

***N*-(Acyloxy)phthalimides as tertiary alkyl radical  
precursors in the visible light photocatalyzed tandem  
radical cyclization of *N*-arylacrylamides to 3,3-dialkyl  
substituted oxindoles**

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**General:**

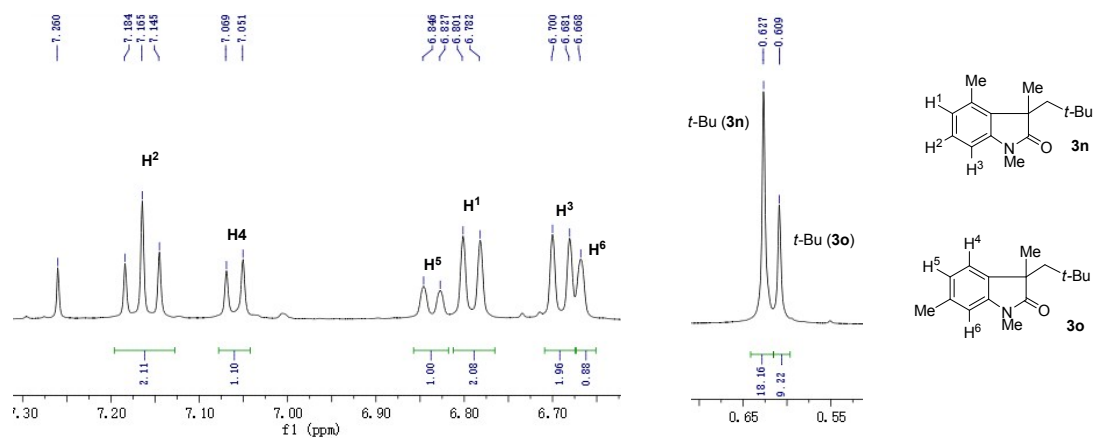
Column chromatography silica gel (200-300 mesh) and TLC plate were purchased from Qingdao Meijin Chemical Inc(Qingdao; China); HRMS data were obtained in the ESI mode on a Agilent 6530 Q-TOF/MS system; <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on Bruker 400 MHz spectrometer and chemical shifts were given in  $\delta$  with TMS as an internal reference.

***Representative experimental procedure for visible light promoted tandem radical cyclization of N-arylacrylamides***

A solution of *N*-arylacrylamides **1** (0.4 mmol), 3.0 eq of *N*-(acyloxy)phthalimides **2**, Ru(bpy)<sub>3</sub>Cl<sub>2</sub>·6H<sub>2</sub>O (2 mol%) and 3.0 eq of *i*-Pr<sub>2</sub>NEt in MeCN (4 mL) was irradiated with 25W compact fluorescent lamp for 24 h at room temperature. After the reaction was completed, the resulting mixture was poured into water (50 mL) and then extracted with EtOAc (20 mL×3). The combined organic solution was then washed with water (20 mL×3). The organic layers were washed with brine and dried over MgSO<sub>4</sub>. The solvent were removed via vacuo and the residue was purified by flash column chromatography (SiO<sub>2</sub>) with petroleum ether/acetone (30:1) to give target compounds **3** and **4**.

### <sup>1</sup>H NMR analysis of the mixture of **3n** and **3o**

According to NMR analysis, the tandem cyclization of *meta*-methyl substituted *N*-arylacrylamide with *N*-(pivaloyloxy)phthalimide (**2a**) give **3n** and **3o** as a mixture in ratio of 2:1.



**Figure 1s** Part of <sup>1</sup>H NMR spectra of the mixture of **3n** and **3o** (solvent: CDCl<sub>3</sub>)

## HPLC-Q-TOF and $^1\text{H}$ NMR analysis of the mixture of **3p** and **3q**

According to HPLC-Q-TOF and NMR analysis, the tandem cyclization of *meta*-chloro substituted *N*-arylacrylamide with *N*-(pivaloyloxy)phthalimide (**2a**) give **3q** and **3p** as a mixture in ratio of about 13:1.

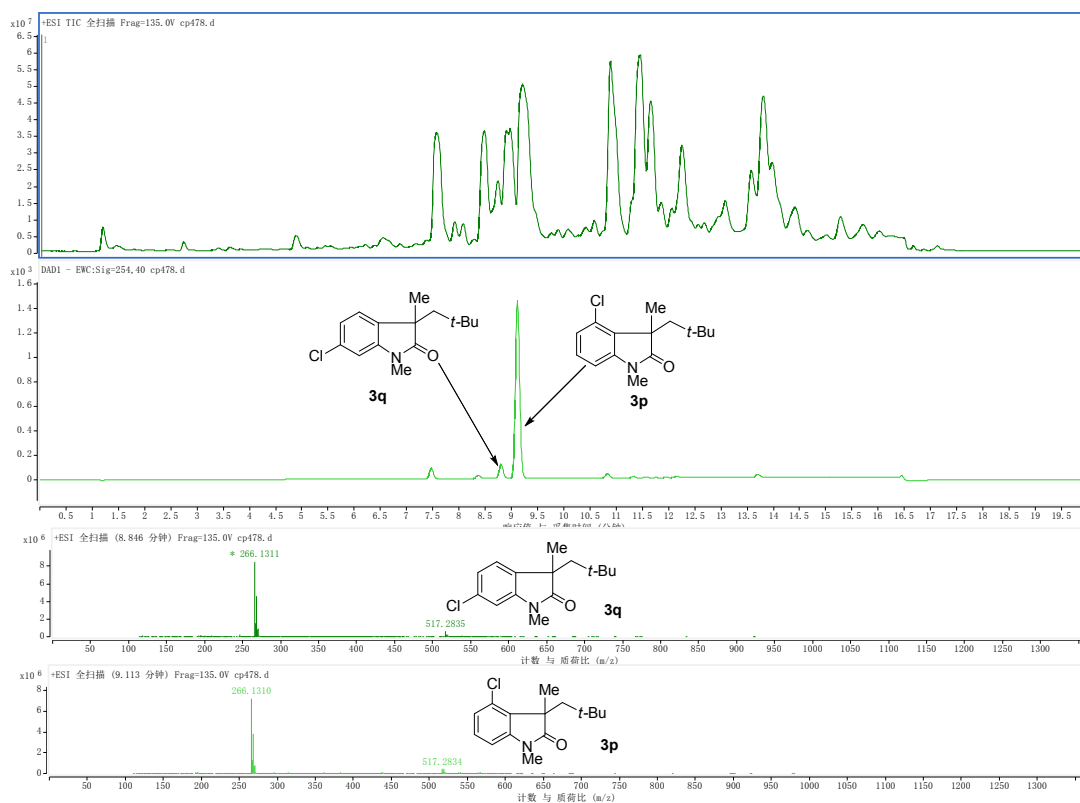


Figure 2s HPLC-Q-Tof analysis of the mixture of **3p** and **3q**

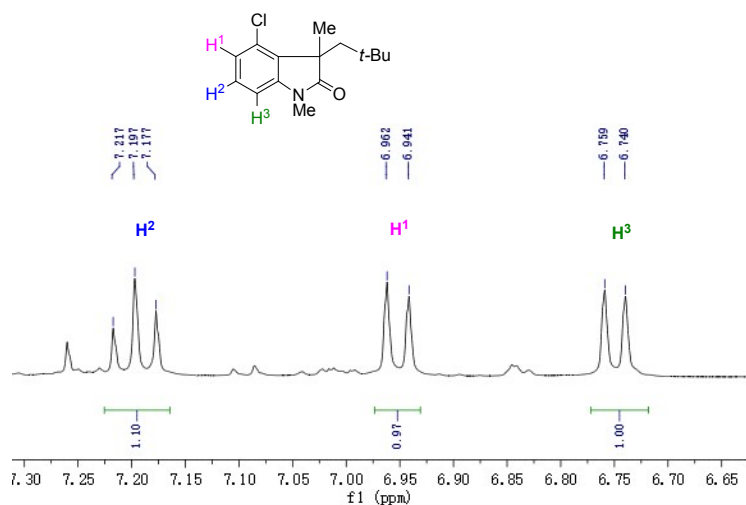
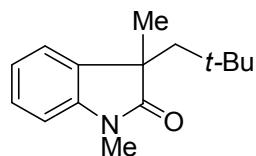


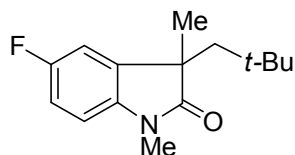
Figure 3s Low field part of  $^1\text{H}$  NMR spectra of the mixture of **3p** and **3q** (solvent:  $\text{CDCl}_3$ )

### Characterization data of compounds 3 and 4



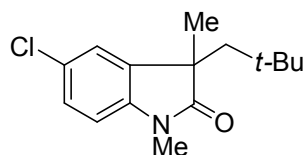
#### **1,3-dimethyl-3-neopentylindolin-2-one (3a) :**

Obtained as colorless oil,  $^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.29-7.19 (m, 2H), 7.03 (t,  $J = 8.8$  Hz, 1H), 6.85 (d,  $J = 7.6$  Hz, 1H), 3.22 (s, 3H), 2.16 (d,  $J = 14.4$  Hz, 1H), 1.86 (d,  $J = 14.4$  Hz, 1H) 1.30 (s, 3H), 0.62 (s, 9H).  $^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ ):  $\delta$  181.2, 142.9, 134.7, 127.6, 123.9, 122.1, 108.1, 50.9, 47.5, 31.8, 30.9( $\times 3$ ), 28.3, 27.1. HRMS (ESI $^+$ ): calcd 232.1696 for  $\text{C}_{15}\text{H}_{22}\text{NO}^+$  [M+H] $^+$ ; found, 232.1707.



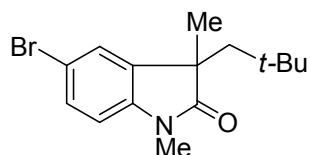
#### **5-fluoro-1,3-dimethyl-3-neopentylindolin-2-one (3b):**

Obtained as white wax,  $^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ ):  $\delta$  6.96-6.91 (m, 2H), 6.75-6.72 (m, 1H), 3.18 (s, 3H), 2.14 (d,  $J = 14.4$  Hz, 1H), 1.80 (d,  $J = 14.4$  Hz, 1H), 1.26 (s, 3H), 0.60 (s, 9H).  $^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.4, 158.9 (d,  $^1J_{\text{F-C}} = 238$  Hz), 138.6, 135.8 (d,  $^3J_{\text{F-C}} = 8.0$  Hz), 113.5 (d,  $^2J_{\text{F-C}} = 23.4$  Hz), 111.6 (d,  $^2J_{\text{F-C}} = 24.2$  Hz), 108.2 (d,  $^3J_{\text{F-C}} = 8.1$  Hz), 50.6, 47.7, 31.6, 30.6( $\times 3$ ), 28.0, 26.1. HRMS (ESI $^+$ ): calcd 250.1602 for  $\text{C}_{15}\text{H}_{21}\text{FNO}^+$  [M+H] $^+$ ; found, 250.1608.



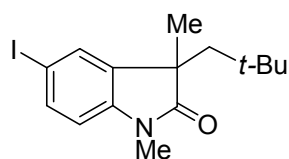
#### **5-chloro-1,3-dimethyl-3-neopentylindolin-2-one (3c):**

Obtained as white wax,  $^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.18 (dd,  $J = 8.4, 2.0$  Hz, 1H), 7.12 (d,  $J = 2.0$  Hz, 1H), 6.72 (d,  $J = 8.0$  Hz, 1H), 3.15 (s, 3H), 2.11 (d,  $J = 14.4$  Hz, 1H), 1.78 (d,  $J = 14.4$  Hz, 1H), 1.23 (s, 3H), 0.57 (s, 9H).  $^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.4, 141.5( $\times 2$ ), 136.1, 127.5, 124.3, 109.0, 50.8, 47.7, 31.8, 30.9( $\times 3$ ), 28.2, 26.4. HRMS (ESI $^+$ ): calcd 266.1306 for  $\text{C}_{15}\text{H}_{21}\text{ClNO}^+$  [M+H] $^+$ ; found, 266.1310.



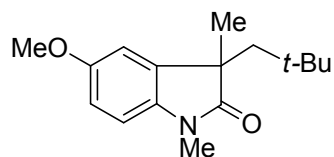
#### **5-bromo-1,3-dimethyl-3-neopentylindolin-2-one (3d):**

Obtained as white wax,  $^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.34-7.32 (m, 1H), 7.27-7.26 (m, 1H), 6.68 (dd,  $J = 8.0, 2.4$  Hz, 1H), 3.15 (s, 3H), 2.10 (d,  $J = 14.4$  Hz, 1H), 1.78 (d,  $J = 14.4$  Hz, 1H), 1.24 (s, 3H), 0.58 (s, 9H).  $^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.4, 142.0, 136.5, 130.4, 127.1, 114.8, 109.5, 50.9, 47.7, 31.8, 30.9( $\times 3$ ), 28.2, 26.4. HRMS (ESI $^+$ ): calcd 310.0801 for  $\text{C}_{15}\text{H}_{21}\text{BrNO}^+$  [M+H] $^+$ ; found, 310.0798.



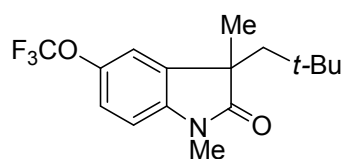
**5-iodo-1,3-dimethyl-3-neopentylindolin-2-one (3e):**

Obtained as white wax,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.53 (dd,  $J = 8.0, 1.6$  Hz, 1H), 7.44 (s, 1H), 6.60 (d,  $J = 8.4$  Hz), 3.15 (s, 3H), 2.09 (d,  $J = 14.4$  Hz, 1H), 1.78 (d,  $J = 14.4$  Hz, 1H), 1.24 (s, 3H), 0.58 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.1, 142.6, 136.9, 136.4, 132.7, 110.2, 84.6, 50.8, 47.5, 31.8, 30.9( $\times 3$ ), 28.2, 26.3. HRMS (ESI $^+$ ): calcd 358.0662 for  $\text{C}_{15}\text{H}_{21}\text{INO}^+$  [M+H] $^+$ ; found, 358.0656.



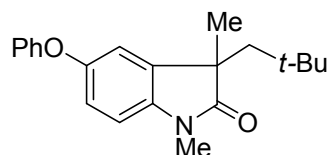
**5-methoxy-1,3-dimethyl-3-neopentylindolin-2-one (3f):**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  6.81-6.72 (m, 3H), 3.78 (s, 3H), 3.18 (s, 3H), 2.14 (d,  $J = 14.4$  Hz, 1H), 1.71 (d,  $J = 14.4$  Hz, 1H), 1.27 (s, 3H), 0.61 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.8, 155.8, 136.7, 135.8, 111.9, 111.7, 108.3, 56.0, 50.9, 48.0, 31.9, 30.9( $\times 3$ ), 28.4, 26.4. HRMS (ESI $^+$ ): calcd 262.1802 for  $\text{C}_{16}\text{H}_{24}\text{NO}_2^+$  [M+H] $^+$ ; found, 262.1794.



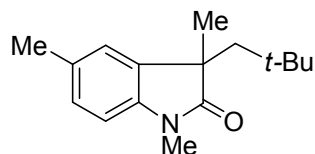
**1,3-dimethyl-3-neopentyl-5-(trifluoromethoxy)indolin-2-one (3g):**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.12 (d,  $J = 8.4$  Hz, 1H), 7.08 (s, 1H), 6.82 (d,  $J = 8.4$  Hz, 1H), 3.24 (s, 3H), 2.16 (d,  $J = 14.4$  Hz, 1H), 1.84 (d,  $J = 14.4$  Hz, 1H), 1.29 (s, 3H), 0.60 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.9, 144.6, 141.7, 136.0, 120.8 (q,  $^1J_{\text{F-C}} = 254.5$  Hz), 120.9, 118.1, 108.5, 51.0, 47.9, 31.9, 30.9( $\times 3$ ), 28.2, 26.5. HRMS (ESI $^+$ ): calcd 316.1519 for  $\text{C}_{16}\text{H}_{21}\text{F}_3\text{NO}_2^+$  [M+H] $^+$ ; found, 316.1518.



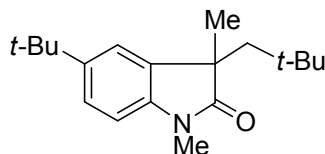
**1,3-dimethyl-3-neopentyl-5-phenoxyindolin-2-one (3h):**

Obtained as white wax,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.26 (dt,  $J = 7.6, 2.0$  Hz, 2H), 7.03 (t,  $J = 8.8$  Hz, 1H), 6.97-6.92 (m, 3H), 6.90 (s, 1H), 6.80 (d,  $J = 8.0$  Hz, 1H), 3.21 (s, 3H), 2.14 (d,  $J = 14.4$  Hz, 1H), 1.78 (d,  $J = 14.4$  Hz, 1H), 1.27 (s, 3H), 0.64 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.8, 158.6, 152.1, 139.0, 136.0, 129.7 ( $\times 2$ ), 122.6, 119.0, 117.6 ( $\times 2$ ), 116.6, 108.7, 50.8, 47.8, 31.8, 30.9( $\times 3$ ), 28.3, 26.4. HRMS (ESI $^+$ ): calcd 324.1958 for  $\text{C}_{21}\text{H}_{26}\text{NO}_2^+$  [M+H] $^+$ ; found, 324.1951.



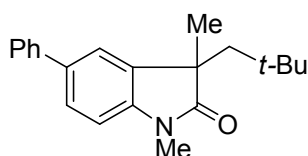
**1,3,5-trimethyl-3-neopentylindolin-2-one (3i):**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.40 (d,  $J = 7.6$  Hz, 1H), 7.00 (s, 1H), 6.72 (d,  $J = 8.0$  Hz, 1H), 3.18 (s, 3H), 2.32 (s, 3H), 2.13 (d,  $J = 14.4$  Hz, 1H), 1.82 (d,  $J = 14.4$  Hz, 1H), 1.27 (s, 3H), 0.60 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  181.2, 140.6, 134.4, 131.5, 127.8, 124.8, 107.8, 50.9, 47.6, 31.9, 30.9( $\times 3$ ), 28.4, 26.4, 21.2. HRMS (ESI $^+$ ): calcd 246.1852 for  $\text{C}_{16}\text{H}_{24}\text{NO}^+$  [M+H] $^+$ ; found, 246.1858.



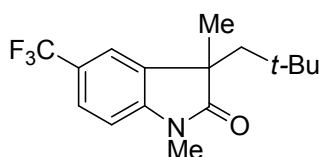
**5-(tert-butyl)-1,3-dimethyl-3-neopentylindolin-2-one (3J):**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.27 (dd,  $J = 8.0, 2.0$  Hz, 1H), 7.24 (d,  $J = 2.0$  Hz, 1H), 6.77 (d,  $J = 8.0$  Hz, 1H), 3.20 (s, 3H), 2.16 (d,  $J = 14.4$  Hz, 1H), 1.86 (d,  $J = 14.4$  Hz, 1H), 1.32 (s, 9H), 1.30 (s, 3H), 0.60 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  181.4, 145.2, 140.6, 133.8, 123.9, 121.5, 107.4, 50.9, 47.6, 34.6, 31.8, 31.6( $\times 3$ ), 30.9( $\times 3$ ), 28.4, 26.3. HRMS (ESI $^+$ ): calcd 288.2322 for  $\text{C}_{19}\text{H}_{30}\text{NO}^+$  [M+H] $^+$ ; found, 288.2319.



**1,3-dimethyl-3-neopentyl-5-phenylindolin-2-one (3k):**

Obtained as white wax,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.60 (d,  $J = 8.0$  Hz, 2H), 7.49 (d,  $J = 8.0$  Hz, 1H), 7.45-7.41 (m, 3H), 7.32 (t,  $J = 8.8$  Hz, 1H), 6.91 (d,  $J = 8.0$  Hz, 1H), 3.51 (s, 3H), 2.21 (d,  $J = 14.4$  Hz, 1H), 1.93 (d,  $J = 14.4$  Hz, 1H), 1.35 (s, 3H), 0.67 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  181.1, 142.4, 141.2, 135.5, 134.8, 128.8( $\times 2$ ), 126.9, 126.8( $\times 2$ ), 126.5, 122.9, 108.3, 50.9, 47.6, 31.9, 30.9( $\times 3$ ), 28.4, 26.4. HRMS (ESI $^+$ ): calcd 308.2009 for  $\text{C}_{21}\text{H}_{26}\text{NO}^+$  [M+H] $^+$ ; found, 308.2005.

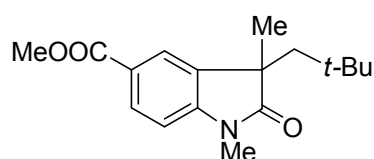


**1,3-dimethyl-3-neopentyl-5-(trifluoromethyl)indolin-2-one (3l):**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.53 (d,  $J = 8.0$  Hz, 1H), 7.40 (s, 1H), 6.90 (d,  $J = 8.0$  Hz, 1H), 3.22 (s, 3H), 2.16 (d,  $J = 14.4$  Hz, 1H), 1.86 (d,  $J = 14.4$  Hz, 1H), 1.29 (s, 3H), 0.57 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.8, 145.6, 134.7, 125.1 (q,  $^3J_{\text{F-C}} = 3.9$  Hz), 124.7 (q,  $^1J_{\text{F-C}} = 278$  Hz),

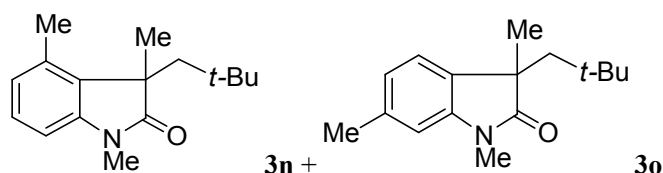


124.1 (q,  $^2J_{F-C} = 32.5$  Hz), 120.6 (q,  $^3J_{F-C} = 3.4$  Hz), 107.6, 50.6, 47.2, 31.5, 30.6( $\times 3$ ), 27.9, 26.4.  
 HRMS (ESI<sup>+</sup>): calcd 300.1570 for C<sub>16</sub>H<sub>21</sub>F<sub>3</sub>NO<sup>+</sup> [M+H]<sup>+</sup>; found, 300.1584.

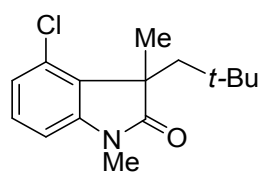


**methyl 1,3-dimethyl-3-neopentyl-2-oxindoline-5-carboxylate (3m):**

Obtained as colorless oil, <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>):  $\delta$  7.95 (dd,  $J = 8.0, 2.0$  Hz, 1H), 7.81 (d,  $J = 2.0$  Hz, 1H), 6.83 (d,  $J = 8.0$  Hz, 1H), 3.84 (s, 3H), 3.19 (s, 3H), 2.19 (d,  $J = 14.4$  Hz, 1H), 1.85 (d,  $J = 14.4$  Hz, 1H), 1.24 (s, 3H), 0.53 (s, 9H). <sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>):  $\delta$  181.2, 167.0, 147.0, 134.3, 130.4, 125.0, 124.0, 107.6, 52.0, 50.8, 47.2, 31.8, 30.8( $\times 3$ ), 28.1, 26.4. HRMS (ESI<sup>+</sup>): calcd 290.1751 for C<sub>17</sub>H<sub>24</sub>NO<sub>3</sub><sup>+</sup> [M+H]<sup>+</sup>; found, 290.1760.



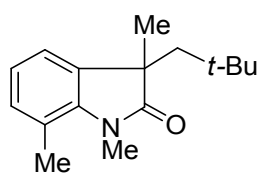
**1,3,4-trimethyl-3-neopentylindolin-2-one (3n)** and **1,3,6-trimethyl-3-neopentylindolin-2-one (3o)** were obtained as colorless oil mixture. <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>):  $\delta$  7.17 (t,  $J = 7.6$  Hz, 1H) (**3n**), 7.06 (d,  $J = 7.6$  Hz, 1H) (**3o**), 6.84 (d,  $J = 7.6$  Hz, 1H) (**3o**), 6.79 (d,  $J = 7.6$  Hz, 1H) (**3o**), 6.69 (d,  $J = 7.6$  Hz, 1H) (**3n**), 6.67 (s, 1H) (**3o**), 3.91 (s, NMe, overlapped), 2.39 (s, 3H) (**3o**), 2.38 (s, 3H) (**3n**), 2.13 (d,  $J = 14.4$  Hz, 1H) (**3o**), 2.08 (d,  $J = 2.0$  Hz, 2H) (**3n**), 1.82 (d,  $J = 14.4$  Hz, 1H) (**3o**), 1.36 (s, 3H) (**3o**), 1.26 (s, 3H) (**3n**), 0.63 (s, 9H) (**3n**), 0.61 (s, 9H) (**3o**). <sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>):  $\delta$  181.9(**3o**), 181.5(**3n**), 143.6, 143.4, 138.0, 135.4, 131.7, 127.9, 125.4, 124.1, 123.0, 109.4, 106.2, 51.2, 49.6, 48.7, 32.2, 31.3, 30.5 (overlapped), 28.8, 26.8, 26.6, 25.7, 22.2(**3o**), 19.2(**3n**). HRMS (ESI<sup>+</sup>): calcd 246.1852 for C<sub>16</sub>H<sub>24</sub>NO<sup>+</sup> [M+H]<sup>+</sup>; found, 246.1859.



**4-chloro-1,3-dimethyl-3-neopentylindolin-2-one (3p):**

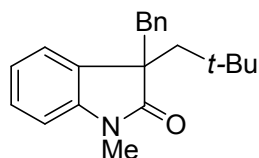
Obtained as colorless oil, <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>):  $\delta$  7.20 (t,  $J = 8.0$  Hz, 1H), 6.95 (d,  $J = 8.0$  Hz, 1H), 6.75 (d,  $J = 8.0$  Hz, 1H), 3.20 (s, 3H), 2.36 (d,  $J = 14.4$  Hz, 1H), 2.20 (d,  $J = 14.4$  Hz, 1H), 1.43 (s, 3H), 0.65 (s, 9H). <sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>):  $\delta$  108.2, 144.5, 131.3, 130.4, 128.6, 123.4, 106.3, 48.6, 48.0, 31.5, 29.8( $\times 3$ ), 26.3, 24.2. HRMS (ESI<sup>+</sup>): calcd 266.1306 for C<sub>15</sub>H<sub>21</sub>ClNO<sup>+</sup> [M+H]<sup>+</sup>; found, 266.1306.

266.1310.



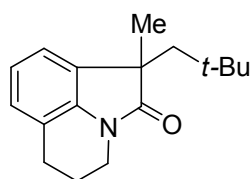
**1,3,7-trimethyl-3-neopentylindolin-2-one (3r):**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.01 (d,  $J = 7.2$  Hz, 1H), 6.97 (d,  $J = 7.2$  Hz, 1H), 6.90 (t,  $J = 7.2$  Hz, 1H), 3.50 (s, 3H), 2.59 (s, 3H), 2.13 (d,  $J = 14.4$  Hz, 1H), 1.82 (d,  $J = 14.4$  Hz, 1H), 1.27 (s, 3H), 0.61 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  182.0, 140.8, 135.0, 131.4, 122.0( $\times 2$ ), 119.7, 51.2, 46.9, 31.9, 30.9( $\times 3$ ), 29.7, 28.8. HRMS (ESI $^+$ ): calcd 246.1852 for  $\text{C}_{16}\text{H}_{24}\text{NO}^+$  [M+H] $^+$ ; found, 246.1862.



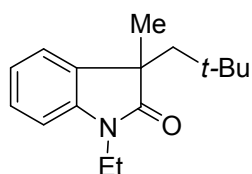
**3-benzyl-1-methyl-3-neopentylindolin-2-one (3u):**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.21 (d,  $J = 7.2$  Hz, 1H), 7.15 (td,  $J = 7.6, 1.2$  Hz, 1H), 7.03-6.95 (m, 4H), 6.72 (dd,  $J = 7.2, 1.6$  Hz, 2H), 6.53 (d,  $J = 8.0$  Hz, 1H), 3.05 (d,  $J = 12.4$  Hz, 1H), 2.92 (d,  $J = 12.4$  Hz, 1H), 2.89 (s, 3H), 2.30 (d,  $J = 14.4$  Hz, 1H), 2.00 (d,  $J = 14.4$  Hz, 1H), 0.66 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  179.4, 143.6, 135.3, 131.3, 130.0( $\times 2$ ), 127.8, 127.3( $\times 2$ ), 126.5, 125.1, 121.5, 107.8, 53.8, 49.6, 47.7, 32.0, 31.3( $\times 3$ ), 25.9. HRMS (ESI $^+$ ): calcd 308.2009 for  $\text{C}_{21}\text{H}_{26}\text{NO}^+$  [M+H] $^+$ ; found, 308.2015.



**1-methyl-1-neopentyl-5,6-dihydro-1H-pyrrolo[3,2,1-i]quinolin-2(4H)-one (3v):**

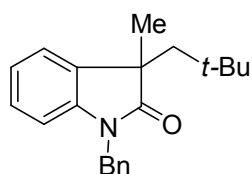
Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.02 (d,  $J = 7.6$  Hz, 1H), 7.00 (d,  $J = 7.6$  Hz, 1H), 6.91 (t,  $J = 7.6$  Hz, 1H), 3.71 (q,  $J = 4.8$  Hz, 2H), 2.79 (q,  $J = 5.6$  Hz, 2H), 2.14 (d,  $J = 14.4$  Hz, 1H), 2.02-1.99 (m, 2H), 1.84 (d,  $J = 14.4$  Hz, 1H), 1.30 (s, 3H), 0.64 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.1, 138.8, 132.9, 126.4, 122.0, 121.6, 120.2, 50.8, 48.9, 39.1, 31.9, 31.0( $\times 3$ ), 28.2, 24.9, 21.4. HRMS (ESI $^+$ ): calcd 258.1852 for  $\text{C}_{17}\text{H}_{24}\text{NO}^+$  [M+H] $^+$ ; found, 258.1849.



**1-ethyl-3-methyl-3-neopentylindolin-2-one (3y):**

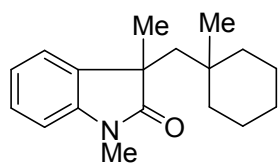
Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.23 (dd,  $J = 7.6, 0.8$  Hz, 1H), 7.20 (d,  $J = 7.2$  Hz, 1H), 7.01 (dt,  $J = 7.6, 0.8$  Hz,

1H), 6.86 (d,  $J = 7.6$  Hz, 1H), 3.89-3.66 (m, 2H), 2.16 (d,  $J = 14.4$  Hz, 1H), 1.86 (d,  $J = 14.4$  Hz, 1H), 1.28 (s, 3H), 1.26 (q,  $J = 7.2$  Hz, 3H), 0.63 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.7, 142.1, 134.6, 127.6, 124.2, 121.8, 108.3, 50.7, 47.6, 34.7, 32.0, 31.0, 28.8, 12.4. HRMS (ESI<sup>+</sup>): calcd 246.1852 for  $\text{C}_{16}\text{H}_{24}\text{NO}^+$  [M+H]<sup>+</sup>; found, 246.1849.



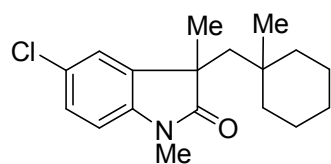
**1-benzyl-3-methyl-3-neopentylindolin-2-one (3z)**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.32-7.20 (m, 7H), 7.00 (d,  $J = 7.6$  Hz, 1H), 6.78 (d,  $J = 8.0$  Hz, 1H), 5.06 (d,  $J = 15.6$  Hz, 1H), 4.08 (d,  $J = 15.6$  Hz, 1H), 2.22 (d,  $J = 14.4$  Hz, 1H), 1.91 (d,  $J = 14.4$  Hz, 1H), 1.35 (s, 3H), 0.65 (s, 9H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  181.2, 142.2, 136.2, 134.3, 128.8 ( $\times 2$ ), 127.7 ( $\times 2$ ), 127.6, 127.5, 124.1, 122.1, 109.2, 50.6, 47.6, 44.0, 32.0, 31.0, 29.2. HRMS (ESI<sup>+</sup>): calcd 308.2009 for  $\text{C}_{21}\text{H}_{26}\text{NO}^+$  [M+H]<sup>+</sup>; found, 308.2013.



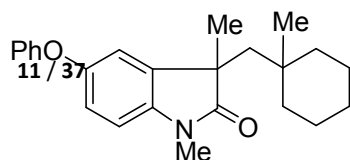
**1,3-dimethyl-3-((1-methylcyclohexyl)methyl)indolin-2-one (4a):**

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.28-7.21 (m, 2H), 7.03 (t,  $J = 7.2$  Hz, 1H), 6.85 (d,  $J = 7.2$  Hz, 1H), 3.22 (s, 3H), 2.12 (d,  $J = 14.4$  Hz, 1H), 1.93 (d,  $J = 14.4$  Hz, 1H), 1.30 (s, 3H), 1.34-0.90 (m, 10H), 0.51 (s, 3H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  181.3, 142.8, 134.6, 127.6, 123.9, 122.0, 108.1, 50.7, 47.2, 39.3, 39.1, 34.3, 28.7, 26.3 ( $\times 2$ ), 24.3, 22.0, 21.9. HRMS (ESI<sup>+</sup>): calcd 272.2009 for  $\text{C}_{18}\text{H}_{26}\text{NO}^+$  [M+H]<sup>+</sup>; found, 272.2010.



**5-chloro-1,3-dimethyl-3-((1-methylcyclohexyl)methyl)indolin-2-one (4b):**

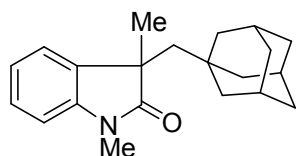
Obtained as white wax,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.31 (d,  $J = 8.0$  Hz, 1H), 7.26 (s, 1H), 6.84 (dd,  $J = 8.0, 2.0$  Hz, 1H), 3.28 (brs, 3H), 2.20 (brd,  $J = 14.4$  Hz, 1H), 1.95 (brd,  $J = 14.4$  Hz, 1H), 1.46-0.98 (m, 10H), 1.36 (brs, 3H), 0.59 (brs, 3H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.4, 141.1, 136.2, 127.2 ( $\times 2$ ), 124.0, 108.7, 50.5, 47.1, 39.1, 38.7, 34.0, 28.3, 26.2, 26.0, 24.1, 21.7, 21.6. HRMS (ESI<sup>+</sup>): calcd 306.1619 for  $\text{C}_{18}\text{H}_{25}\text{ClNO}^+$  [M+H]<sup>+</sup>; found, 306.1621.



**1,3-dimethyl-3-((1-methylcyclohexyl)methyl)-5-**

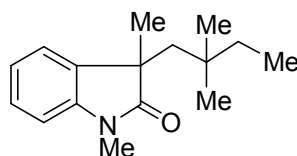
***phenoxyindolin-2-one (4c):***

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.29 (t,  $J = 8.0$  Hz, 2H), 7.04 (t,  $J = 7.6$  Hz, 1H), 7.00-6.90 (m, 4H), 6.80 (d,  $J = 8.0$  Hz, 1H), 3.22 (s, 3H), 2.10 (d,  $J = 14.4$  Hz, 1H), 1.83 (d,  $J = 14.4$  Hz, 1H), 1.28 (s, 3H), 1.38-0.92 (m, 10H), 0.56 (s, 3H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  180.8, 158.4, 151.7, 138.6, 136.1, 129.4( $\times 2$ ), 122.3, 118.9, 117.3( $\times 2$ ), 116.4, 108.4, 50.6, 47.3, 39.1, 38.9, 34.1, 28.4, 26.2, 26.0, 23.9, 21.7( $\times 2$ ). HRMS (ESI $^+$ ): calcd 364.2271 for  $\text{C}_{24}\text{H}_{30}\text{NO}_2^+$  [M+H] $^+$ ; found, 364.2271.



***3-((3r,5r,7r)-adamantan-1-ylmethyl)-1,3-dimethylindolin-2-one (4d):***

Obtained as white wax,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.24 (t,  $J = 7.6$ , 0.8 Hz, 1H), 7.18 (d,  $J = 7.6$  Hz, 1H), 7.01 (t,  $J = 7.6$  Hz, 1H), 6.83 (d,  $J = 8.0$  Hz, 1H), 3.22 (s, 3H), 1.99 (d,  $J = 14.4$  Hz, 1H), 1.73 (d,  $J = 14.4$  Hz, 1H), 1.70 (brs, 3H), 1.50 (brd,  $J = 12.0$  Hz, 3H), 1.37 (brd,  $J = 12.0$ Hz, 3H), 1.26 (s, 3H), 1.20-1.13 (m, 6H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  181.3, 142.8, 134.8, 127.6, 123.7, 122.1, 108.1, 52.2, 46.8, 43.5( $\times 3$ ), 36.8( $\times 3$ ), 34.0, 28.7, 28.7( $\times 3$ ), 26.3. HRMS (ESI $^+$ ): calcd 310.2165 for  $\text{C}_{21}\text{H}_{28}\text{NO}^+$  [M+H] $^+$ ; found, 310.2163.



***3-(2,2-dimethylbutyl)-1,3-dimethylindolin-2-one (4e):***

Obtained as colorless oil,  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ ):  $\delta$  7.26 (t,  $J = 7.6$  Hz, 1H), 7.20 (d,  $J = 7.2$  Hz, 1H), 7.03 (t,  $J = 7.6$  Hz, 1H), 6.84 (d,  $J = 7.6$  Hz, 1H), 3.22 (s, 3H), 2.12 (d,  $J = 14.4$  Hz, ), 1.87 (d,  $J = 14.4$  Hz, 1H), 1.29 (s, 3H), 0.73 (q,  $J = 7.2$  Hz, 2H), 0.57 (s,3H), 0.48 (s, 3H).  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  181.3, 143.0, 134.6, 127.7, 123.9, 122.1, 108.2, 48.7, 47.4, 36.4, 34.3, 27.7, 27.0, 26.4, 8.5. HRMS (ESI $^+$ ): calcd 246.1852 for  $\text{C}_{16}\text{H}_{24}\text{NO}^+$  [M+H] $^+$ ; found, 246.1846.

***<sup>1</sup>H NMR and <sup>13</sup>C NMR spectra of compounds 3 and 4***

