Electronic Supplementary Information

Room temperature decarboxylation/C-H functionalization cascade by visible-light photoredox catalysis

Jin Xie, Pan Xu, Huamin Li, Qicai Xue, Hongming Jin, Yixiang Cheng and Chengjian Zhu

State Key Laboratory of Coordination Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, P. R. China.

State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Shanghai 200032, P. R. China.

E-mail: cjzhu@nju.edu.cn

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Part I. General Information

Unless otherwise stated, all the reactions were performed under argon atmosphere. Solvents and reagents were used as received from suppliers unless otherwise stated. $^1$H NMR, $^{13}$C NMR and $^{19}$F NMR data were obtained on Bruker Advance III 400 MHz nuclear resonance spectrometers with CDCl$_3$ as solvents at ambient temperature. Chemical shifts were reported in units (ppm) by assigning chloroform residue in the $^1$H NMR spectrum as 7.26 ppm. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet and m = multiplet), coupling constant (J values) in Hz and integration. Chemical shifts for $^{13}$C NMR spectra were recorded in ppm from chloroform using the central peak of CDCl$_3$ (77.0 ppm) as the internal standard. Flash column chromatography was performed using 200-300 mesh silica with the indicated solvent system according to standard techniques. Reactions were monitored by TLC on silica gel plates (GF254), and the analytical thin-layer chromatography (TLC) was performed on precoated, glass-backed silica gel plates. Visualization of the developed chromatogram was performed by UV absorbance (254 nm). Low resolution mass spectra were obtained using ThermoFisher Scientific LCQ FLEET mass spectrometer or Daojin (Japan) LC-MS 2020 spectrometer. High resolution mass spectra were obtained using an Agilent 6210 Series TOF LC-MS or G6520B Accurate-Mass Q-TOF LC/MS equipped with electrospray ionization (ESI) probe operating in positive ion mode. Melting points (m.p.) were determined with a digital electrothermal apparatus without further correction. The alkene substrates 1 were prepared according to the literature. The 35 W fluorescent light bulb was directly got from the supermarket (daylight, energy saving, 220 V, 50 Hz).
Part II. Optimization of the Tandem Reaction Conditions.

Table 1. Optimization of the Decarboxylation/C-H Functionalization Tandem Reaction

<table>
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<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>Solvent</th>
<th>Additives</th>
<th>Time/h</th>
<th>Yield(%)</th>
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<tr>
<td>1</td>
<td>(1%)</td>
<td>MeCN</td>
<td>DIB (3.0 equiv)</td>
<td>20</td>
<td>51</td>
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<tr>
<td>2</td>
<td>(1%)</td>
<td>MeCN</td>
<td>DIB (3.0 equiv)</td>
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<td>72</td>
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<tr>
<td>3</td>
<td>(1%)</td>
<td>DCM</td>
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<tr>
<td>4</td>
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<td>DMF</td>
<td>DIB (3.0 equiv)</td>
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<td>83</td>
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<td>5</td>
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<td>K$_2$HPO$_4$ (2.0 equiv)</td>
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<td></td>
<td>DIB (3.0 equiv)</td>
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<td>DMF</td>
<td>NaHCO$_3$ (2.0 equiv)</td>
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<td>KOBu-t (2.0 equiv)</td>
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<td>DIB (3.0 equiv)</td>
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<td>DIB (3.0 equiv)</td>
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<td>DIB (1.0 equiv)</td>
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<td>40</td>
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<td>DIB (4.0 equiv)</td>
<td>24</td>
<td>83</td>
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<tr>
<td>19</td>
<td>-</td>
<td>DMF</td>
<td>DIB (3.0 equiv)</td>
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<td>NP</td>
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<td>20$^c$</td>
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<td>DMF</td>
<td>DIB (3.0 equiv)</td>
<td>72</td>
<td>NP</td>
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$^a$Reaction conditions: 1a (1.0 equiv), DIB 2a (1.0-4.0 equiv), fac-Ir(ppy)$_3$ (1-3 mol%), argon atmosphere, room temperature, 35 W fluorescent light bulb. $^b$Isolated yields. $^c$In the dark.
The $^{13}$C NMR (400 MHz, $d^6$DMSO) of Eq. 1.

Scheme 1. Screening of another reaction substrates.
Part III. General Experimental Details of the Visible-Light-Mediated Decarboxylation/Radical-C-H Functionalization Tandem Reactions and Characterization Data for Products

General procedure

Method A:

An oven-dried Schlenk tube (20 mL) was equipped with a magnetic stir bar, 1a-p (0.3 mmol), \( \text{fac-Ir(ppy)}_3 \) (2.0 mg, 0.01 equiv), PhI(OAc)_2 (3.0 equiv) and DMF (2.0 mL). The tube was degassed by alternating vacuum evacuation (5 min) and argon backfill three times. The tube was placed at a distance (app. 5 cm) from 35 W fluorescent light bulb, and the resulting yellow solution was stirred at ambient temperature under visible-light irradiation. When the reaction finished, the mixture was diluted with ethyl acetate and added to a separatory funnel containing 15 mL saturated K_2CO_3 solution. The layers were separated and the aqueous layer was extracted with ethyl acetate (3 x 15 mL). The combined organic layers were washed with saturated brine, dried (Na_2SO_4) and filtered. The filtrate was concentrated in vacuo and the residue was purified by chromatography on silica gel (petroleum ether 60-90 : EtOAc, 10:1-1:1 v/v) to afford the oxindoles 3a-q.

Method B:

In a 25 mL round flask was equipped with a magnetic stir bar, aliphatic carboxylic acid (3.0 mmol, 2.0 equiv), PhI(OAc)_2 (1.5 mmol, 1.0 equiv) and CHCl_3 (10 mL). The phenyliodine(III) dicarboxylate can be easily obtained as a white solid or a viscous oil
at 30-40 °C under reduced pressure to remove the HOAc, and it can be directly used without further purification. Then, an oven-dried Schlenk tube (20 mL) was equipped with a magnetic stir bar, 1a (0.3 mmol), \textit{fac}-Ir(ppy)\textsubscript{3} (2.0 mg, 0.01 equiv), phenylidiodine(III) dicarboxylate (3.0 equiv) and DMF (2.0 mL). The tube was degassed by alternating vacuum evacuation (5 min) and argon backfill three times. The tube was palced at a distance (app. 5 cm) from 35 W fluorescent light bulb, and the resulting yellow solution was stirred at ambient temperature under visible-light irradiation. When the reaction finished, the mixture was diluted with ethyl acetate and added to a separatory funnel containing 15 mL saturated K\textsubscript{2}CO\textsubscript{3} solution. The layers were separated and the aqueous layer was extracted with ethyl acetate (3 x 15 mL). The combined organic layers were washed with saturated brine, dried (Na\textsubscript{2}SO\textsubscript{4}) and filtered. The filtrate was concentrated in vacuo and the residue was purified by chromatography (petroleum ether 60-90 : EtOAc, 10:1-3:1 v/v) to afford the oxindoles 4a-u.

**Characterization data**

**3-ethyl-1,3-dimethylindolin-2-one 3a**

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 24 h, and purified by flash column chromatography in 83% yield. Colorless oil, TLC (PE:EA, 5:1): R\textsubscript{f} = 0.38. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): \(\delta\) (ppm) = 7.30-7.24 (m, 1 H), 7.19-7.15 (m, 1 H), 7.09-7.04 (m, 1 H), 6.84 (d, \(J = 8.0\) Hz, 1 H), 3.22 (s, 3 H), 1.98-1.86 (m, 1 H), 1.82-1.71 (m, 1 H), 1.35 (s, 3 H), 0.59 (t, \(J = 7.2, 7.6\) Hz, 3 H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): \(\delta\) (ppm) = 180.8, 143.5, 134.0, 127.6, 122.5, 122.4, 107.8, 49.0, 31.5, 26.1, 23.3, 8.8; MS (ESI) \(m/z\): 212.33 [M+Na]\textsuperscript{+}; HRMS (ESI) \(m/z\) calcd for C\textsubscript{12}H\textsubscript{16}NO [M+H]\textsuperscript{+}: 190.1226; found:190.1225.
1,3-diethyl-3-methylindolin-2-one 3b

The title compound was prepared according to the general method described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 80% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.40. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.29-7.23 (m, 1 H), 7.20-7.15 (m, 1 H), 7.09-7.02 (m, 1 H), 6.89-6.83 (m, 1 H), 3.90-3.66 (m, 2 H), 1.99-1.89 (m, 1 H), 1.82-1.70 (m, 1 H), 1.34 (s, 3 H), 1.25 (t, $J$ = 7.2 Hz, 3 H), 0.57 (t, $J$ = 7.6 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 180.3, 142.6, 134.2, 127.5, 122.7, 122.2, 108.0, 48.8, 34.5, 34.6, 23.4, 12.8, 8.8; MS (ESI) m/z: 226.33 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{13}$H$_{17}$NNaO [M+Na]$^+$: 226.1202; found: 226.1201.

3-ethyl-1-isopropyl-3-methylindolin-2-one 3c

The title compound was prepared according to the general method described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 88% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.60. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.26-7.13 (m, 2 H), 7.07-6.99 (m, 2 H), 4.72-4.61 (m, 1 H), 1.99-1.87 (m, 1 H), 1.80-1.68 (m, 1 H), 1.48 (d, $J$ = 3.2 Hz, 3 H), 1.46 (d, $J$ = 3.2 Hz, 3 H), 1.33 (s, 3 H), 0.55 (t, $J$ = 7.6 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 180.4, 142.2, 134.5, 127.3, 122.7, 121.8, 109.6, 48.5, 43.5, 31.8, 23.5, 19.6, 19.4, 8.7; MS (ESI) m/z: 240.42 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{14}$H$_{20}$NO [M+H]$^+$: 218.1539; found: 218.1538.

1-benzyl-3-ethyl-3-methylindolin-2-one 3d

The title compound was prepared according to the general method described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 73% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.49. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.34-7.23 (m, 5 H), 7.20-7.12 (m, 2 H), 7.07-6.99 (m, 1 H), 6.72 (d, $J$ = 8.0 Hz, 1 H),
4.99 (d, $J = 15.6$ Hz, 1 H), 4.85 (d, $J = 15.6$ Hz, 1 H), 2.07-1.95 (m, 1 H), 1.89-1.77 (m, 1 H), 1.41 (s, 3 H), 0.64 (t, $J = 7.2$ Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 180.8, 142.6, 136.2, 133.9, 128.7 (2C), 127.5 (2C), 127.3 (2C), 122.6, 122.4, 108.9, 49.0, 43.7, 31.5, 23.8, 9.1; MS(ESI): MS (ESI) $m/z$: 288.33 [M+Na]$^+$; HRMS (ESI) $m/z$ calcd for C$_{18}$H$_{19}$NNaO [M+Na]$^+$: 288.1359; found: 288.1354.

3-ethyl-3-methyl-1-phenylindolin-2-one 3e

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 24 h, and purified by flash column chromatography in 90% yield. Colorless oil, TLC (PE:EA, 5:1): $R_f = 0.54$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.56-7.48 (m, 2 H), 7.44-7.37 (m, 3 H), 7.25-7.06 (m, 3 H), 6.86-6.80 (m, 1 H), 2.11-1.97 (m, 1 H), 1.92-1.79 (m, 1 H), 1.47 (s, 3 H), 0.71 (t, $J = 7.2$ Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 180.2, 143.4, 134.7, 133.7, 129.6 (2C), 127.9, 127.5, 126.6 (2C), 122.9, 122.8, 109.2, 49.0, 32.1, 23.6, 8.9; MS (ESI) $m/z$: 274.33 [M+Na]$^+$; HRMS (ESI) $m/z$ calcd for C$_{17}$H$_{17}$NNaO [M+Na]$^+$: 274.1202; found: 274.1197.

3-ethyl-1,3,5-trimethylindolin-2-one 3f

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 80% yield. Colorless oil, TLC (PE:EA, 5:1): $R_f = 0.34$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.09-7.03 (m, 1 H), 6.98 (s, 1 H), 6.73 (d, $J = 7.6$ Hz, 1 H), 3.19 (s, 3 H), 2.35 (s, 3 H), 1.97-1.86 (m, 1 H), 1.80-1.69 (m, 1 H), 1.34 (s, 3 H), 0.59 (t, $J = 7.6$, 7.2 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 180.7, 141.1, 134.0, 131.9, 127.8, 123.4, 49.0, 31.5, 26.1, 23.4, 21.2, 8.9. MS (ESI) $m/z$: 226.42 [M+Na]$^+$; HRMS (ESI) $m/z$ calcd for C$_{13}$H$_{17}$NNaO [M+Na]$^+$: 226.1202; found:226.1201.
3-ethyl-5-methoxy-1,3-dimethylindolin-2-one 3g

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 76% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.22. $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 6.82-6.71 (m, 3 H), 3.01 (s, 3 H), 3.19 (s, 3 H), 1.99-1.88 (m, 1 H), 1.80-1.68 (m, 1 H), 1.34 (s, 3 H), 0.59 (t, J = 7.2, 7.6 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 180.4, 156.1, 137.1, 135.4, 111.5, 110.4, 108.0, 55.8, 49.4, 31.5, 26.2, 23.4, 8.9; MS (ESI) m/z: 242.33 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{13}$H$_{17}$NNaO$_2$ [M+Na]$^+$: 242.1151; found: 242.1149.

5-chloro-3-ethyl-1,3-dimethylindolin-2-one 3h

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 85% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.36. $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.25-7.21 (m, 1 H), 7.17-7.12 (m, 1 H), 6.76 (d, J = 8.4 Hz, 1 H), 3.19 (s, 3 H), 1.99-1.87 (m, 1 H), 1.82-1.70 (m, 1 H), 1.34 (s, 3 H), 0.59 (t, J = 7.2 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 180.2, 142.1, 135.7, 127.8, 127.6, 123.1, 108.7, 9.3, 31.4, 26.2, 23.3, 8.8; MS (ESI) m/z: 246.42 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{12}$H$_{15}$ClNO [M+H]$^+$: 224.0837; found: 224.0838.

5-bromo-3-ethyl-1,3-dimethylindolin-2-one 3i

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 81% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.40. $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.42-7.36 (m, 1 H), 7.30-7.25 (m, 1 H), 6.72 (d, J = 8.0 Hz, 1 H), 3.19 (s, 3 H), 1.98-1.88 (m, 1 H), 1.80-1.69 (m, 1 H), 1.34 (s, 3 H), 0.60 (t, J = 7.6 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 180.1, 142.6, 136.1, 130.5,

3,3-diethyl-1-methylindolin-2-one 3k

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 82% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.43. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.29-7.23 (m, 1 H), 7.16-7.03 (m, 2 H), 6.83 (d, J = 7.6 Hz, 1 H), 3.21 (s, 3 H), 1.98-1.86 (m, 2 H), 1.84-1.72 (m, 2 H), 0.56 (t, J = 7.2 Hz, 6 H); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 180.1, 144.4, 132.0, 127.6, 122.7, 122.3, 107.6, 54.3, 30.6 (2C), 25.9, 8.7 (2C); MS (ESI) m/z: 226.33 [M+Na]⁺; HRMS (ESI) m/z calcd for C₁₃H₁₇NNaO [M+Na]⁺: 226.1202; found:226.1200.

3-allyl-3-ethyl-1-methylindolin-2-one 3l

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 24 h, and purified by flash column chromatography in 83% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.42. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.30-7.23 (m, 1 H), 7.19-7.14 (m, 1 H), 7.09-7.03 (m, 1 H), 6.84 (d, J = 7.6 Hz, 1 H), 5.47-5.33 (m, 1 H), 5.00-4.84 (m, 2 H), 3.19 (s, 3 H), 2.59-2.47 (m, 2 H), 2.00-1.75 (m, 2 H), 0.57 (t, J = 7.2 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 178.4, 143.0, 131.5, 130.6, 126.7, 122.0, 121.3, 117.4, 106.7, 52.5, 40.8, 29.1, 25.0, 7.6; MS (ESI) m/z: 238.33 [M+Na]⁺; HRMS (ESI) m/z calcd for C₁₄H₁₈NO [M+H]⁺: 216.1383; found: 216.1381.

3-benzyl-3-ethyl-1-methylindolin-2-one 3m

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb
for 36 h, and purified by flash column chromatography in 73% yield. White solid, m.p. 71-73 °C; TLC (PE:EA, 5:1): Rf = 0.44. $^1$H NMR (400 MHz, CDCl3): δ (ppm) = 7.20-7.10 (m, 2 H), 7.08-6.99 (m, 4 H), 6.87-6.79 (m, 2 H), 6.58 (d, J = 8.0 Hz, 1 H), 3.12 (d, J = 12.8 Hz, 1 H), 3.00 (d, J = 12.8 Hz, 1 H), 2.96 (s, 3 H), 2.16-2.07 (m, 1 H), 1.96-1.86 (m, 1 H), 0.59 (t, J = 7.2 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl3): δ (ppm) = 179.2, 144.1, 136.1, 131.0, 129.9, 127.7, 127.4, 126.3, 123.4, 122.0, 107.6, 55.5, 44.0, 30.1, 25.8, 8.9; MS (ESI) m/z: 288.33 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{18}$H$_{19}$NNaO [M+Na]$^+$: 288.1359; found: 288.1354.

3-ethyl-1,3-dimethyl-1H-pyrrolo[2,3-b]pyridin-2(3H)-one 3n

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 40 h at 40 °C with 3.0 equiv K$_2$HPO$_4$, and purified by flash column chromatography in 61% yield. Colorless oil, TLC (PE:EA, 3:2): Rf = 0.49. $^1$H NMR (400 MHz, CDCl3): δ (ppm) = 8.17 (dd, J = 1.6, 5.2 Hz, 1 H), 7.39 (dd, J = 7.2, 1.6 Hz), 6.96 (dd, J = 7.2, 5.2 Hz, 1 H), 3.30 (s, 3 H), 2.00-1.89 (m, 1 H), 1.83-1.73 (m, 1 H), 1.37 (s, 3 H), 0.64 (t, J = 7.2 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl3): δ (ppm) = 179.3, 156.0, 145.6, 128.8, 127.3, 117.0, 47.6, 30.0, 24.2, 21.7, 7.8; MS (ESI) m/z: 213.33 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{11}$H$_{15}$N$_2$O [M+H]$^+$: 191.1179; found: 191.1186.

3-ethyl-1,3,6-trimethyl-1H-pyrrolo[2,3-b]pyridin-2(3H)-one 3o

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 40 h at 40 °C with 3.0 equiv K$_2$HPO$_4$, and purified by flash column chromatography in 70% yield. White solid, m.p. 84-86 °C; TLC (PE:EA, 5:1): Rf = 0.60. $^1$H NMR (400 MHz, CDCl3): δ (ppm) = 7.28 (d, J = 7.2 Hz, 1 H), 6.80 (d, J = 7.2 Hz, 1 H), 3.29 (s, 3 H), 2.51 (s, 3 H), 1.97-1.86 (m, 1 H), 1.81-1.71 (m, 1 H), 1.34 (s, 3 H), 0.63 (t, J = 7.32 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl3): δ (ppm) = 180.8, 156.6, 155.9, 130.1, 124.9, 116.9, 48.4, 31.0, 25.2, 24.2,
22.8, 8.9; MS (ESI) m/z: 227.25 [M+Na]+; HRMS (ESI) m/z calcd for C₁₂H₁₇N₂O [M+H]+: 205.1335; found: 205.1340.

3-ethyl-1,3-dimethyl-1H-pyrrolo[3,2-b]pyridin-2(3H)-one 3p

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 40 h at 40 °C with 3.0 equiv K₂HPO₄, and purified by flash column chromatography in 48% yield. Colorless oil, TLC (PE:EA, 3:2): Rf = 0.27. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.27-8.20 (m, 1 H), 7.15 (dd, J = 5.2, 4.0 Hz, 1 H), 7.05 (dd, J = 4.0, 1.2 Hz, 1 H), 3.22 (s, 3 H), 2.05-1.90 (m, 2 H), 1.40 (s, 3 H), 0.58 (t, J = 7.2 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 178.3, 154.2, 141.8, 137.6, 121.3, 112.7, 48.3, 29.3, 24.8, 20.6, 7.8; MS (ESI) m/z: 213.42 [M+Na]+; HRMS (ESI) m/z calcd for C₁₁H₁₅N₂O [M+H]+: 191.1179; found: 191.1186.

1-methyl-3-(1-phenylethyl)indolin-2-one 3q

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 38% yield. Semi-solid, TLC (PE:EA, 5:1): Rf = 0.32. ¹H NMR (400 MHz, CDCl₃, 8:1 mixture of diastereoisomers): δ (ppm) = 7.37-7.21 (m, 4 H), 7.19-6.80 (m, 5 H), [4.06 (d, J = 6.4 Hz), 3.86 (d, J = 9.2 Hz), 1 H], [3.45 (s), 3.41 (s), 3 H], [3.08-3.00 (m), 2.98-2.88 (m), 1 H], 1.15 (d, J = 7.2 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃, major diastereoisomer): δ (ppm) = 171.2, 140.1, 138.9, 127.8 (2C), 127.7, 127.5, 127.3 (2C), 126.8, 126.1, 121.9, 113.5, 47.9, 41.1, 28.9, 14.5; MS (ESI) m/z: 274.33 [M+Na]+; HRMS (ESI) m/z calcd for C₁₇H₁₈NO [M+H]+: 252.1383; found: 252.1382.
1,3-dimethyl-3-pentylindolin-2-one 4a

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 82% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.52. ^1H NMR (400 MHz, CDCl₃): δ (ppm) = 7.31-7.23 (m, 1 H), 7.19-7.14 (m, 1 H), 7.10-7.02 (m, 1 H), 6.87-6.81 (m, 1 H), 3.21 (s, 3 H), 1.92-1.82 (m, 1 H), 1.77-1.66 (m, 1 H), 1.35 (s, 3 H), 1.23-1.07 (m, 4 H), 1.05-0.81 (m, 2 H), 0.78 (t, J = 6.8 Hz, 3 H); ^13C NMR (100 MHz, CDCl₃): δ (ppm) = 180.9, 143.4, 134.4, 127.6, 122.5, 122.4, 107.8, 48.5, 38.5, 31.9, 26.1, 24.1, 23.8, 22.3, 14.0; MS (ESI) m/z: 254.33 [M+Na]^+; HRMS (ESI) m/z calcd for C₁₅H₂₂NO [M+H]^+: 232.1696; found: 232.1700.

3-hexyl-1,3-dimethylindolin-2-one 4b

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 80% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.50. ^1H NMR (400 MHz, CDCl₃): δ (ppm) = 7.30-7.23 (m, 1 H), 7.19-7.14 (m, 1 H), 7.10-7.03 (m, 1 H), 6.84 (d, J = 7.6 Hz, 1 H), 3.21 (s, 3 H), 1.93-1.83 (m, 1 H), 1.76-1.67 (m, 1 H), 1.34 (s, 3 H), 1.24-0.83 (m, 8 H), 0.81 (t, J = 7.2 Hz, 3 H); ^13C NMR (100 MHz, CDCl₃): δ (ppm) = 180.9, 143.4, 134.4, 127.6, 122.5, 122.4, 107.8, 48.5, 38.6, 31.5, 29.4, 26.1, 24.4, 23.8, 22.6, 14.0; MS (ESI) m/z: 268.42 [M+Na]^+; HRMS (ESI) m/z calcd for C₁₆H₂₄NO [M+H]^+: 246.1852; found: 246.1847.

1,3-dimethyl-3-octylindolin-2-one 4c

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 75% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.51. ^1H NMR (400 MHz, CDCl₃): δ (ppm) = 7.30-7.23 (m, 1 H), 7.19-7.14
(m, 1 H), 7.10-7.03 (m, 1 H), 6.83 (d, J = 7.6 Hz, 1 H), 3.21 (s, 3 H), 1.93-1.83 (m, 1 H), 1.76-1.64 (m, 1 H), 1.34 (s, 3 H), 1.28-1.08 (m, 10 H), 1.04-0.74 (m, 5 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = 180.9, 143.4, 134.4, 127.6, 122.5, 122.4, 107.8, 48.5, 38.6, 31.8, 29.8, 29.3, 29.2, 26.1, 24.5, 23.8, 22.6, 14.1; MS (ESI) m/z: 296.42 [M+Na]$^{+}$; HRMS (ESI) m/z calcd for C$_{18}$H$_{27}$NNaO [M+Na]$^{+}$: 296.1985; found: 296.1989.

1,3-dimethyl-3-(3-methylpentyl)indolin-2-one 4d

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 80% yield. Colorless oil, TLC (PE:EA, 5:1): R$_f$ = 0.51. $^1$H NMR (400 MHz, CDCl$_3$, 1:1 mixture of diastereoisomers): $\delta$ (ppm) = 7.30-7.23 (m, 1 H), 7.19-7.13 (m, 1 H), 7.11-7.04 (m, 1 H), 6.88-6.81 (m, 1 H), 3.22 (s, 3 H), 1.97-1.65 (m, 2 H), 1.35 (m, 3 H), 1.24-0.94 (m, 4 H), 0.88-0.68 (m, 7 H); $^{13}$C NMR (100 MHz, CDCl$_3$, 1:1 mixture of diastereoisomers): $\delta$ (ppm) = 180.9 (2C), 143.4 (2C), 134.4 (2C), 127.5 (2C), 122.4 (2C), 107.8 (2C), 48.4 (2C), 36.0, 35.9, 34.5 (2C), 30.8 (2C), 29.1 (2C), 28.9 (2C), 26.1 (2C), 23.9 (2C), 19.1, 19.0, 11.3, 11.2; MS (ESI) m/z: 268.42 [M+Na]$^{+}$; HRMS (ESI) m/z calcd for C$_{16}$H$_{24}$NO [M+H]$^+$: 246.1852; found: 246.1847.

1,3-dimethyl-3-(3,5,5-trimethylhexyl)indolin-2-one 4e

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 84% yield. Colorless oil, TLC (PE:EA, 5:1): R$_f$ = 0.57. $^1$H NMR (400 MHz, CDCl$_3$, 1:1 mixture of diastereoisomers): $\delta$ (ppm) = 7.30-7.24 (m, 1 H), 7.18-7.14 (m, 1 H), 7.10-7.02 (m, 1 H), 6.83 (d, J = 7.6 Hz, 1 H), 3.21 (s, 3 H, 1:1), 1.96-1.82 (m, 1 H), 1.78-1.64 (m, 1 H), 1.37-1.28 (m, 4 H), 1.13-0.59 (m, 16 H); $^{13}$C NMR (100 MHz, CDCl$_3$, 1:1 mixture of diastereoisomers): $\delta$ (ppm) = 180.9 (2C), 143.4 (2C), 134.4 (2C), 127.5 (2C), 122.4 (2C), 107.8 (2C), 48.4 (2C), 36.0, 35.9, 34.5 (2C), 30.8 (2C), 29.1 (2C), 28.9 (2C), 26.1 (2C), 23.9 (2C), 19.1, 19.0, 11.3, 11.2; MS (ESI) m/z: 268.42 [M+Na]$^{+}$; HRMS (ESI) m/z calcd for C$_{16}$H$_{24}$NO [M+H]$^+$: 246.1852; found: 246.1847.
of diastereoisomers): δ (ppm) = 180.8 (2C), 143.4 (2C), 134.3 (2C), 127.5 (2C), 122.5 (2C), 122.4 (2C), 107.8 (2C), 50.8 (2C), 48.4 (2C), 36.1, 36.0, 33.7, 33.6, 31.0, 30.9, 30.0 (3C), 29.9 (3C), 29.4, 29.3, 26.1 (2C), 23.9 (2C), 22.5, 22.4; MS (ESI) m/z: 310.50 [M+Na]+; HRMS (ESI) m/z calcd for C19H30NO [M+H]+: 288.2322; found: 288.2318.

1,3-dimethyl-3-(3-phenylpropyl)indolin-2-one 4f

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 72% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.41. 1H NMR (400 MHz, CDCl3): δ (ppm) = 7.28-7.18 (m, 3 H), 7.19-7.09 (m, 2 H), 7.09-7.00 (m, 3 H), 6.81 (d, J = 8.0 Hz, 1 H), 3.19 (s, 3 H), 2.58-2.39 (m, 2 H), 2.02-1.91 (m, 1 H), 1.83-1.72 (m, 1 H), 1.38-1.27 (m, 4 H), 1.22-1.08 (m, 1 H); 13C NMR (100 MHz, CDCl3): δ (ppm) = 180.7, 143.3, 141.9, 134.0, 128.4 (2C), 128.3 (2C), 127.7, 125.8, 122.5, 107.9, 48.4, 38.2, 36.0, 26.4, 26.2, 23.9; MS (ESI) m/z: 302.33 [M+Na]+; HRMS (ESI) m/z calcd for C19H30NO [M+Na]+: 302.1515; found: 302.1511.

3-(6-chlorohexyl)-1,3-dimethylindolin-2-one 4g

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 68% yield. Colorless oil. TLC (PE:EA, 5:1): Rf = 0.31. 1H NMR (400 MHz, CDCl3): δ (ppm) = 7.29-7.24 (m, 1 H), 7.19-7.09 (m, 2 H), 7.09-7.00 (m, 1 H), 6.84 (d, J = 7.6 Hz, 1 H), 3.44 (t, J = 6.4 Hz, 2 H), 3.21 (s, 3 H), 1.94-1.85 (m, 1 H), 1.77-1.60 (m, 3 H), 1.34 (s, 3 H), 1.31-1.12 (m, 4 H), 1.05-0.75 (m, 2 H); 13C NMR (100 MHz, CDCl3): δ (ppm) = 179.9, 142.3, 133.2, 126.6, 121.4 (2C), 106.9, 47.4, 44.0, 37.3, 31.4, 27.9, 25.5, 25.1, 23.3, 22.8; MS (ESI) m/z: 302.42 [M+Na]+; HRMS (ESI) m/z calcd for C16H22ClNNO [M+Na]+: 302.1275; found: 302.1282; found: 302.1275.
(Z)-1,3-dimethyl-3-(octadec-9-en-1-yl)indolin-2-one 4h

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 65% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.62. 1H NMR (400 MHz, CDCl3): δ (ppm) = 7.29-7.24 (m, 1 H), 7.20-7.14 (m, 1 H), 7.09-7.04 (m, 1 H), 6.83 (d, J = 7.6 Hz, 1 H), 5.38-5.28 (m, 2 H), 3.21 (s, 3 H), 2.06-1.94 (m, 4 H), 1.92-1.82 (m, 1 H), 1.76-1.66 (m, 1 H), 1.34 (s, 3 H), 1.32-1.10 (m, 22 H), 1.04-0.74 (m, 5 H); 13C NMR (100 MHz, CDCl3): δ (ppm) = 180.9, 143.4 (2C) 129.9 (2C), 127.6, 122.5, 122.4, 107.8, 48.5, 38.6, 31.9, 29.8 (2C), 29.7, 29.5 (2C), 29.3 (3C), 29.2, 27.2 (2C), 26.1, 24.5, 23.8, 22.7, 14.1; MS (ESI) m/z: 434.83 [M+Na]+; HRMS (ESI) m/z calcd for C28H46NO [M+H]+: 412.3574; found: 412.3580.

1,3-dimethyl-3-(3,3,3-trifluoropropyl)indolin-2-one 4i

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 72% yield. Colorless oil. TLC (PE:EA, 5:1): Rf = 0.36. 1H NMR (400 MHz, CDCl3): δ (ppm) = 7.34-7.28 (m, 1 H), 7.21-7.16 (m, 1 H), 7.14-7.08 (m, 1 H), 6.87 (d, J = 8.0 Hz, 1 H), 3.23 (s, 3 H), 2.22-2.12 (m, 1 H), 2.00-1.77 (m, 2 H), 1.73-1.60 (m, 1 H), 1.40 (s, 3 H); 13C NMR (100 MHz, CDCl3): δ (ppm) = 179.4, 143.1, 132.6, 128.4, 126.8 (d, J = 274.4 Hz), 123.0, 122.5, 108.3, 47.0, 30.2 (q, J = 2.7 Hz), 29.3 (q, J = 28.7 Hz), 26.3, 23.6; 19F NMR (377 MHz, CDCl3): δ (ppm) = -66.6. MS (ESI) m/z: 280.33 [M+Na]+; HRMS (ESI) m/z calcd for C13H14F3NNaO [M+Na]+: 280.0920; found: 280.0931.
1,3-dimethyl-3-(4,4,4-trifluorobutyl)indolin-2-one 4j

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 80% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.38. $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.33-7.24 (m, 1 H), 7.20-7.15 (m, 1 H), 7.12-7.05 (m, 1 H), 6.86 (d, J = 7.6 Hz, 1 H), 3.22 (s, 3 H), 2.06-1.74 (m, 4 H), 1.37 (s, 3 H), 1.29-1.08 (m, 2 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 180.2, 143.2, 133.3, 128.0, 126.8 (q, J = 127.2 Hz), 122.7, 122.4, 108.2, 48.1, 37.3, 33.7 (q, J = 29.0), 26.2, 23.9, 17.3 (q, J = 2.8 Hz); $^{19}$F NMR (377 MHz, CDCl$_3$): δ (ppm) = -66.3. MS (ESI) m/z: 294.42 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{14}$H$_{17}$F$_3$NO [M+H]$^+$: 272.1257; found: 272.1260.

3-isobutyl-1,3-dimethylindolin-2-one 4l

The title compound was prepared according to the general method A described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 70% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.48. $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.023-7.17 (m, 1 H), 7.12-7.06 (m, 1 H), 7.03-6.94 (m, 1 H), 6.77 (d, J = 7.6 Hz, 1 H), 3.14 (s, 3 H), 1.92-1.82 (m, 1 H), 1.73-1.64 (m, 1 H), 1.25 (s, 3 H), 1.20-1.12 (m, 1 H), 0.58 (d, J = 6.4 Hz, 3 H), 0.53 (d, J = 6.4 z, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 180.0, 142.2, 133.2, 126.5, 121.8, 121.3, 106.9, 47.1, 45.7, 25.2, 25.1, 24.5, 23.1, 21.8; MS (ESI) m/z: 240.33 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{14}$H$_{20}$NO [M+H]$^+$: 218.1539; found: 218.1538.

3-(2-ethylbutyl)-1,3-dimethylindolin-2-one 4m

The title compound was prepared according to the general method B described above by irradiation with 35 W...
fluorescent light bulb for 36 h, and purified by flash column chromatography in 70% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.54. $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.29-7.22 (m, 1 H), 7.19-7.13 (m, 1 H), 7.09-7.02 (m, 1 H), 6.83 (d, J =7.6 Hz, 1 H), 3.21 (s, 3 H), 1.95-1.87 (m, 1 H), 1.79-1.70 (m, 1 H), 1.34 (s, 3 H), 1.16-0.95 (m, 4 H), 0.88-0.78 (m, 1 H), 0.69 (t, J = 7.2 Hz, 3 H), 0.64 (t, J = 7.2 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 180.0, 142.3, 133.3, 126.5, 121.8, 121.2, 106.8, 47.1, 40.5, 36.1, 25.1, 25.0, 24.6, 24.5, 9.4, 9.3; MS (ESI) m/z: 268.42 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{16}$H$_{24}$NO [M+H]$^+$: 246.1847; found: 246.1847.

1,3-dimethyl-3-(2-propylpentyl)indolin-2-one 4n

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 78% yield. Colorless oil. TLC (PE:EA, 5:1): Rf = 0.63. $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.30-7.24 (m, 1 H), 7.20-7.14 (m, 1 H), 7.08-7.00 (m, 1 H), 6.83 (d, J = 8.0 Hz, 1 H), 3.21 (s, 3 H), 1.96-1.88 (m, 1 H), 1.80-1.71 (m, 1 H), 1.33 (s, 3 H), 1.23-0.83 (m, 9 H), 0.71 (t, J = 7.2 Hz, 3 H), 0.64 (t, J = 7.2 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm) = 181.1, 143.3, 134.3, 127.6, 122.9, 122.2, 107.8, 48.1, 42.5, 36.5, 36.1, 33.9, 26.1, 25.5, 19.1 (2C), 14.3, 14.1; MS (ESI) m/z: 296.50 [M+Na]$^+$; HRMS (ESI) m/z calcd for C$_{18}$H$_{28}$NO [M+H]$^+$: 274.2165; found: 274.2168.

3-(cyclopentylmethyl)-1,3-dimethylindolin-2-one 4o

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 61% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.49. $^1$H NMR (400 MHz, CDCl$_3$): δ (ppm) = 7.30-7.24 (m, 1 H), 7.20-7.14 (m, 1 H), 7.09-7.03 (m, 1 H), 6.84 (d, J = 8.0 Hz, 1 H), 3.22 (s, 3 H), 2.10-2.03 (m, 1 H), 1.93-1.85 (m, 1 H), 1.52-1.38 (m, 3 H), 1.34 (s, 3 H), 1.33-1.19 (m, 4 H), 1.06-0.94 (m, 1 H), 0.88-0.76 (m, 3 H).
3-(cyclohexylmethyl)-1,3-dimethylindolin-2-one 4p

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 66% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.44. ^1H NMR (400 MHz, CDCl3): δ (ppm) = 7.29-7.22 (m, 1 H), 7.18-7.11 (m, 1 H), 7.11-7.02 (m, 1 H), 6.84 (d, J = 8.0 Hz, 1 H), 3.21 (s, 3 H), 1.96-1.89 (m, 1 H), 1.76-1.68 (m, 1 H), 1.54-1.40 (m, 3 H), 1.39-1.16 (m, 5 H), 1.03-0.69 (m, 6 H); ^13C NMR (100 MHz, CDCl3): δ (ppm) = 181.1, 143.1, 134.4, 127.5, 122.7, 122.3, 107.9, 47.8, 45.4, 34.7, 34.5, 33.6, 26.2 (2C), 26.1 (2C), 26.0; MS (ESI) m/z: 280.33 [M+Na]^+; HRMS (ESI) m/z calcd for C_{17}H_{24}NO [M+H]^+: 258.1852; found: 258.1850.

3-((S)-2-chloropropyl)-1,3-dimethylindolin-2-one 4q

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 24 h, and purified by flash column chromatography in 86% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.29. ^1H NMR (400 MHz, CDCl3, 5:2 mixture of diastereoisomers): δ (ppm) = 7.34-7.02 (m, 3 H), 6.90-6.82 (m, 1 H), 3.76-3.59 (m, 1 H), [3.22 (s), 3.21 (s), 3 H], 2.61-2.10 (m, 2 H), [1.38 (s), 1.37 (s), 3 H], [1.33 (d, J = 6.4 Hz), 1.28 (d, J = 6.4 Hz), 3 H]; ^13C NMR (100 MHz, CDCl3, 5:2 mixture of diastereoisomers): δ (ppm) = 180.2, 179.8, 143.7, 143.0, 132.7, 132.4, 128.2, 128.1, 123.1, 122.7, 122.6, 122.3, 122.1, 108.4, 108.3, 54.6, 54.5, 47.6 (2C), 47.4, 47.2, 26.5, 26.3, 26.2, 25.6, 25.5; MS (ESI) m/z: 260.33 [M+Na]^+; HRMS (ESI) m/z calcd for C_{13}H_{17}ClNO [M+H]^+: 238.0993; found: 238.0989.
1,3-dimethyl-3-neopentyldolin-2-one 4s

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 30 h, and purified by flash column chromatography in 87% yield. White solid, m.p. 77-79 °C; TLC (PE:EA, 5:1): Rf = 0.53. \(^1\)H NMR (400 MHz, CDCl\(_3\)): δ (ppm) = 7.32-7.17 (m, 2 H), 7.06-7.00 (m, 1 H), 6.88-6.83 (m, 1 H), 3.22 (s, 3 H), 2.16 (d, \(J = 14.4\) Hz, 1 H), 1.86 (d, \(J = 14.4\) Hz, 1 H), 1.30 (s, 3 H), 0.61 (s, 9 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): δ (ppm) = 181.0, 142.9, 134.2, 127.5, 123.9, 122.0, 108.0, 50.8, 47.4, 31.8 (3C), 30.8, 28.3, 26.3. MS (ESI) \(m/z\): 254.42 [M+Na\]^+; HRMS (ESI) \(m/z\) calcd for C\(_{15}\)H\(_{22}\)NO [M+Na\]^+: 232.1700; found: 232.1700.

3-(-adamantan-1-ylmethyl)-1,3-dimethylindolin-2-one 4t

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 30 h, and purified by flash column chromatography in 88% yield. White solid, m.p. 107-109 °C. TLC (PE:EA, 5:1): Rf = 0.51. \(^1\)H NMR (400 MHz, CDCl\(_3\)): δ (ppm) = 7.29-7.16 (m, 2 H), 7.07-7.00 (m, 1 H), 6.85 (d, \(J = 7.6\) Hz, 1 H), 3.23 (s, 3 H), 2.00 (d, \(J = 14.4\) Hz, 1 H), 1.77-1.68 (m, 4 H), 1.55-1.47 (m, 3 H), 1.42-1.34 (m, 3 H), 1.27 (s, 3 H), 1.23-1.12 (m, 6 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): δ (ppm) = 181.2, 142.7, 134.7, 127.5, 123.6, 122.0, 108.0, 52.1, 46.7, 43.4 (3C), 36.7 (3C), 33.9, 28.6 (4C), 26.3; MS (ESI) \(m/z\): 332.50 [M+Na\]^+; HRMS (ESI) \(m/z\) calcd for C\(_{21}\)H\(_{27}\)NNaO [M+Na\]^+: 332.1985; found: 332.1986.

3-(2,2-dimethylbutyl)-1,3-dimethylindolin-2-one 4u

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h, and purified by flash column chromatography in 71% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.49. \(^1\)H NMR (400 MHz, CDCl\(_3\)): δ (ppm) = 7.29-7.18 (m, 2 H), 7.06-7.01 (m,
(3R,8R,9S,10S,13R,14S,17R)-17-((2R)-5-(1,3-dimethyl-2-oxoindolin-3-yl)pentan-2-yl)-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-3-yl acetate 4v

The title compound was prepared according to the general method B described above by irradiation with 35 W fluorescent light bulb for 36 h with a mixture solvent (DMF:DCM=2:1), and purified by flash column chromatography in 52% yield. Colorless oil, TLC (PE:EA, 5:1): Rf = 0.43. ¹H NMR (400 MHz, CDCl₃, 1:1 mixture of diastereoisomers): δ (ppm) = 7.29-7.23 (m, 1 H), 7.20-7.14 (m, 1 H), 7.10-7.03 (m, 1 H), 6.84 (d, J = 7.6 Hz, 1 H), 4.77-4.66 (m, 1 H), 3.22 (s, 3 H), 1.95-0.68 (m, 39 H), 0.58 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, 1:1 mixture of diastereoisomers): δ (ppm) = 180.9 (2C), 170.6, 143.3, 134.4, 127.6, 122.5, 122.4, 107.8, 74.4, 56.5 (2C), 48.5 (2C), 42.7, 41.9, 40.4, 40.1, 38.8 (2C), 36.0, 35.9, 35.8, 35.5 (2C), 35.0, 34.6, 32.3, 28.2, 27.0, 26.6, 26.3, 26.1, 24.2, 23.9, 23.3, 21.5, 21.2 (2C), 20.8, 18.5 (2C), 12.0; MS (ESI) m/z: 570.40 [M+Na]⁺; HRMS (ESI) m/z calcd for C₃₆H₅₃NNaO₃ [M+Na]⁺: 570.3918; found: 570.3927.
References:


Copies of the $^1$H, $^{13}$C and $^{19}$F NMR and MS spectra

The $^1$H NMR spectrum of 3a

The $^{13}$C NMR spectrum of 3a
The $^1$H NMR spectrum of 3b

The $^{13}$C NMR spectrum of 3b
The $^1$H NMR spectrum of 3c

The $^{13}$C NMR spectrum of 3c
The $^1$H NMR spectrum of 3d

The $^{13}$C NMR spectrum of 3d
The $^1$H NMR spectrum of 3e

The $^{13}$C NMR spectrum of 3e
The $^1$H NMR spectrum of 3f

The $^{13}$C NMR spectrum of 3f
The $^1$H NMR spectrum of 3g

The $^{13}$C NMR spectrum of 3g
The $^1$H NMR spectrum of 3h

The $^{13}$C NMR spectrum of 3h
The $^1$H NMR spectrum of $3i$

![The $^1$H NMR spectrum of $3i$](image)

The $^{13}$C NMR spectrum of $3i$

![The $^{13}$C NMR spectrum of $3i$](image)
The $^1$H NMR spectrum of 3k

The $^{13}$C NMR spectrum of 3k
The $^1$H NMR spectrum of 3l

The $^{13}$C NMR spectrum of 3l
The $^1$H NMR spectrum of 3m

The $^{13}$C NMR spectrum of 3m
The $^1$H NMR spectrum of 3n

![The $^1$H NMR spectrum of 3n](image1)

The $^{13}$C NMR spectrum of 3n

![The $^{13}$C NMR spectrum of 3n](image2)
The $^1$H NMR spectrum of 3o

The $^{13}$C NMR spectrum of 3o
The $^{13}$C NMR spectrum of 3p
The $^1$H NMR spectrum of 3q

The $^{13}$C NMR spectrum of 3q
The $^1$H NMR spectrum of 4a

The $^{13}$C NMR spectrum of 4a
The $^1$H NMR spectrum of 4b

The $^{13}$C NMR spectrum of 4b
The $^1$H NMR spectrum of 4c

The $^{13}$C NMR spectrum of 4c
The $^1$H NMR spectrum of 4d

The $^{13}$C NMR spectrum of 4d
The $^1$H NMR spectrum of 4e

The $^{13}$C NMR spectrum of 4e
The $^1$H NMR spectrum of 4f

The $^{13}$C NMR spectrum of 4f
The $^1$H NMR spectrum of 4g

The $^{13}$C NMR spectrum of 4g
The $^1$H NMR spectrum of 4h

The $^{13}$C NMR spectrum of 4h
The $^1$H NMR spectrum of 4i

The $^{13}$C NMR spectrum of 4i

The $^{19}$F NMR spectrum of 4i
The $^1$H NMR spectrum of 4j

The $^{13}$C NMR spectrum of 4j

The $^{19}$F NMR spectrum of 4j
The $^1$H NMR spectrum of 4l

The $^{13}$C NMR spectrum of 4l
The $^1$H NMR spectrum of 4m

The $^{13}$C NMR spectrum of 4m
The $^1$H NMR spectrum of 4n

The $^{13}$C NMR spectrum of 4n
The $^1$H NMR spectrum of 4o

The $^{13}$C NMR spectrum of 4o
The $^1$H NMR spectrum of 4p

The $^{13}$C NMR spectrum of 4p
The $^1$H NMR spectrum of 4q

The $^{13}$C NMR spectrum of 4q
The $^1$H NMR spectrum of 4s

![NMR spectrum of 4s](image1)

The $^{13}$C NMR spectrum of 4s

![NMR spectrum of 4s](image2)
The $^1$H NMR spectrum of 4t

The $^{13}$C NMR spectrum of 4t
The $^1$H NMR spectrum of 4u

The $^{13}$C NMR spectrum of 4u
The $^1$H NMR spectrum of 4v

The $^{13}$C NMR spectrum of 4v
The MS(ESI) spectrum of 3e

The MS(ESI) spectrum of 3m
The MS(ESI) spectrum of 4f

The MS(ESI) spectrum of 4v