 2 H) 1.64-1.23 (m, 6 H), 0.96-0.83 (m, 3 H); ¹³C NMR (CDCl₃, 100 MHz, ppm): δ = 191.6, 166.7, 134.5, 133.2, 129.6, 128.9, 42.1, 38.7, 14.1, 12.8.

3y N-hexadecyl-2-oxo-2-phenylacetamide

¹H NMR (CDCl₃, 400 MHz, ppm): δ = 8.34-8.32 (m, 2 H), 7.63-7.60 (m, 1 H), 7.50-7.46 (m, 2 H), 7.09 (b, 1 H), 3.39-3.34 (m, 2 H), 1.68-1.59 (m, 2 H), 1.01-0.96 (m, 3 H); ¹³C NMR (CDCl₃, 100 MHz, ppm): δ = 187.9, 161.7, 134.3, 133.4, 131.2, 128.4, 39.4, 31.9, 29.6, 29.5, 29.3, 29.2, 26.9, 22.6, 14.1.

3z 2-oxo-2-phenyl-N-propylacetamide

¹H NMR (CDCl₃, 400 MHz, ppm): δ = 8.36-8.33 (m, 2 H), 7.64-7.60 (m, 1 H), 7.50-7.46 (m, 2 H), 7.03 (b, 1 H), 6.28 (b, 1 H); ¹³C NMR (CDCl₃, 100 MHz, ppm): δ = 187.4, 164.1, 134.5, 133.0, 131.1, 128.4, 41.1, 22.5, 11.3.

6a 2-oxo-2-phenylacetamide

¹H NMR (CDCl₃, 400 MHz, ppm): δ = 8.30-8.28 (m, 2 H), 7.65-7.61 (m, 1 H), 7.52-7.46 (m, 2 H), 7.03 (b, 1 H), 6.28 (b, 1 H); ¹³C NMR (CDCl₃, 100 MHz, ppm): δ = 187.4, 164.1, 134.5, 133.0, 131.1, 128.5.

6b 2-(4-fluorophenyl)-2-oxoacetamide
6c 2-oxo-2-(4-(trifluoromethyl) phenyl) acetamide

6d 2-(4-bromophenyl)-2-o xoacetamide

6e 2-(4-methoxyphenyl)-2-o xoacetamide

6f 2-(napthalen-2-yl)-2-o xoacetamide

6g 2-oxo-2-(o-tolyl) acetamide
$^1$HNMR (DMSO-D$_6$, 400 MHz, ppm): $\delta$ = 8.28 (b, 1 H), 7.92 (b, 1 H), 7.71 (d, $J$ = 8 Hz, 1 H), 7.55-7.50 (m, 1 H), 7.38-7.34 (m, 2 H) 2.49 (s, 3 H); $^{13}$CNMR (DMSO-D$_6$, 100 MHz, ppm): $\delta$ = 193.5, 167.4, 139.3, 132.8, 132.3, 131.7, 131.6, 125.8, 20.5.

6h 2-oxo-2-(m-tolyl) acetamide

$^1$HNMR (DMSO-D$_6$, 400 MHz, ppm): $\delta$ = 8.31 (b, 1 H), 7.97 (b, 1 H), 7.90-7.88 (m, 2 H), 7.49-7.46 (m, 1 H), 2.39 (s, 3 H); $^{13}$CNMR (DMSO-D$_6$, 100 MHz, ppm): $\delta$ = 190.9, 167.2, 138.3, 135.0, 132.7, 129.7, 128.8, 126.9, 20.7.

6i 2-oxo-2-(p-tolyl) acetamide

$^1$HNMR (DMSO-D$_6$, 400 MHz, ppm): $\delta$ = 8.28 (b, 1 H) 7.94 (b, 1 H), 7.90-7.88 (m, 2 H), 7.39-7.37 (m, 2 H), 2.39 (s, 3 H); $^{13}$CNMR (DMSO-D$_6$, 100 MHz, ppm): $\delta$ = 190.3, 167.3, 145.1, 130.2, 129.7, 129.4, 21.2.
3. NMR Spectra of all products
Electronic Supplementary Material (ESI) for Chemical Communications
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