I$_2$-Mediated Oxidative Dehydrogenation of β-Acylamino ketones for the Highly Stereoselective Synthesis of Z-β-Ketoenamides

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**General Information**

$^1$H NMR spectra were recorded at 400 MHz and $^{13}$C NMR spectra were measured at 100 MHz using Bruker AVANCE III NMR spectrometers with CDCl$_3$ as the solvent. Chemical shifts (δ) were measured in ppm and referenced to the deuterated chloroform ($^1$H: δ = 7.26 ppm, $^{13}$C: δ = 77.00 ppm). The multiplicities of signals were described using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, tt = triplet of triplets. High-resolution mass spectra (HRMS) were performed on a microOTOF-Q II instrument with an ESI source. Melting points were measured with a RD-II type melting point apparatus. Substrates 1a-1r were prepared following our previous report, and carbamates 1s and 1t were prepared according to the known procedure.

**Table S1. Base Screening for Dehydrogenation Reaction**

<table>
<thead>
<tr>
<th>Entry</th>
<th>Base</th>
<th>Yield (%)$^b$</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>2a</td>
</tr>
<tr>
<td>1</td>
<td>K$_2$CO$_3$</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>DABCO</td>
<td>60</td>
</tr>
<tr>
<td>3</td>
<td>DBU</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>4</td>
<td>TMG</td>
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$^a$ Reaction conditions: 0.2 mmol of 1a, 0.24 mmol of I$_2$, 0.6 mmol of base, in 2 mL of $p$-xylene, at 60 °C for 4-6 h. $^b$ Isolated yield.
Table S2. Catalyst and Oxidant Screening for the Synthesis of 2a$^a$

<table>
<thead>
<tr>
<th>Entry</th>
<th>Oxidant</th>
<th>Catalyst</th>
<th>Solvent</th>
<th>Yield (%)$^b$</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>TBHP</td>
<td>KI</td>
<td>p-xylene</td>
<td>29</td>
</tr>
<tr>
<td>2</td>
<td>TBHP</td>
<td>TBAI</td>
<td>p-xylene</td>
<td>30</td>
</tr>
<tr>
<td>3</td>
<td>TBHP</td>
<td>I$_2$</td>
<td>p-xylene</td>
<td>52</td>
</tr>
<tr>
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<td>I$_2$</td>
<td>1.4-dioxane</td>
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<td>THF</td>
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<td>6</td>
<td>H$_2$O$_2$</td>
<td>I$_2$</td>
<td>p-xylene</td>
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<tr>
<td>7</td>
<td>TBPB</td>
<td>I$_2$</td>
<td>p-xylene</td>
<td>42</td>
</tr>
</tbody>
</table>

$^a$ Reaction conditions: 0.2 mmol of 1a, 0.02 mmol of catalyst, 0.4 mmol of DABCO, 0.4 mmol of oxidant, in 2 mL of solvent, at 60 °C for 4-6 h.$^b$ Isolated yield.

Characterization of Products

Dehydrogenation; General Procedure

A 10 mL oven-dried reaction vessel was charged with 1a (53 mg, 0.2 mmol), DABCO (67 mg, 0.6 mmol), and iodine (61 mg, 0.24 mmol) in paraxylene (2.0 mL). The resulting solution was stirred at 60 °C for 5 h. After the reaction was complete, sat. Na$_2$S$_2$O$_3$ aqueous solution (10 mL) was added to quench the reaction, and the mixture was extracted by ethyl acetate (3 × 10 mL). The organic layer was separated and dried over anhydrous Na$_2$SO$_4$. After the removal of the solvent under vacuo, the residue was purified by flash column chromatography with PE/EtOAc (9 : 1) to give 2a.

(Z)-N-(3-oxo-1,3-diphenylprop-1-en-1-yl)acetamide (2a)

Yield: 27 mg (60%); time: 5 h; white solid; m.p. 60-62 °C; TLC, $R_t = 0.35$ (PE:EtOAc = 4:1); $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 12.27 (s,
1H), 7.98-7.95 (m, 2H), 7.57 (tt, 1H, J = 4.8, 0.8 Hz), 7.50-7.46 (m, 4H), 7.45-7.40 (m, 3H), 6.33 (s, 1H), 2.25 (s, 3H); 13C NMR (CDCl₃, 100 MHz): δ 191.7, 168.9, 156.3, 138.6, 136.2, 132.7, 129.8, 128.7, 128.1, 127.8, 127.4, 104.8, 25.1; HRMS (ESI) m/z calcd. for C₁₇H₁₆NO₂ [M+H]+: 266.1176, found: 266.1179.

(Z)-N-(3-(4-chlorophenyl)-3-oxo-1-phenylprop-1-en-1-yl) acetamide (2b)

Yield: 37 mg (60%); time: 5 h; white solid; m.p. 106-109 °C; TLC, Rf = 0.35 (PE:EtOAc = 4:1); 1H NMR (CDCl₃, 400 MHz): δ 12.23 (s, 1H), 7.90 (d, 2H, J = 6.8 Hz), 7.49-7.40 (m, 7H), 6.25 (s, 1H), 2.24 (s, 3H); 13C NMR (CDCl₃, 100 MHz): δ 190.2, 168.8, 156.9, 139.2, 136.9, 136.1, 129.9, 129.2, 129.0, 128.1, 127.4, 104.2, 25.1. HRMS (ESI) m/z calcd. for C₁₇H₁₅ClNO₂ [M+H]+: 300.0786, found: 300.0791.

(Z)-N-(3-(4-bromophenyl)-3-oxo-1-phenylprop-1-en-1-yl) acetamide (2c)

Yield: 50 mg (69%); time: 6 h; white solid; m.p. 99-102 °C; TLC, Rf = 0.36 (PE:EtOAc = 4:1); 1H NMR (CDCl₃, 400 MHz): δ 12.32 (s, 1H), 7.82 (d, 2H, J = 8.4 Hz), 7.61 (d, 2H, J = 8.4 Hz), 7.48-7.40 (m, 5H), 6.25 (s, 1H), 2.24 (s, 3H); 13C NMR (CDCl₃, 100 MHz): δ 190.4, 168.8, 157.0, 137.4, 136.1, 132.0, 130.0, 129.3, 128.1, 127.4, 104.2, 25.1. HRMS (ESI) m/z calcd. for C₁₇H₁₅BrNO₂ [M+H]+: 344.0281, found: 344.0282.

(Z)-N-(3-oxo-1-phenyl-3-(p-tolyl)prop-1-en-1-yl) acetamide (2d)

Yield: 32 mg (56%); time: 8 h; white solid; m.p. 117-120 °C; TLC, Rf = 0.34 (PE:EtOAc = 4:1); 1H NMR (CDCl₃, 400 MHz): δ 12.29 (s, 1H), 7.87 (d, 2H, J = 8.4 Hz), 7.48-7.38 (m, 5H), 7.28 (d, 2H, J = 8.0 Hz), 6.31 (s, 1H), 2.43 (s, 3H), 2.24 (s, 3H); 13C NMR (CDCl₃, 100 MHz): δ 191.4, 168.9, 155.9, 143.7, 136.4, 136.0, 129.8, 129.4, 128.1, 128.0, 127.4,

(Z)-N-(3-(4-methoxyphenyl)-3-oxo-1-phenylprop-1-en-1-yl) acetamide (2e)

Yield: 31 mg (51%); time: 7 h; white solid; m.p. 125-128 °C; TLC, Rf = 0.36 (PE:EtOAc = 4:1); ¹H NMR (CDCl₃, 400 MHz): δ 12.29 (s, 1H), 7.96 (d, 2H, J = 9.2 Hz), 7.47-7.38 (m, 5H), 6.96 (d, 2H, J = 9.2 Hz ), 6.29 (s, 1H), 3.88 (s, 3H), 2.23 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 190.3, 168.9, 163.4, 155.5, 136.4, 131.4, 130.1, 129.6, 128.1, 127.3, 113.9, 104.7, 55.5, 25.1. HRMS (ESI) m/z calcd. for C₁₈H₁₈NO₃[M+H]⁺: 296.1281, found: 296.1280.

(Z)-N-(3-(naphthalen-2-yl)-3-oxo-1-phenylprop-1-en-1-yl) acetamide (2f)

Yield: 37 mg (58%); time: 6 h; white solid; m.p. 112-115 °C; TLC, Rf = 0.35 (PE:EtOAc = 4:1); ¹H NMR (CDCl₃, 400 MHz): δ 12.36 (s, 1H), 8.48 (s, 1H), 8.05 (dd, 1H, J =8.8, 2.0 Hz ), 7.98-7.88 (m, 3H), 7.62-7.42 (m, 7H), 6.49 (s, 1H), 2.27 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 191.5, 168.9, 156.2, 136.3, 135.9, 135.4, 132.6, 129.8, 129.5, 129.2, 128.6, 128.4, 128.1, 127.7, 127.4, 126.8, 123.8, 104.8, 25.1. HRMS (ESI) m/z calcd. for C₂₁H₁₈NO₂[M+H]⁺: 316.1332, found: 316.1326.

(Z)-N-(3-(furan-2-yl)-3-oxo-1-phenylprop-1-en-1-yl) acetamide (2g)

Yield: 27 mg (53%); time: 5 h; yellow oil; TLC, Rf = 0.32 (PE:EtOAc = 4:1); ¹H NMR (CDCl₃, 400 MHz): δ 12.06 (s, 1H), 7.60 (d, 1H, J = 1.2 Hz ), 7.47-7.36 (m, 5H ), 7.23 (d, 1H, J = 3.6 Hz), 6.56 (dd, 1H, J = 4.4, 1.6 Hz), 6.23 (s, 1H), 2.21(s, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 179.8, 168.7, 156.2, 153.4, 146.4, 135.9, 129.8, 128.0, 127.3, 116.7, 112.6,
104.3, 25.0. HRMS (ESI) m/z calcd. for C\textsubscript{15}H\textsubscript{14}NO\textsubscript{3} [M+H]\(^+\): 256.0968, found: 256.0967.

(Z)-N-(3-oxo-1-phenyl-3-(thiophen-2-yl)prop-1-en-1-yl) acetamide (2h)

Yield: 33 mg (61%); time 5 h; yellow oil; TLC, R\textsubscript{f} = 0.31 (PE:EtOAc = 4:1); \(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz): \(\delta\) 12.06 (s, 1H), 7.73 (dd, 1H, \(J = 4.0, 0.8\) Hz), 7.66 (dd, 1H, \(J = 4.8, 1.2\) Hz), 7.47-7.38 (m, 5H), 7.14 (dd, 1H, \(J = 5.2, 4.0\) Hz), 6.19 (s, 1H), 2.21 (s, 3H); \(^{13}\)C NMR (CDCl\textsubscript{3}, 100 MHz): \(\delta\) 183.9, 168.7, 155.9, 145.7, 135.9, 133.8, 130.9, 129.8, 128.3, 128.0, 127.3, 104.7, 25.0. HRMS (ESI) m/z calcd. for C\textsubscript{15}H\textsubscript{14}NO\textsubscript{2}S [M+H]\(^+\): 272.0740, found: 272.0746.

(Z)-N-(1-(naphthalen-2-yl)-3-oxo-3-phenylprop-1-en-1-yl) acetamide (2i)

Yield: 39 mg (64%); time: 7 h; yellow oil; TLC, R\textsubscript{f} = 0.35 (PE:EtOAc = 4:1); \(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz): \(\delta\) 12.37 (s, 1H), 8.02-7.97 (m, 3H), 7.92-7.83 (m, 3H), 7.59-7.47 (m, 6H), 6.45 (s, 1H), 2.27 (s, 3H); \(^{13}\)C NMR (CDCl\textsubscript{3}, 100 MHz): \(\delta\) 191.7, 168.9, 156.2, 138.6, 133.96, 133.91, 132.83, 132.77, 128.7, 128.5, 127.8, 127.7, 127.4, 127.0, 126.6, 126.5, 125.1, 105.0, 25.1. HRMS (ESI) m/z calcd. for C\textsubscript{21}H\textsubscript{18}NO\textsubscript{2} [M+H]\(^+\): 316.1332, found: 316.1331.

(Z)-N-(3-oxo-3-phenyl-1-(p-tolyl)prop-1-en-1-yl) acetamide (2j)

Yield: 34 mg (59%); time: 8 h; colorless oil; TLC, R\textsubscript{f} = 0.35 (PE:EtOAc = 4:1); \(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz): \(\delta\) 12.26 (s, 1H), 7.97 (d, 2H, \(J = 7.2\) Hz), 7.56 (t, 1H, \(J = 7.2\) Hz), 7.48 (t, 2H, \(J = 7.2\) Hz), 7.38 (d, 2H, \(J = 8.4\) Hz), 7.22 (d, 2H, \(J = 8.4\) Hz), 6.33 (s, 1H), 2.41 (s, 3H), 2.25 (s, 3H); \(^{13}\)C NMR (CDCl\textsubscript{3}, 100 MHz): \(\delta\) 191.6, 168.9, 156.4, 140.2, 138.7, 133.2, 132.6, 128.8, 128.6, 127.7, 127.3, 104.4, 25.1, 21.4. HRMS (ESI) m/z calcd. for C\textsubscript{18}H\textsubscript{18}NO\textsubscript{2} [M+H]\(^+\): 280.1332, found: 280.1330.
(Z)-N-(1-(2-methoxyphenyl)-3-oxo-3-phenylprop-1-en-1-yl) acetamide (2k)
Yield: 29 mg (50%); time: 12 h; white solid; m.p. 97-99 °C; TLC, Rf = 0.42 (PE:EtOAc = 4:1); ¹H NMR (CDCl₃, 400 MHz): δ 12.57 (s, 1H), 7.94 (dd, 2H, J = 8.0, 1.2 Hz), 7.54 (t, 1H, J = 7.6 Hz), 7.46 (t, 2H, J = 7.6 Hz), 7.42-7.38 (m, 1H), 7.27 (dd, 1H, J = 7.6, 2.0 Hz), 7.01 (td, 1H, J = 7.2, 0.8 Hz), 6.89 (d, 1H, J = 8.4 Hz), 6.17 (s, 1H), 3.82 (s, 3H), 2.20 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 191.9, 168.2, 156.9, 154.1, 138.7, 132.5, 130.7, 128.60, 128.56, 127.7, 125.9, 120.4, 110.3, 103.4, 55.6, 24.9. HRMS (ESI) m/z calcd. for C₁₉H₁₇NO₃Na [M+Na]⁺: 318.1101, found: 318.1100.

(Z)-N-(1-(4-bromophenyl)-3-oxo-3-phenylprop-1-en-1-yl) acetamide (2l)
Yield: 41 mg (60%); time: 6 h; yellow solid; m.p. 94-96 °C; TLC, Rf = 0.45 (PE:EtOAc = 4:1); ¹H NMR (CDCl₃, 400 MHz): δ 12.24 (s, 1H), 7.95 (d, 2H, J = 7.2 Hz), 7.60-7.45 (m, 5H), 7.33 (d, 2H, J = 8.4 Hz ), 6.29 (s, 1H), 2.25 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 191.7, 168.9, 154.9, 138.4, 135.1, 132.9, 131.3, 128.9, 128.7, 127.8, 124.2, 104.8, 25.1. HRMS (ESI) m/z calcd. for C₁₇H₁₄BrNO₂Na [M+Na]⁺: 366.0100, found: 366.0091.

(Z)-N-(1-(4-chlorophenyl)-3-oxo-3-phenylprop-1-en-1-yl) acetamide (2m)
Yield: 35 mg (58%); time 6 h; yellow oil; TLC, Rf = 0.40 (PE:EtOAc = 4:1); ¹H NMR (CDCl₃, 400 MHz): δ 12.25(s, 1H), 7.95 (d, 2H, J = 7.6 Hz ), 7.58 (t, 1H, J = 7.6 Hz ), 7.49 (t, 2H, J = 7.6 Hz ), 7.41-7.34 (m, 4H), 6.29 (s, 1H), 2.25 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 191.7, 168.9, 154.9, 138.4, 135.9, 134.6, 132.9, 128.71, 128.70, 128.4, 127.8, 104.8,
25.1. HRMS (ESI) m/z calcd. for C_{17}H_{15}ClNO_2 [M+H]^+: 300.0786, found: 300.0781.

(Z)-N-(1-(2-chlorophenyl)-3-oxo-3-phenylprop-1-en-1-yl) acetamide (2n)

Yield: 30 mg (50%); time: 6 h; colorless oil; TLC, R_f = 0.34 (PE:EtOAc = 4:1); ^1H NMR (CDCl_3, 400 MHz): δ 12.55 (s, 1H), 7.95 (d, 2H, J = 7.2 Hz), 7.56 (t, 1H, J = 7.2 Hz), 7.47 (t, 1H, J = 7.2 Hz), 7.42-7.39 (m, 1H), 7.37-7.31 (m, 3H), 6.15 (s, 1H), 2.18 (s, 3H); ^13C NMR (CDCl_3, 100 MHz): δ 191.9, 168.1, 153.1, 138.3, 135.8, 132.8, 132.1, 130.0, 129.2, 128.9, 128.7, 127.8, 126.6, 103.7, 24.7. HRMS (ESI) m/z calcd. for C_{17}H_{15}ClNO_2 [M+H]^+: 300.0786, found: 300.0776.

(Z)-N-(1-(4-nitrophenyl)-3-oxo-3-phenylprop-1-en-1-yl) acetamide (2o)

Yield: 44 mg (70%); time: 6 h; white solid; m.p. 158-161 °C; TLC, R_f = 0.34 (PE:EtOAc = 4:1); ^1H NMR (CDCl_3, 400 MHz): δ 12.26 (s, 1H), 8.26 (d, 2H, J = 8.8 Hz), 7.98-7.94 (m, 2H), 7.63-7.57 (m, 3H), 7.50 (t, 2H, J = 7.6 Hz), 6.32 (s, 1H), 2.26 (s, 3H); ^13C NMR (CDCl_3, 100 MHz): δ 191.7, 168.9, 153.3, 148.2, 142.8, 138.1, 133.3, 128.8, 128.2, 128.0, 123.4, 105.7, 24.9. HRMS (ESI) m/z calcd. for C_{17}H_{15}N_2O_4 [M+H]^+: 311.1026, found: 311.1024.

(Z)-N-(1-(3-nitrophenyl)-3-oxo-3-phenylprop-1-en-1-yl) acetamide (2p)

Yield: 48 mg (78%); time: 6 h; white solid; m.p. 134-136 °C; TLC, R_f = 0.35 (PE:EtOAc = 4:1); ^1H NMR (CDCl_3, 400 MHz): δ 12.28 (s, 1H), 8.32-8.36 (m, 2H), 7.97 (d, 2H, J = 7.2 Hz), 7.76 (d, 1H, J = 6.4 Hz), 7.62-7.55 (m, 2H), 7.53-7.47 (m, 2H), 6.33 (s, 1H), 2.27 (s, 3H); ^13C NMR (CDCl_3, 100 MHz): δ 191.7, 169.0, 153.1, 147.9, 138.1, 138.0,
133.3, 133.2, 129.0, 128.8, 127.9, 124.2, 122.3, 105.5, 24.9. HRMS (ESI) m/z calcd. for C_{17}H_{15}N_{2}O_{4} [M+H]^{+}: 311.1026, found: 311.1018.

(Z)-N-(3-oxo-1,3-diphenylprop-1-en-1-yl)benzamide (2r)\textsuperscript{3b}

Yield: 23 mg (35%); time: 2 h; white solid; m.p. 99-101 °C; TLC, \( R_{f} = 0.29 \) (PE:EtOAc = 9:1); \(^1\)H NMR (CDCl\(_3\), 400 MHz): \( \delta \) 12.37 (s, 1H), 8.14-8.10 (m, 2H), 8.03-7.99 (m, 2H), 7.63-7.52 (m, 6H), 7.51-7.43 (m, 5H), 6.46 (s, 1H); \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \( \delta \) 192.1, 165.3, 157.1, 138.6, 136.4, 133.4, 132.79, 132.76, 129.8, 128.9, 128.7, 128.1, 127.9, 127.4, 105.4. HRMS (ESI) m/z calcd. for C\(_{22}\)H\(_{18}\)NO\(_2\) [M+H]^{+}: 380.1257, found: 380.1255.

(Z)-ethyl (3-oxo-1,3-diphenylprop-1-en-1-yl)carbamate (2s)

Yield: 44 mg (75%); time: 2 h; yellow oil; TLC, \( R_{f} = 0.51 \) (PE:EtOAc = 9:1); \(^1\)H NMR (CDCl\(_3\), 400 MHz): \( \delta \) 11.95 (s, 1H), 7.97 (d, 2H, \( J = 7.2 \) Hz), 7.57-7.41 (m, 8H), 6.28 (s, 1H), 4.13 (q, 2H, \( J = 7.2 \) Hz), 1.26 (t, 3H, \( J = 7.2 \) Hz); \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \( \delta \) 191.2, 157.0, 152.9, 138.7, 136.0, 132.5, 129.8, 128.6, 128.0, 127.8, 127.5, 103.5, 61.9, 14.2. HRMS (ESI) m/z calcd. for C\(_{18}\)H\(_{17}\)NO\(_3\)Na [M+Na]^{+}: 318.1101, found: 318.1104.

(Z)-benzyl (3-oxo-1,3-diphenylprop-1-en-1-yl)carbamate (2t)

Yield: 53 mg (78%); time: 2 h; yellow oil; TLC, \( R_{t} = 0.52 \) (PE:EtOAc = 9:1); \(^1\)H NMR (CDCl\(_3\), 400 MHz): \( \delta \) 12.03 (s, 1H), 7.96 (d, 2H, \( J = 7.2 \) Hz), 7.58-7.32 (m, 13H), 6.30 (s, 1H), 5.12 (s, 2H); \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \( \delta \) 191.3, 156.7, 152.7, 138.6, 135.9, 135.4, 132.6, 129.9, 128.6, 128.5, 128.31, 128.29, 128.0, 127.8, 127.6, 103.8, 67.5. HRMS (ESI) m/z calcd. for C\(_{23}\)H\(_{19}\)NO\(_3\)Na[M+Na]^{+}: 380.1257, found: 380.1255.
References:


NMR Spectra of Products

$^1$H NMR, 400 MHz, CDCl$_3$

$^1$H NMR of compound 2a shows signals at various ppm values.

$^{13}$C NMR, 100 MHz, CDCl$_3$

$^{13}$C NMR of compound 2a shows signals at various ppm values.
$^{1}H$ NMR 400MHz CDCl$_3$

2b

$^{13}C$ NMR 100 MHz CDCl$_3$

2b
$^1$H NMR 400MHz CDCl$_3$

$^1$C NMR 100MHz CDCl$_3$
$2t$

$^1H$ NMR 400MHz CDCl$_3$

$^13C$ NMR 100MHz CDCl$_3$