

# Supporting Information

## Photocatalytic Dehydrogenated Oxidation/Amination of 2-Alkyl Benzamides under Transition-Metal-Free Conditions

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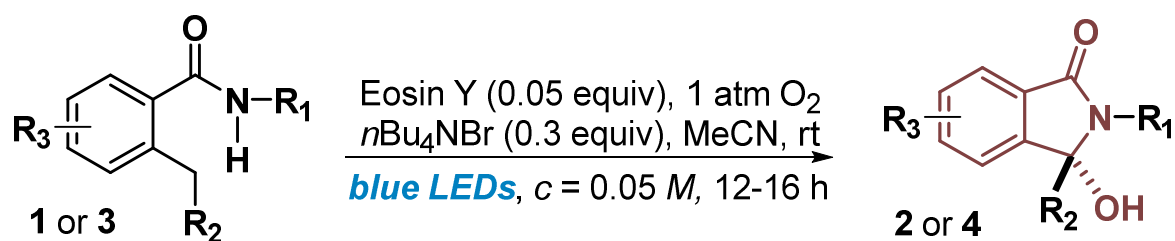
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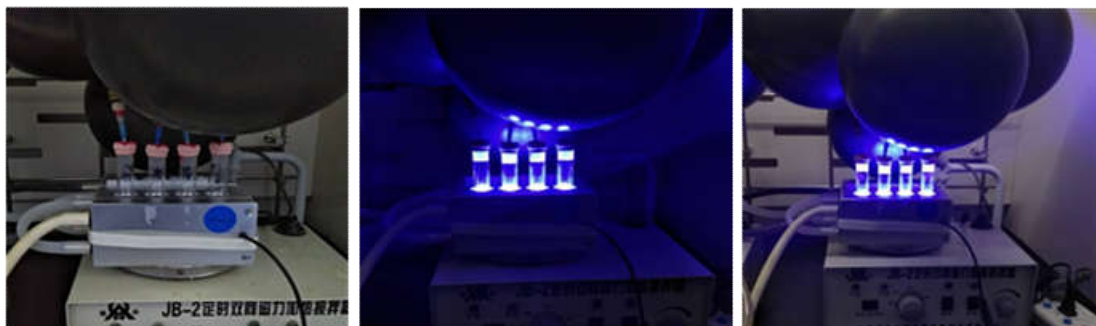
## Materials and methods

All the chemicals were purchased commercially, and used without further purification. Thin-layer chromatography (TLC) was conducted with 0.25 mm Tsingdao silica gel plates (60F-254) and visualized by exposure to UV light (254 nm) or stained with potassium permanganate. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040–0.063 mm). Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated.  $^1\text{H}$  NMR spectra were recorded on JEOL spectrometers (at 400 MHz) and were reported relative to deuterated solvent signals. Data for  $^1\text{H}$  NMR spectra were reported as follows: chemical shift ( $\delta$  ppm), multiplicity, coupling constant (Hz) and integration.  $^{13}\text{C}$  NMR spectra were recorded on JEOL Spectrometers (at 100 MHz). Data for  $^{13}\text{C}$  NMR spectra were reported in terms of chemical shift. Mass spectrometric data were obtained using Bruker Apex IV RTMS. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad.

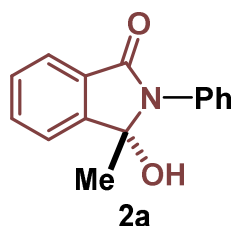
### General procedure for isoindolinone synthesis



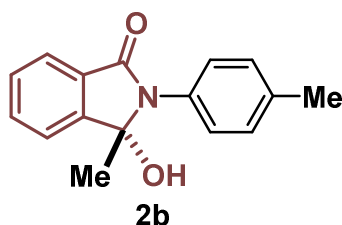
A flame-dried reaction tube was equipped with magnetic stir bar and charged with 2-ethyl-*N*-phenylbenzamide **1** or **3** (0.13 mmol, 1.0 equiv), Eosin Y (0.0067 mmol, 0.05 equiv), *n*Bu<sub>4</sub>NBr (0.04 mmol, 0.3 equiv) and MeCN (3.0 mL). The reaction mixture was irradiated by blue LEDs (12 W, wavelength 450 nm) under a balloon oxygen atmosphere at room temperature until the starting material disappeared from the TLC. After that, the reaction mixture was directly concentrated under reduced pressure and the crude residue was purified by silica gel column chromatography using hexane/EtOAc (*v/v* = 2/1) to afford the desired pure product **2** or **4** in 53-92% yields.



<sup>1</sup>H, <sup>13</sup>C and <sup>19</sup>F spectra data of compounds 2a-2s, 4a-4y

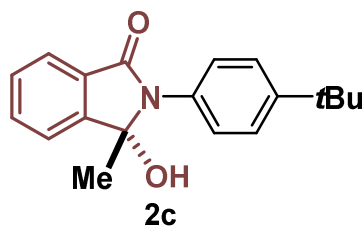


**3-hydroxy-3-methyl-2-phenylisoindolin-1-one (2a):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.61-7.59 (m, 3H), 7.49-7.47 (m, 2H), 7.44-7.41 (m, 1H), 7.39-7.36 (m, 1H), 7.33-7.29 (m, 1H), 3.68 (s, 1H), 1.61 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.7, 147.9, 135.4, 132.9, 130.1, 129.8, 129.2, 129.0, 127.2, 123.8, 121.8, 90.5, 24.4. These data are consistent with literature values, see: Kanako Nozawa-Kumada, Yuta Matsuzawa, Kanako Ono, Masanori Shigeno and Yoshinori Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 29.3 mg, 92% isolated yield)

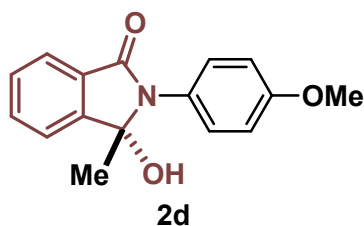


**3-hydroxy-3-methyl-2-(p-tolyl)isoindolin-1-one (2b):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.69-7.67 (d, *J* = 7.6 Hz, 1H), 7.62-7.60 (m, 2H), 7.47-7.43 (m, 1H), 7.37-7.34 (d, *J* = 8.8 Hz, 2H), 7.21-7.19 (d, *J* = 8.4 Hz, 2H), 3.27 (s, 1H), 2.39 (s, 3H), 1.62 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.6, 147.8, 137.3, 132.9, 132.6, 130.3, 129.8, 129.7, 127.3, 123.9, 121.8, 90.3, 24.5, 21.2; HRMS calculated for C<sub>19</sub>H<sub>16</sub>NO<sub>2</sub> (M + H<sup>+</sup>): 254.1182, found: 254.1176. (Yellow solid, 22.2 mg, 70% isolated yield)

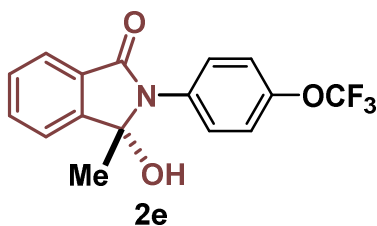




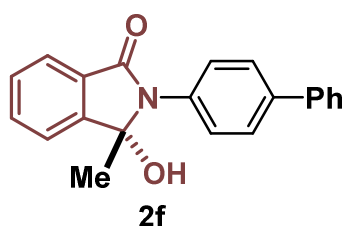
**2-(4-(tert-butyl)phenyl)-3-hydroxy-3-methylisoindolin-1-one (2c):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57-7.56 (d,  $J=4.4$  Hz, 2H), 7.53-7.51 (d,  $J=7.6$  Hz, 1H), 7.39-7.36 (m, 5H), 3.90 (s, 1H), 1.59 (s, 3H), 1.35 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.8, 150.1, 148.0, 132.8, 132.5, 130.2, 129.6, 126.8, 126.0, 123.7, 121.7, 90.4, 34.7, 31.5, 24.5; HRMS calculated for  $\text{C}_{19}\text{H}_{22}\text{NO}_2$  ( $\text{M} + \text{H}^+$ ): 296.1651, found: 296.1645. (White solid, 28.6 mg, 91% isolated yield)



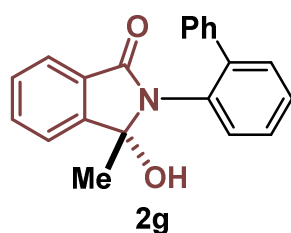
**4-hydroxy-2-(4-methoxyphenyl)-3-methylisoindolin-1-one (2d):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58-7.57 (d,  $J=3.6$  Hz, 2H), 7.49-7.47 (d,  $J=8.0$  Hz, 1H), 7.39-7.36 (m, 1H), 7.33-7.31 (d,  $J=8.8$  Hz, 2H), 6.88-6.86 (d,  $J=9.6$  Hz, 2H), 3.82 (s, 3H), 1.57 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.8, 158.7, 147.9, 132.7, 130.2, 129.7, 128.8, 127.8, 123.6, 121.8, 114.3, 90.3, 55.5, 24.5; HRMS calculated for  $\text{C}_{16}\text{H}_{16}\text{NO}_3$  ( $\text{M} + \text{H}^+$ ): 270.1130, found: 270.1125. (White solid, 27.5 mg, 87% isolated yield)



**3-hydroxy-3-methyl-2-(4-(trifluoromethoxy)phenyl)isoindolin-1-one (2e):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62-7.60 (m, 2H), 7.59-7.55 (m, 2H), 7.50-7.48 (d,  $J = 7.2$  Hz, 1H), 7.40-7.36 (m, 1H), 7.20-7.18 (m, 2H), 3.94 (s, 1H), 1.60 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.8, 147.8, 147.6, 134.1, 133.2, 129.9, 129.6, 127.9, 127.7, 123.8, 121.9, 121.8, 121.4, 90.8, 24.3;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.8; HRMS calculated for  $\text{C}_{16}\text{H}_{13}\text{F}_3\text{NO}_3$  ( $\text{M} + \text{H}^+$ ): 324.0848, found: 324.0842. (White solid, 27.6 mg, 88% isolated yield)

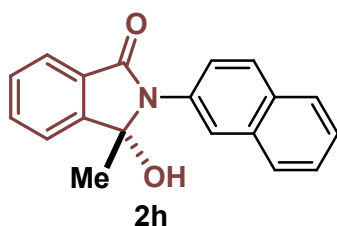


**2-([1,1'-biphenyl]-4-yl)-3-hydroxy-3-methylisoindolin-1-one (2f):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63-7.61 (m, 2H), 7.59-7.58 (m, 1H), 7.57-7.54 (m, 4H), 7.53-7.51 (m, 2H), 7.43-7.38 (m, 3H), 7.37-7.35 (m, 1H), 3.84 (s, 1H), 1.65 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.8, 148.0, 140.5, 139.8, 134.7, 133.0, 130.0, 129.8, 128.9, 127.8, 127.6, 127.4, 127.1, 127.0, 123.7, 121.8, 90.7, 24.4; HRMS calculated for  $\text{C}_{21}\text{H}_{17}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 338.1157, found: 338.1152. (Yellow oil, 23.5 mg, 75% isolated yield)

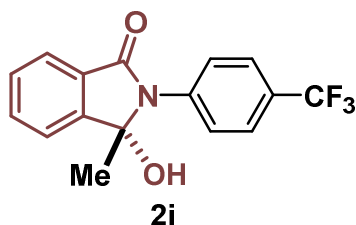


**2-([1,1'-biphenyl]-2-yl)-3-hydroxy-3-methylisoindolin-1-one (2g):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67-7.63 (m, 3H), 7.62-7.60 (m, 4H), 7.58-7.56 (m, 2H), 7.48-7.41 (m, 3H), 7.38-7.34 (m, 1H), 1.68 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.6, 152.5, 150.4, 147.9, 140.6, 140.0, 134.7, 133.0, 130.2, 130.0, 128.9, 127.7, 127.5, 127.2, 127.1, 123.9,

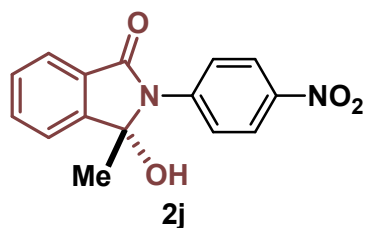
121.8, 90.6, 24.4; HRMS calculated for  $C_{21}H_{17}NO_2Na$  ( $M + Na^+$ ): 338.1157, found: 338.1152. (Yellow oil, 24.2 mg, 77% isolated yield)



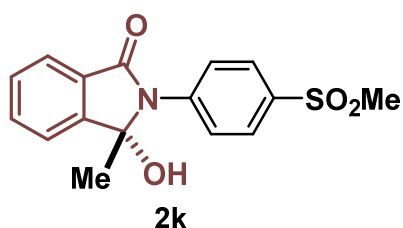
**3-hydroxy-3-methyl-2-(naphthalen-2-yl)isoindolin-1-one (2h):**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.91-7.90 (d,  $J = 2.0$  Hz, 1H), 7.76-7.73 (d,  $J = 8.4$  Hz, 1H), 7.65-7.63 (d,  $J = 8.8$  Hz, 1H), 7.60-7.51 (m, 4H), 7.46-7.40 (m, 2H), 7.37-7.30 (m, 2H), 4.34 (s, 1H), 1.57 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  167.0, 148.1, 133.4, 133.0, 132.2, 130.0, 129.7, 128.5, 128.2, 127.5, 126.1, 126.0, 125.0, 124.9, 123.6, 121.8, 90.9, 24.4; HRMS calculated for  $C_{19}H_{15}NO_2Na$  ( $M + Na^+$ ): 312.1000, found: 312.0095. (White solid, 24.9 mg, 79% isolated yield)



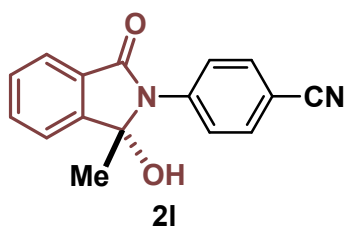
**3-hydroxy-3-methyl-2-(4-(trifluoromethyl)phenyl)isoindolin-1-one (2i):**  $^1H$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  7.89-7.87 (d,  $J = 8.4$  Hz, 2H), 7.82-7.80 (d,  $J = 8.8$  Hz, 2H), 7.76-7.74 (d,  $J = 7.6$  Hz, 1H), 7.71-7.70 (d,  $J = 3.6$  Hz, 2H), 7.58-7.54 (m, 1H), 7.01 (s, 1H), 1.56 (s, 3H);  $^{13}C$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  166.1, 149.3, 140.8, 133.8, 130.1, 130.0, 127.1, 126.9, 126.3, 126.2, 123.6, 122.8, 90.8, 25.1;  $^{19}F$  NMR (376 MHz,  $d_6$ -DMSO)  $\delta$  -60.7; HRMS calculated for  $C_{16}H_{12}F_3NO_2Na$  ( $M + Na^+$ ): 330.0718, found: 330.0712. (White solid, 28.0 mg, 89% isolated yield)



**3-hydroxy-3-methyl-2-(4-nitrophenyl)isoindolin-1-one (2j):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  8.33-8.29 (m, 2H), 8.07-8.03 (m, 2H), 7.78-7.76 (d,  $J = 7.2$  Hz, 1H), 7.74-7.71 (m, 2H), 7.60-7.56 (m, 1H), 1.61 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  166.2, 149.3, 144.8, 143.6, 134.1, 130.2, 129.7, 125.5, 124.8, 123.8, 122.8, 91.3, 25.0; HRMS calculated for  $\text{C}_{15}\text{H}_{11}\text{N}_2\text{O}_4$  ( $\text{M} - \text{H}^+$ ): 283.0719, found: 283.0724. (Yellow solid, 26.2 mg, 83% isolated yield)

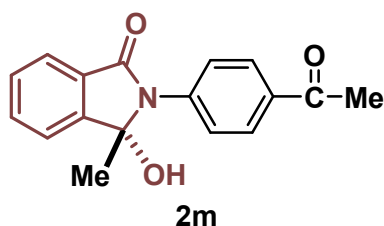


**3-hydroxy-3-methyl-2-(4-(methylsulfonyl)phenyl)isoindolin-1-one (2k):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  8.00-7.93 (m, 4H), 7.77-7.75 (d,  $J = 8.0$  Hz, 1H), 7.72-7.71 (d,  $J = 3.6$  Hz, 1H), 7.59-7.55 (m, 1H), 7.06 (s, 1H), 3.23 (s, 3H), 1.58 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  166.1, 149.3, 141.8, 138.3, 133.9, 130.1, 130.0, 128.2, 126.4, 123.6, 122.8, 91.0, 44.1, 25.1; HRMS calculated for  $\text{C}_{16}\text{H}_{15}\text{NO}_4\text{SNa}$  ( $\text{M} + \text{Na}^+$ ): 340.0619, found: 340.0614. (White solid, 27.3 mg, 87% isolated yield)

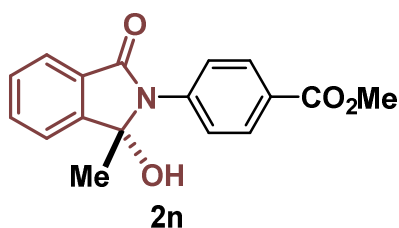


**4-(1-hydroxy-1-methyl-3-oxoisoindolin-2-yl)benzonitrile (2l):**  $^1\text{H}$  NMR (400 MHz,

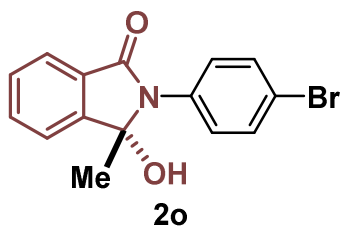
CDCl<sub>3</sub>)  $\delta$  7.94-7.89 (m, 4H), 7.77-7.75 (d,  $J = 7.2$  Hz, 1H), 7.72-7.71 (m, 2H), 7.59-7.55 (m, 1H), 1.58 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.1, 149.3, 141.6, 134.0, 133.4, 130.1, 129.9, 126.2, 123.7, 122.8, 119.3, 108.6, 91.1, 25.1; HRMS calculated for C<sub>16</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>Na (M + Na<sup>+</sup>): 287.0796, found: 287.0791. (White solid, 24.7 mg, 78% isolated yield)



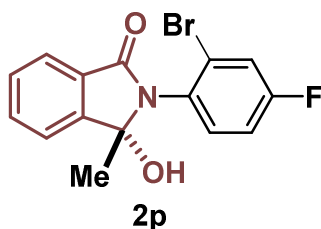
**2-(4-acetylphenyl)-3-hydroxy-3-methylisoindolin-1-one (2m):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.85-7.82 (m, 2H), 7.73-7.70 (m, 2H), 7.61-7.60 (m, 2H), 7.39-7.37 (d,  $J = 7.6$  Hz, 1H), 7.35-7.31 (m, 1H), 4.59 (s, 1H), 2.58 (s, 3H), 1.62 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  197.6, 166.8, 148.0, 140.4, 134.5, 133.4, 129.8, 129.3, 129.0, 124.8, 123.7, 121.7, 91.4, 26.6, 24.2; HRMS calculated for C<sub>17</sub>H<sub>15</sub>NO<sub>3</sub>Na (M + Na<sup>+</sup>): 304.0950, found: 304.0944. (White solid, 25.6 mg, 81% isolated yield)



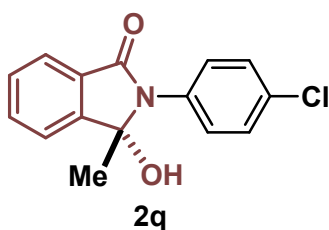
**methyl 4-(1-hydroxy-1-methyl-3-oxoisoindolin-2-yl)benzoate (2n):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.01-7.99 (m, 2H), 7.75-7.73 (m, 2H), 7.64-7.58 (m, 3H), 7.44-7.40 (m, 1H), 3.93 (s, 3H), 1.65 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.7, 166.6, 147.9, 140.3, 133.4, 130.3, 130.0, 129.5, 127.8, 124.9, 124.0, 121.7, 91.2, 52.3, 24.2; HRMS calculated for C<sub>17</sub>H<sub>15</sub>NO<sub>4</sub>Na (M + Na<sup>+</sup>): 320.0899, found: 320.0893. (Yellow solid, 25.2 mg, 80% isolated yield)



**2-(4-bromophenyl)-3-hydroxy-3-methylisoindolin-1-one (2o):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61-7.57 (m, 2H), 7.45-7.34 (m, 6H), 4.13 (s, 1H), 1.57 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.6, 147.9, 134.6, 133.2, 132.0, 129.8, 129.5, 127.7, 123.7, 121.7, 120.5, 90.8, 24.2; HRMS calculated for  $\text{C}_{15}\text{H}_{12}\text{BrNO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 339.9949, found: 339.9944. (White solid, 27.6 mg, 88% isolated yield)

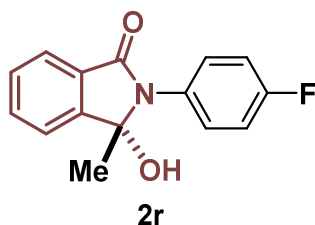


**2-(2-bromo-4-fluorophenyl)-3-hydroxy-3-methylisoindolin-1-one (2p):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  7.76-7.67 (m, 4H), 7.57-7.51 (m, 2H), 7.41-7.36 (m, 1H), 6.79 (s, 1H), 1.49 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  165.2, 160.5, 149.7, 133.9, 133.5, 131.7, 131.6, 130.2, 130.0, 125.7, 125.6, 123.6, 122.9, 120.9, 120.6, 116.0, 115.8, 90.1, 24.4;  $^{19}\text{F}$  NMR (376 MHz,  $d_6$ -DMSO)  $\delta$  -110.9; HRMS calculated for  $\text{C}_{15}\text{H}_{11}\text{BrFNO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 357.9855, found: 357.9849. (Yellow solid, 28.2 mg, 90% isolated yield)

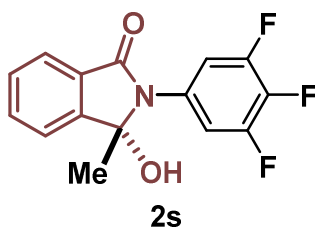


**2-(4-chlorophenyl)-3-hydroxy-3-methylisoindolin-1-one (2q):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63-7.58 (m, 2H), 7.46-7.43 (m, 2H), 7.41-7.39 (d,  $J = 7.2$  Hz, 1H), 7.37-7.33

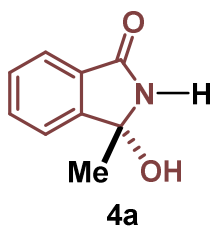
(m, 1H), 7.31-7.26 (m, 2H), 4.07 (s, 1H), 1.58 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.7, 147.9, 134.0, 133.2, 132.5, 129.8, 129.6, 129.0, 127.5, 123.7, 121.7, 90.8, 24.2; HRMS calculated for  $\text{C}_{15}\text{H}_{12}\text{ClNO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 296.0454, found: 296.0499. (White solid, 25.6 mg, 81% isolated yield)



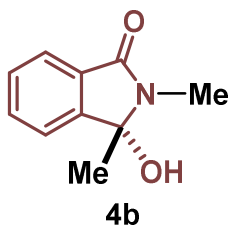
**2-(4-fluorophenyl)-3-hydroxy-3-methylisoindolin-1-one (2r):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61-7.59 (m, 2H), 7.46-7.41 (m, 3H), 7.39-7.35 (m, 1H), 7.06-7.02 (t,  $J = 8.8$  Hz, 2H), 1.57 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.7, 160.3, 147.8, 133.0, 131.2, 129.8, 128.8, 128.7, 123.7, 121.8, 115.9, 115.7, 90.5, 24.3;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -114.5; HRMS calculated for  $\text{C}_{15}\text{H}_{12}\text{FNO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 280.0750, found: 280.0744. (White solid, 28.9 mg, 91% isolated yield)



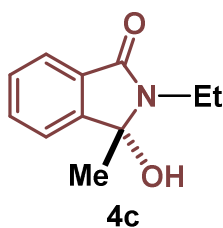
**3-hydroxy-3-methyl-2-(3,4,5-trifluorophenyl)isoindolin-1-one (2s):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65-7.58 (m, 2H), 7.34-7.26 (m, 4H), 4.42 (s, 1H), 1.61 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.6, 147.7, 133.7, 129.9, 128.8, 123.7, 121.8, 109.9, 109.8, 109.7, 109.6, 91.4, 24.0;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -133.7, -162.5; HRMS calculated for  $\text{C}_{15}\text{H}_{10}\text{F}_3\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 316.0561, found: 316.0556. (White solid, 28.0 mg, 89% isolated yield)



**3-hydroxy-3-methylisoindolin-1-one (4a):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60-7.53 (m, 3H), 7.42-7.38 (m, 1H), 7.09 (s, 1H), 3.79 (s, 1H), 1.78 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.1, 149.4, 133.0, 130.0, 129.6, 123.6, 121.8, 86.1, 25.9; HRMS calculated for  $\text{C}_9\text{H}_9\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 186.0531, found: 186.0526. (White solid, 23.3 mg, 71% isolated yield)



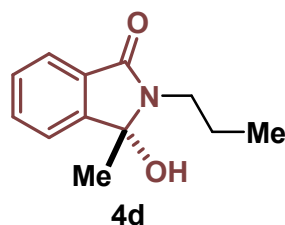
**3-hydroxy-2,3-dimethylisoindolin-1-one (4b):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.59-7.51 (m, 3H), 7.41-7.37 (m, 1H), 2.82 (s, 3H), 1.64 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.9, 148.2, 132.3, 130.4, 129.5, 123.2, 121.7, 88.3, 23.4, 23.2; HRMS calculated for  $\text{C}_{10}\text{H}_{11}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 200.0687, found: 200.0682. (White solid, 17.3 mg, 53% isolated yield)



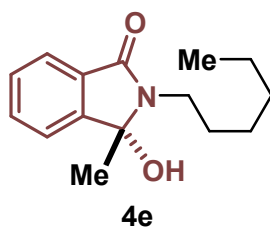
**2-ethyl-3-hydroxy-3-methylisoindolin-1-one (4c):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58-7.54 (m, 3H), 7.44-7.39 (m, 1H), 3.49-3.40 (m, 1H), 3.32-3.21 (m, 1H), 1.69 (s, 1H),



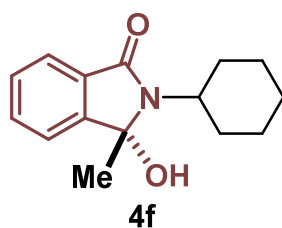
1.24-1.21 (t,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.9, 148.2, 132.3, 130.6, 129.5, 123.2, 121.6, 88.9, 33.2, 24.4, 14.8; HRMS calculated for  $\text{C}_{11}\text{H}_{13}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 214.0844, found: 214.0839. (White solid, 17.5 mg, 54% isolated yield)



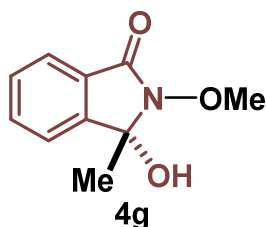
**3-hydroxy-3-methyl-2-propylisoindolin-1-one (4d):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62-7.60 (m, 1H), 7.56-7.54 (m, 2H), 7.44-7.40 (m, 1H), 3.41-3.34 (m, 1H), 3.20-3.13 (m, 1H), 1.75-1.71 (m, 1H), 1.70 (s, 3H), 1.67-1.59 (m, 1H), 0.95-0.91 (t,  $J = 7.2$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.1, 148.1, 132.3, 130.6, 129.5, 123.3, 121.6, 88.9, 40.4, 24.4, 22.6, 11.8; HRMS calculated for  $\text{C}_{12}\text{H}_{15}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 228.1000, found: 228.0995. (White solid, 16.7 mg, 52% isolated yield)



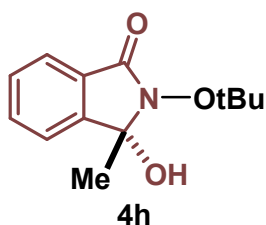
**2-hexyl-3-hydroxy-3-methylisoindolin-1-one (4e):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61-7.58 (m, 1H), 7.55-7.54 (m, 2H), 7.43-7.39 (m, 1H), 3.40-3.33 (m, 1H), 3.17-3.10 (m, 1H), 1.69 (s, 1H), 1.60-1.56 (m, 1H), 1.34-1.24 (m, 7H), 0.90-0.87 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.0, 148.2, 132.3, 130.6, 129.5, 123.2, 121.6, 88.9, 38.8, 31.6, 29.3, 27.1, 24.4, 22.7, 14.1; HRMS calculated for  $\text{C}_{15}\text{H}_{21}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 270.1470, found: 270.1465. (White solid, 18.1 mg, 57% isolated yield)



**2-cyclohexyl-3-hydroxy-3-methylisoindolin-1-one (4f):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63-7.61 (d,  $J = 7.2$  Hz, 1H), 7.51-7.48 (m, 2H), 7.44-7.40 (m, 1H), 3.47-3.41 (m, 1H), 2.86 (s, 1H), 2.40-2.31 (m, 2H), 1.87-1.82 (m, 2H), 1.70-1.66 (m, 6H), 1.37-1.27 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.3, 147.6, 132.0, 131.7, 129.5, 123.1, 121.4, 89.3, 52.2, 30.7, 30.3, 26.5, 25.3, 24.4; HRMS calculated for  $\text{C}_{15}\text{H}_{19}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 268.1313, found: 268.1308. (White solid, 24.5 mg, 77% isolated yield)

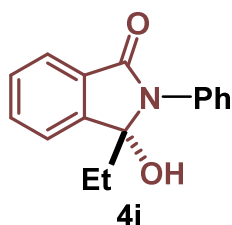


**3-hydroxy-2-methoxy-3-methylisoindolin-1-one (4g):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71-7.69 (d,  $J = 8.0$  Hz, 1H), 7.63-7.59 (m, 1H), 7.54-7.52 (d,  $J = 7.2$  Hz, 1H), 7.49-7.45 (m, 1H), 4.06 (s, 3H), 1.76 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  163.9, 145.6, 133.2, 129.9, 128.0, 123.7, 121.8, 88.8, 66.0, 23.4; HRMS calculated for  $\text{C}_{10}\text{H}_{11}\text{NO}_3\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 216.0637, found: 216.0631. (Yellow oil, 22.6 mg, 70% isolated yield)

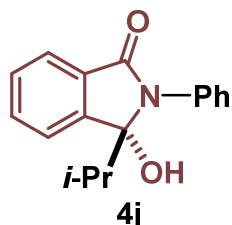


**2-(tert-butoxy)-3-hydroxy-3-methylisoindolin-1-one (4h):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.77-7.50 (d,  $J = 7.6$  Hz, 1H), 7.65-7.61 (m, 1H), 7.58-7.56 (d,  $J = 8.0$  Hz, 1H), 7.52-7.48 (m, 1H), 1.69 (s, 3H), 1.43 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.4,

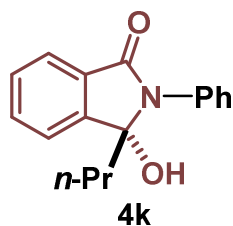
146.8, 133.4, 129.9, 128.1, 1232.8, 122.1, 89.2, 82.9, 28.1, 24.1; HRMS calculated for  $C_{13}H_{17}NO_3Na$  ( $M + Na^+$ ): 258.1106, found: 258.1101. (Yellow oil, 23.6 mg, 74% isolated yield)



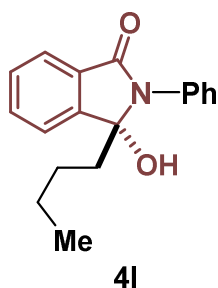
**3-ethyl-3-hydroxy-2-phenylisoindolin-1-one (4i):**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.63-7.53 (m, 5H), 7.44-7.40 (t,  $J = 8.0$  Hz, 1H), 7.38-7.34 (m, 2H), 7.29-7.26 (m, 1H), 2.14-1.96 (m, 2H), 0.49-0.45 (t,  $J = 7.2$  Hz, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  167.2, 146.1, 135.6, 132.9, 131.1, 129.8, 128.9, 126.9, 126.3, 123.8, 121.8, 94.0, 28.9, 7.9. These data are consistent with literature values, see: K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 27.0 mg, 85% isolated yield)



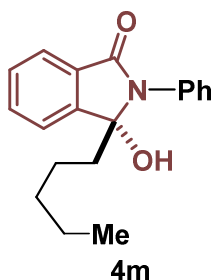
**3-hydroxy-3-isopropyl-2-phenylisoindolin-1-one (4j):**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.64-7.61 (m, 1H), 7.59-7.55 (m, 4H), 7.45-7.41 (m, 1H), 7.37-7.33 (m, 2H), 7.30-7.25 (m, 1H), 2.28-2.21 (m, 1H), 1.22-1.20 (d,  $J = 4.8$  Hz, 3H), 0.48-0.46 (d,  $J = 6.8$  Hz, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  167.0, 145.0, 135.9, 132.4, 131.7, 129.7, 128.9, 126.9, 126.8, 123.8, 123.1, 95.9, 34.0, 17.2, 16.7; HRMS calculated for  $C_{17}H_{17}NO_2Na$  ( $M + Na^+$ ): 290.1157, found: 290.1152. (White solid, 25.6 mg, 81% isolated yield)



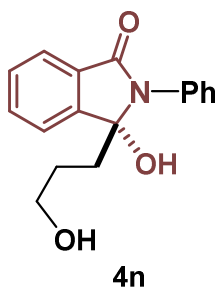
**3-hydroxy-2-phenyl-3-propylisoindolin-1-one (4k):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.59-7.46 (m, 5H), 7.34-7.24 (m, 4H), 4.06 (s, 1H), 2.03-1.85 (m, 2H), 1.01-0.93 (m, 1H), 0.72-0.62 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.1, 146.6, 135.5, 132.8, 130.8, 129.6, 128.9, 126.8, 126.4, 123.7, 121.9, 93.4, 38.1, 16.8, 13.8. These data are consistent with literature values, see: K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 26.9 mg, 85% isolated yield)



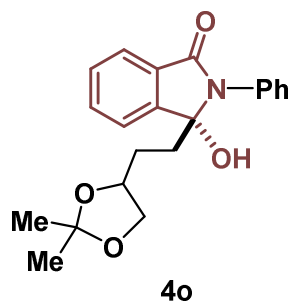
**3-butyl-3-hydroxy-2-phenylisoindolin-1-one (4l):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63-7.59 (m, 2H), 7.55-7.53 (m, 3H), 7.44-7.40 (m, 1H), 7.38-7.34 (m, 2H), 7.30-7.26 (m, 1H), 2.09-1.90 (m, 2H), 1.08-0.88 (m, 3H), 0.71-0.61 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.1, 146.5, 135.5, 132.9, 131.0, 129.8, 128.9, 126.9, 126.5, 123.8, 121.8, 93.4, 35.5, 25.5, 22.3, 13.7; HRMS calculated for  $\text{C}_{18}\text{H}_{19}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 304.1313, found: 304.1308. (Yellow oil, 24.6 mg, 78% isolated yield)



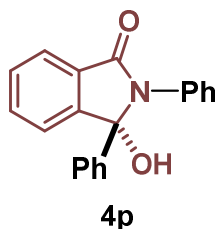
**3-hydroxy-3-pentyl-2-phenylisoindolin-1-one (4m):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67-7.65 (m, 1H), 7.63-7.59 (m, 1H), 7.57-7.54 (m, 3H), 7.46-7.42 (m, 1H), 7.39-7.35 (m, 2H), 7.31-7.27 (m, 1H), 2.09-1.90 (m, 2H), 1.07-0.94 (m, 5H), 0.72-0.64 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.0, 146.4, 135.6, 132.9, 131.0, 129.8, 128.9, 126.9, 126.5, 123.9, 121.8, 93.4, 35.8, 31.3, 23.0, 22.2, 13.8; HRMS calculated for  $\text{C}_{19}\text{H}_{21}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 296.1651, found: 296.1645. (Yellow oil, 27.4 mg, 87% isolated yield)



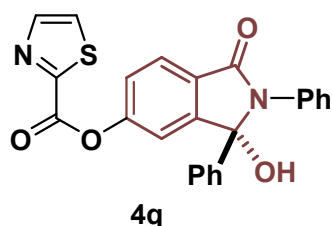
**3-hydroxy-3-(3-hydroxypropyl)-2-phenylisoindolin-1-one (4n):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  7.76-7.70 (m, 2H), 7.65-7.63 (d,  $J = 7.2$  Hz, 1H), 7.60-7.56 (m, 3H), 7.48-7.44 (m, 2H), 7.35-7.31 (m, 1H), 6.92 (s, 1H), 3.13-3.12 (d,  $J = 4.4$  Hz, 2H), 2.09-2.01 (m, 1H), 1.85-1.78 (m, 1H), 1.15-1.07 (m, 1H), 0.83-0.74 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  166.4, 147.9, 136.6, 133.3, 131.4, 129.9, 129.2, 127.4, 127.1, 123.3, 122.9, 93.0, 60.7, 33.4, 27.4; HRMS calculated for  $\text{C}_{17}\text{H}_{17}\text{NO}_3\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 306.1106, found: 306.1101. (Yellow solid, 24.6 mg, 74% isolated yield)



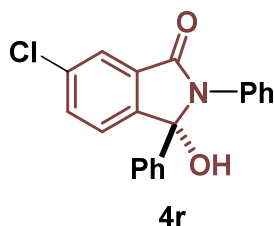
**3-(2-(2,2-dimethyl-1,3-dioxolan-4-yl)ethyl)-3-hydroxy-2-phenylisoindolin-1-one (4o):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66-7.63 (m, 1H), 7.61-7.57 (m, 2H), 7.55-7.52 (m, 2H), 7.47-7.43 (m, 1H), 7.39-7.35 (m, 2H), 7.33-7.29 (m, 1H), 3.82-3.78 (m, 1H), 3.54-3.51 (m, 1H), 3.21-3.17 (t,  $J = 9.2$  Hz, 1H), 2.40-2.34 (m, 1H), 2.20-2.15 (m, 1H), 1.25 (s, 3H), 1.12 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.7, 146.4, 135.7, 132.8, 130.1, 129.0, 127.4, 123.9, 122.4, 108.9, 91.6, 71.9, 69.9, 40.7, 26.7, 25.6; HRMS calculated for  $\text{C}_{21}\text{H}_{23}\text{NO}_4\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 354.1705, found: 354.1700. (Yellow oil, 21.2 mg, 65% isolated yield)



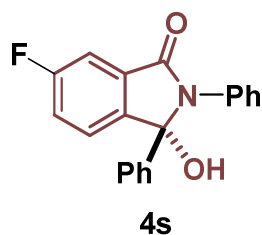
**3-hydroxy-2,3-diphenylisoindolin-1-one (4p):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72-7.70 (d,  $J = 3.2$  Hz, 1H), 7.53-7.49 (m, 1H), 7.43-7.41 (m, 3H), 7.38-7.34 (m, 4H), 7.31-7.29 (d,  $J = 8.0$  Hz, 1H), 7.25-7.20 (m, 3H), 7.19-7.17 (m, 1H), 7.12-7.08 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.6, 148.7, 138.7, 135.9, 133.3, 129.9, 129.8, 128.6, 128.5, 128.4, 126.3, 126.2, 125.5, 123.9, 122.8, 93.1. These data are consistent with literature values, see: D.-M. Yan, Q.-Q. Zhao, L. Rao, J.-R. Chen and W.-J. Xiao. *Chem. Eur. J.* **2018**, *24*, 16895. (White solid, 24.5 mg, 78% isolated yield)



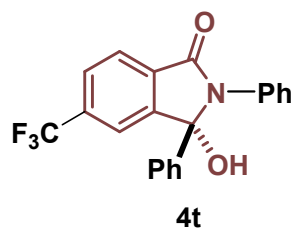
**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl thiazole-2-carboxylate (4q):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.83-8.82 (d,  $J = 2.0$  Hz, 1H), 8.37-8.36 (d,  $J = 1.6$  Hz, 1H), 7.71-7.69 (d,  $J = 8.0$  Hz, 1H), 7.45-7.42 (m, 2H), 7.40-7.36 (m, 2H), 7.34-7.33 (m, 1H), 7.30-7.27 (m, 1H), 7.25-7.21 (m, 3H), 7.18-7.15 (m, 2H), 7.11-7.09 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.7, 159.0, 155.3, 155.2, 150.6, 146.4, 138.4, 135.8, 131.6, 129.5, 128.6, 128.5, 128.4, 127.7, 126.4, 126.3, 126.2, 126.1, 125.6, 125.2, 123.7, 116.6, 92.7; HRMS calculated for  $\text{C}_{24}\text{H}_{16}\text{N}_2\text{O}_4\text{SNa}$  ( $\text{M} + \text{Na}^+$ ): 451.0728, found: 457.0723. (Yellow solid, 26.7 mg, 86% isolated yield)



**6-chloro-3-hydroxy-2,3-diphenylisoindolin-1-one (4r):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  7.82-7.81 (d,  $J = 1.6$  Hz, 1H), 7.64-7.62 (dd,  $J_1 = 2.0$  Hz,  $J_2 = 1.6$  Hz, 1H), 7.45-7.43 (m, 2H), 7.34-7.32 (m, 2H), 7.27-7.24 (m, 3H), 1.22-7.18 (m, 3H), 7.13-7.09 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  165.5, 148.6, 139.8, 136.7, 134.7, 133.7, 132.3, 129.0, 128.9, 128.6, 126.5, 126.4, 126.0, 125.4, 123.3, 92.6. These data are consistent with literature values, see: K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 24.1 mg, 77% isolated yield)

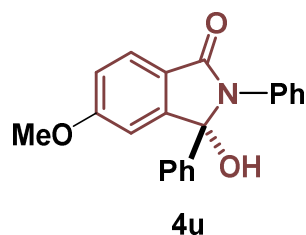


**6-fluoro-3-hydroxy-2,3-diphenylisoindolin-1-one (4s):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  7.89-7.87 (d,  $J = 8.4$  Hz, 3H), 7.82-7.80 (d,  $J = 8.8$  Hz, 2H), 7.76-7.74 (d,  $J = 7.2$  Hz, 1H), 7.71-7.70 (d,  $J = 3.6$  Hz, 2H), 7.58-7.54 (m, 1H), 7.01 (s, 1H), 1.56 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  166.1, 149.3, 140.8, 133.8, 130.1, 130.0, 127.1, 126.9, 126.4, 126.3, 123.6, 122.8, 90.8, 25.1;  $^{19}\text{F}$  NMR (376 MHz,  $d_6$ -DMSO)  $\delta$  -113.1. These data are consistent with literature values, see: K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 25.4 mg, 81% isolated yield)

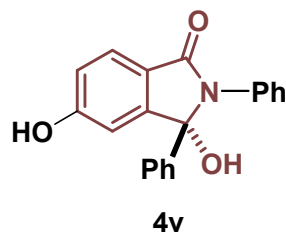


**3-hydroxy-2,3-diphenyl-5-(trifluoromethyl)isoindolin-1-one (4t):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  8.09-8.07 (d,  $J = 8.0$  Hz, 1H), 7.98-7.96 (dd,  $J_1 = 0.8$  Hz,  $J_2 = 1.2$  Hz, 1H), 7.90 (s, 1H), 7.57 (s, 1H), 7.51-7.48 (m, 2H), 7.42-7.39 (m, 2H), 7.31-7.23 (m, 5H), 7.19-7.15 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  165.4, 150.7, 139.4, 136.5, 134.1, 129.1, 129.0, 128.8, 127.4, 127.3, 126.7, 126.6, 126.1, 125.0, 120.1, 92.7;  $^{19}\text{F}$  NMR (376 MHz,  $d_6$ -DMSO)  $\delta$  -60.9. These data are consistent with literature values, see: K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 23.7 mg, 76% isolated yield)

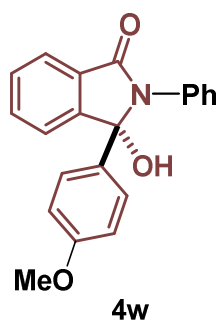




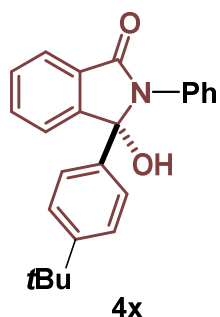
**3-hydroxy-5-methoxy-2,3-diphenylisoindolin-1-one (4u):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.48-7.46 (d,  $J = 8.4$  Hz, 1H), 7.41-7.39 (d,  $J = 8.0$  Hz, 2H), 7.36-7.34 (d,  $J = 2.4$  Hz, 2H), 7.23-7.20 (m, 3H), 7.14-7.10 (m, 2H), 7.06-7.03 (m, 1H), 6.77-6.72 (m, 2H), 4.86 (s, 1H), 3.75 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.7, 164.1, 151.3, 138.9, 136.2, 128.4, 128.3, 126.2, 125.7, 125.3, 125.0, 122.0, 116.3, 107.2, 92.8, 55.8. These data are consistent with literature values, see: K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 23.5 mg, 75% isolated yield)



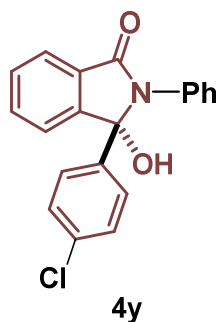
**3,5-dihydroxy-2,3-diphenylisoindolin-1-one (4v):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  10.3 (s, 1H), 7.66-7.64 (dd,  $J = 8.4$  Hz, 1H), 7.58 (s, 1H), 7.52-7.50 (m, 2H), 7.35-7.33 (m, 2H), 7.29-7.20 (m, 5H), 7.10-7.06 (m, 1H), 6.91-6.89 (dd,  $J_1 = 6.4$  Hz,  $J_2 = 2.0$  Hz, 1H), 6.56-6.55 (d,  $J = 2.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  166.9, 162.6, 152.6, 141.0, 137.5, 128.9, 128.7, 128.3, 126.3, 125.7, 125.5, 125.4, 121.0, 117.3, 109.4, 92.1;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -60.9; HRMS calculated for  $\text{C}_{20}\text{H}_{15}\text{NO}_3\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 340.0950, found: 340.0944. (White solid, 24.5 mg, 78% isolated yield)



**3-hydroxy-3-(4-methoxyphenyl)-2-phenylisoindolin-1-one (4w):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66-7.64 (d,  $J = 7.6$  Hz, 1H), 7.52-7.48 (m, 1H), 7.42-7.40 (m, 2H), 7.38-7.34 (m, 1H), 7.30-7.25 (m, 3H), 7.21-7.17 (m, 2H), 7.13-7.09 (m, 1H), 6.76-6.72 (m, 2H), 4.21 (s, 1H), 3.72 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.5, 159.5, 148.9, 135.9, 133.3, 130.7, 129.8, 129.7, 128.6, 127.5, 126.2, 125.6, 123.8, 122.7, 113.8, 93.1, 55.2. These data are consistent with literature values, see: K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 26.6 mg, 85% isolated yield)

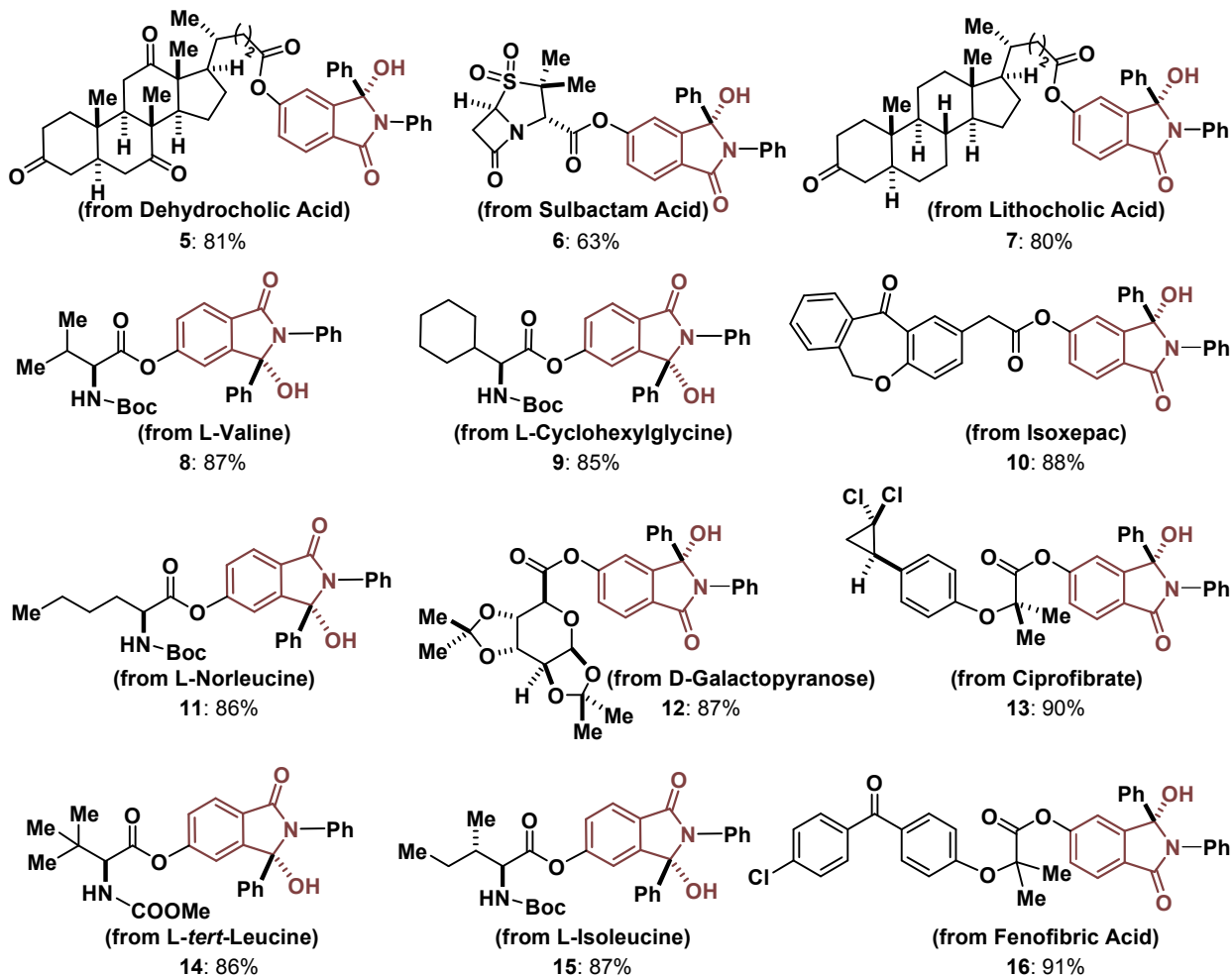


**3-(4-(tert-butyl)phenyl)-3-hydroxy-2-phenylisoindolin-1-one (4x):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72-7.67 (m, 1H), 7.51-7.49 (m, 1H), 7.45-7.43 (m, 2H), 7.38-7.34 (m, 1H), 7.31-7.29 (m, 2H), 7.27-7.24 (m, 2H), 7.22-7.17 (m, 3H), 7.13-7.11 (m, 1H), 1.23 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.6, 151.3, 148.9, 136.0, 135.6, 133.2, 129.8, 129.6, 128.6, 126.1, 125.8, 125.5, 125.4, 123.8, 122.8, 93.2, 34.6, 31.3, 31.1. These data are consistent with literature values, see: J. M. Dennis, C. M. Calyore, J. S. Sjöholm, J. P. Lutz, J. J. Gair and J. B. Johnson. *Synlett*, **2013**, 24, 2567. (White solid, 25.0 mg, 80% isolated yield)

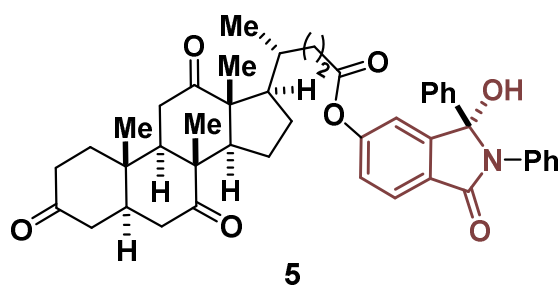


**3-(4-chlorophenyl)-3-hydroxy-2-phenylisoindolin-1-one (4y):**  $^1\text{H}$  NMR (400 MHz,  $d_6$ -DMSO)  $\delta$  7.81-7.80 (d,  $J = 6.8$  Hz, 1H), 7.74 (s, 1H), 7.62-7.53 (m, 2H), 7.45-7.42 (m, 2H), 7.34-7.23 (m, 7H), 7.13-7.09 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $d_6$ -DMSO)  $\delta$  166.8, 149.6, 139.6, 136.8, 133.9, 133.1, 130.3, 130.1, 128.9, 128.6, 126.5, 126.1, 123.7, 123.4, 120.8, 92.3. These data are consistent with literature values, see: K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo. *Chem. Commun.* **2021**, 57, 8604. (White solid, 26.3 mg, 84% isolated yield)

## Late-stage diversification of substances derived from natural products and drugs

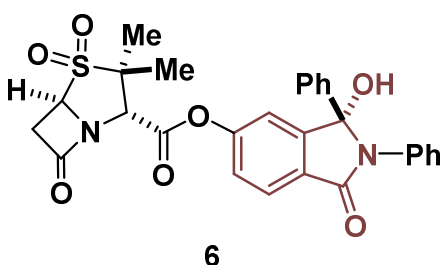


### $^1\text{H}$ and $^{13}\text{C}$ spectra data of compounds 5-16

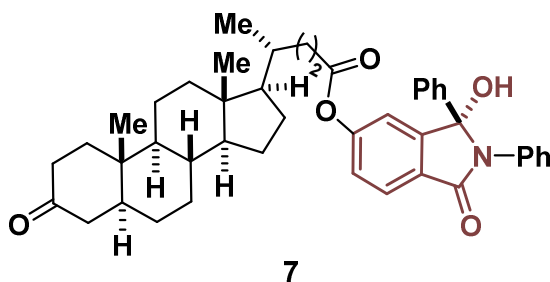


3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 4-(8,10,13-trimethyl-3,7,12-trioxohexadecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)pentanoate (5):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.83-7.77 (m, 1H), 7.46-7.42 (m, 2H), 7.39-7.37 (m, 2H), 7.25-7.16

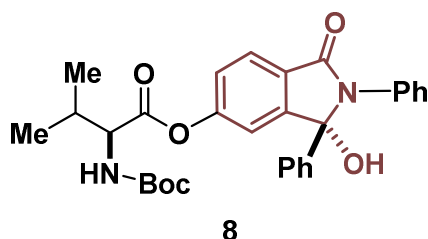
(m, 6H), 7.14-7.09 (m, 1H), 7.04-7.01 (m, 1H), 5.04-4.89 (d,  $J = 58.5$  Hz, 1H), 2.92-2.77 (m, 2H), 2.63-2.54 (m, 1H), 2.48-2.42 (m, 1H), 2.33-2.26 (m, 2H), 2.20-2.10 (m, 3H), 2.04-1.96 (m, 2H), 1.90-1.76 (m, 3H), 1.63-1.43 (m, 3H), 1.38-1.37 (d,  $J = 6.8$  Hz, 3H), 1.31-1.20 (m, 3H), 1.06-1.04 (d,  $J = 9.2$  Hz, 3H), 0.87-0.81 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  212.4, 209.6, 208.9, 172.2, 166.6, 154.7, 150.6, 138.5, 135.9, 128.6, 128.5, 128.4, 127.4, 126.4, 126.2, 125.8, 125.5, 125.2, 123.4, 116.5, 92.5, 57.0, 51.8, 49.0, 46.9, 45.6, 45.0, 42.8, 38.7, 36.4, 36.1, 35.3, 31.5, 30.3, 27.8, 25.2, 21.9, 18.7, 11.8; HRMS calculated for  $\text{C}_{45}\text{H}_{50}\text{NO}_7$  ( $\text{M} + \text{H}^+$ ): 716.3587, found: 716.3582. (White solid, 24.2 mg, 81% isolated yield)



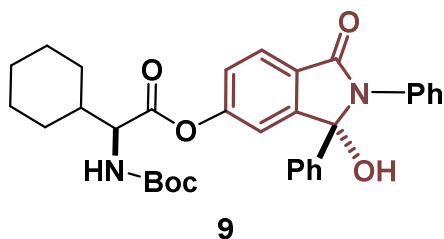
**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylate 4,4-dioxide (6):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80-7.78 (d,  $J = 8.4$  Hz, 1H), 7.42-7.35 (m, 4H), 7.27-7.25 (m, 4H), 7.22-7.13 (m, 3H), 7.07-7.04 (m, 1H), 4.67-4.65 (m, 1H), 4.60-4.58 (d,  $J = 8.8$  Hz, 1H), 4.37-4.35 (m, 1H), 3.51-3.48 (m, 2H), 1.69 (s, 3H), 1.55-1.54 (d,  $J = 4.0$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  173.4, 170.7, 166.2, 150.7, 137.9, 131.0, 128.9, 128.8, 128.7, 126.6, 126.2, 125.7, 125.6, 123.2, 122.9, 116.1, 115.9, 92.6, 63.3, 62.9, 61.2, 23.1, 20.6, 18.8; HRMS calculated for  $\text{C}_{28}\text{H}_{24}\text{N}_2\text{O}_7\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 555.1202, found: 555.1196. (Yellow solid, 19.4 mg, 63% isolated yield)



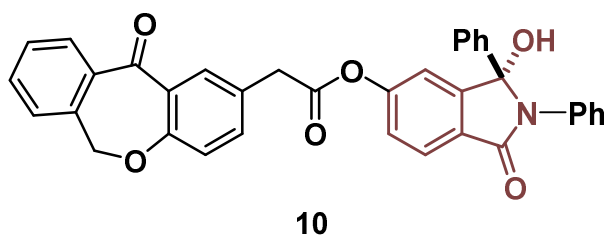
**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 4-(10,13-dimethyl-3-oxohexadecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)pentanoate (7):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.79-7.77 (d,  $J = 8.0$  Hz, 1H), 7.40-7.36 (m, 4H), 7.25-7.18 (m, 5H), 7.15-7.10 (m, 2H), 7.02-7.01 (d,  $J = 2.0$  Hz, 1H), 4.53 (s, 1H), 2.72-2.65 (t,  $J = 13.6$  Hz, 1H), 2.60-2.53 (m, 1H), 2.45-2.28 (m, 2H), 2.17-2.13 (m, 1H), 2.06-1.99 (m, 3H), 1.91-1.79 (m, 4H), 1.63-1.57 (m, 1H), 1.52-1.34 (m, 9H), 1.27-1.21 (m, 2H), 1.16-1.06 (m, 4H), 1.02 (s, 3H), 0.98-0.96 (d,  $J = 6.4$  Hz, 3H), 0.69-0.68 (d,  $J = 2.0$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  213.7, 172.4, 166.6, 154.7, 150.3, 138.4, 135.8, 128.7, 128.6, 128.5, 127.4, 126.5, 126.2, 125.7, 125.3, 123.6, 116.4, 92.5, 56.5, 56.0, 44.4, 42.9, 42.4, 40.8, 40.1, 37.3, 37.1, 35.6, 35.4, 35.0, 31.4, 30.9, 28.3, 26.7, 25.9, 24.2, 22.7, 21.3, 18.4, 12.2; HRMS calculated for  $\text{C}_{44}\text{H}_{51}\text{NO}_5\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 696.3665, found: 696.3659. (Yellow solid, 24.5 mg, 80% isolated yield)



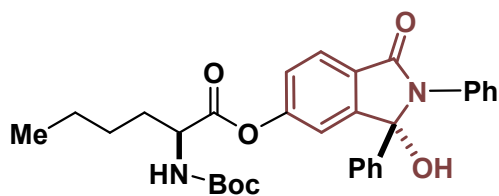
**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl (*tert*-butoxycarbonyl)-*L*-valinate (8):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.77-7.72 (t,  $J = 8.0$  Hz, 1H), 7.41-7.35 (m, 4H), 7.25-7.22 (m, 3H), 7.21-7.16 (m, 2H), 7.14-7.09 (m, 2H), 7.04-7.00 (m, 1H), 5.03-5.01 (d,  $J = 8.8$  Hz, 1H), 4.49 (s, 1H), 4.41-4.37 (m, 1H), 2.29-2.26 (m, 1H), 1.45 (s, 3H), 1.43 (s, 6H), 1.06-1.04 (m, 3H), 1.00-0.97 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.9, 169.0, 166.5, 155.7, 154.3, 150.4, 138.2, 135.7, 128.7, 128.6, 127.8, 126.5, 126.2, 125.7, 125.6, 125.4, 123.5, 123.4, 116.3, 116.2, 92.6, 80.4, 58.9, 31.2, 28.4, 19.2, 17.8; HRMS calculated for  $\text{C}_{30}\text{H}_{32}\text{N}_2\text{O}_6\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 539.2158, found: 539.2153. (White solid, 26.8 mg, 87% isolated yield)



**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 2-((*tert*-butoxycarbonyl)amino)-2-cyclohexylacetate (9):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.74-7.69 (m, 1H), 7.39-7.34 (m, 4H), 7.23-7.22 (m, 3H), 7.20-7.10 (m, 4H), 7.04-7.00 (dd,  $J_1 = 8.8$  Hz,  $J_2 = 1.6$  Hz, 1H), 5.03-5.01 (d,  $J = 8.8$  Hz, 1H), 4.74-4.58 (m, 1H), 4.40-4.34 (m, 1H), 1.88-1.68 (m, 6H), 1.46-1.42 (d,  $J = 9.0$  Hz, 9H), 1.31-1.09 (m, 5H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  176.5, 170.9, 166.6, 155.7, 154.4, 150.4, 138.3, 135.7, 128.6, 128.5, 127.7, 127.6, 126.5, 126.4, 125.7, 125.6, 125.3, 123.4, 116.3, 116.2, 92.6, 80.3, 58.7, 40.8, 40.7, 29.6, 28.4, 26.0; HRMS calculated for  $\text{C}_{33}\text{H}_{36}\text{N}_2\text{O}_6\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 579.2471, found: 579.2466. (Yellow oil, 26.2 mg, 85% isolated yield)



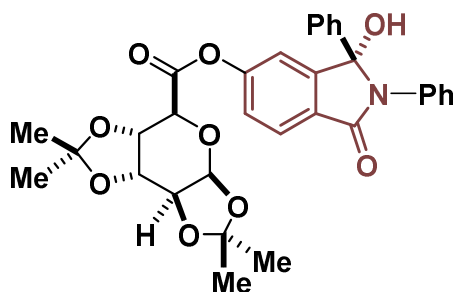
**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 2-(11-oxo-6,11-dihydrodibenzo[*b,e*]oxepin-2-yl)acetate (10):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.16-8.15 (d,  $J = 2.0$  Hz, 1H), 7.89-7.86 (m, 1H), 7.73-7.71 (d,  $J = 8.4$  Hz, 1H), 7.59-7.55 (m, 1H), 7.49-7.43 (m, 2H), 7.37-7.33 (m, 5H), 7.24-7.15 (m, 5H), 7.13-7.10 (m, 2H), 7.06-7.04 (d,  $J = 8.4$  Hz, 1H), 7.02-7.01 (d,  $J = 1.6$  Hz, 1H), 5.19 (s, 2H), 3.85 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  190.9, 169.7, 166.6, 160.9, 154.5, 150.3, 140.4, 138.3, 136.4, 135.7, 135.6, 133.0, 132.8, 129.6, 129.4, 128.6, 128.5, 128.4, 127.9, 127.6, 126.6, 126.4, 126.2, 125.7, 125.3, 125.2, 123.5, 121.5, 116.4, 92.6, 73.7, 40.2; HRMS calculated for  $\text{C}_{36}\text{H}_{25}\text{NO}_6\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 590.1580, found: 590.1574. (White solid, 27.1 mg, 88% isolated yield)



11

**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 2-((*tert*-butoxycarbonyl)amino)**

**hexanoate (11):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.78-7.69 (m, 2H), 7.41-7.31 (m, 4H), 7.25-7.19 (m, 4H), 7.18-7.09 (m, 3H), 7.04-7.01 (dd,  $J_1 = 8.0$  Hz,  $J_2 = 2.0$  Hz, 1H), 4.99-4.97 (d,  $J = 8.0$  Hz, 1H), 4.47-4.24 (m, 1H), 1.96-1.88 (m, 1H), 1.76-1.68 (m, 1H), 1.44-1.43 (d,  $J = 5.6$  Hz, 9H), 1.41-1.33 (m, 4H), 0.94-0.90 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  176.8, 171.8, 166.5, 154.4, 150.4, 130.3, 129.1, 128.7, 128.6, 127.6, 127.5, 126.5, 126.2, 125.7, 125.6, 125.4, 123.5, 116.2, 92.6, 80.4, 53.8, 32.1, 28.4, 27.6, 22.3, 13.9; HRMS calculated for  $\text{C}_{31}\text{H}_{34}\text{N}_2\text{O}_6\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 553.2315, found: 553.2309. (Yellow solid, 26.5 mg, 86% isolated yield)



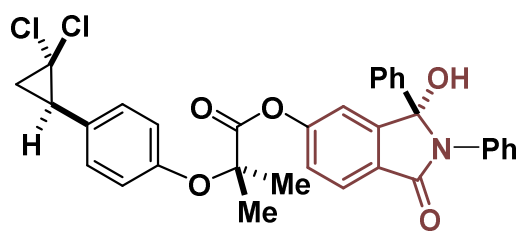
12

**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 2,2,7,7-tetramethyltetrahydro-5H-bis**

**([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-carboxylate (12):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76-7.72 (t,  $J = 7.6$  Hz, 1H), 7.43-7.39 (m, 2H), 7.37-7.34 (m, 3H), 7.25-7.16 (m, 5H), 7.13-7.08 (m, 2H), 5.69-5.68 (d,  $J = 5.2$  Hz, 1H), 4.73-4.62 (m, 3H), 4.44-4.42 (m, 1H), 1.57-1.55 (d,  $J = 6.4$  Hz, 3H), 1.49-1.44 (d,  $J = 18.0$  Hz, 3H), 1.36-1.34 (m, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  176.4, 166.7, 154.3, 138.3, 128.7, 128.5, 127.7, 126.4, 126.2, 125.7, 125.6, 125.4, 123.8, 123.7, 116.5, 110.6, 109.4, 96.6, 92.6, 72.3, 70.9, 70.2, 68.9,

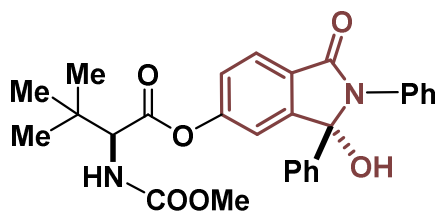


26.1, 26.0, 24.9, 24.8; HRMS calculated for C<sub>32</sub>H<sub>31</sub>NO<sub>9</sub>Na (M + Na<sup>+</sup>): 596.1897, found: 596.1893. (Yellow oil, 26.7 mg, 87% isolated yield)



13

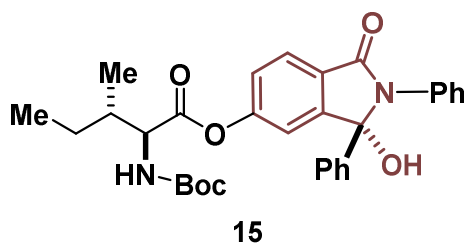
**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (13):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.68-7.65(d, *J* = 2.4 Hz, 1H), 7.37-7.31 (m, 4H), 7.25-7.22 (m, 3H), 7.18-7.09 (m, 5H), 6.92-6.86 (m, 3H), 2.86-2.81 (m, 1H), 1.98-1.93 (m, 1H), 1.80-1.75 (m, 1H), 1.71 (s, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 172.7, 166.6, 154.7, 154.4, 150.4, 138.2, 135.6, 130.0, 128.9, 128.6, 128.5, 127.7, 126.5, 126.2, 125.7, 125.6, 125.3, 123.4, 118.8, 116.2, 92.6, 79.3, 60.9, 34.8, 25.9, 25.7, 25.3; HRMS calculated for C<sub>33</sub>H<sub>27</sub>Cl<sub>2</sub>NO<sub>5</sub>Na (M + Na<sup>+</sup>): 610.1164, found: 610.1159. (Yellow oil, 27.7 mg, 90% isolated yield)



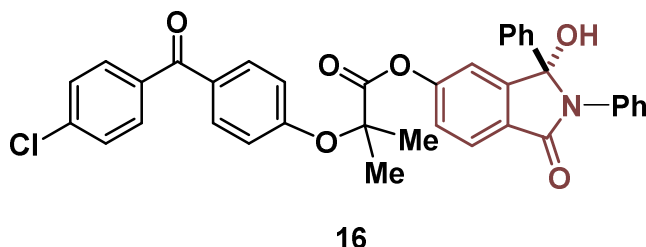
14

**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 2-((methoxycarbonyl)amino)-3,3-dimethylbutanoate (14):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.72-7.63 (m, 1H), 7.37-7.31 (m, 4H), 7.25-7.21 (m, 3H), 7.18-7.13 (m, 2H), 7.12-7.07 (m, 2H), 7.03-7.00 (m, 1H), 5.34-5.32 (d, *J* = 7.2 Hz, 1H), 4.91 (s, 1H), 4.33-4.31 (d, *J* = 8.8 Hz, 1H), 3.69 (s, 3H), 1.08-1.07 (d, *J* = 3.6 Hz, 9H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.2, 166.7, 166.6, 156.9, 154.1, 150.5, 138.2, 137.6, 135.6, 130.3, 128.6, 128.5, 127.8, 127.7, 126.5, 126.4, 126.3, 126.2, 125.8, 125.7, 125.3, 123.5, 123.4, 116.4, 116.2, 92.7, 62.4, 52.7, 342.9, 26.7;

HRMS calculated for  $C_{28}H_{28}N_2O_6Na$  ( $M + Na^+$ ): 511.1845, found: 511.1840. (White solid, 26.6 mg, 86% isolated yield)

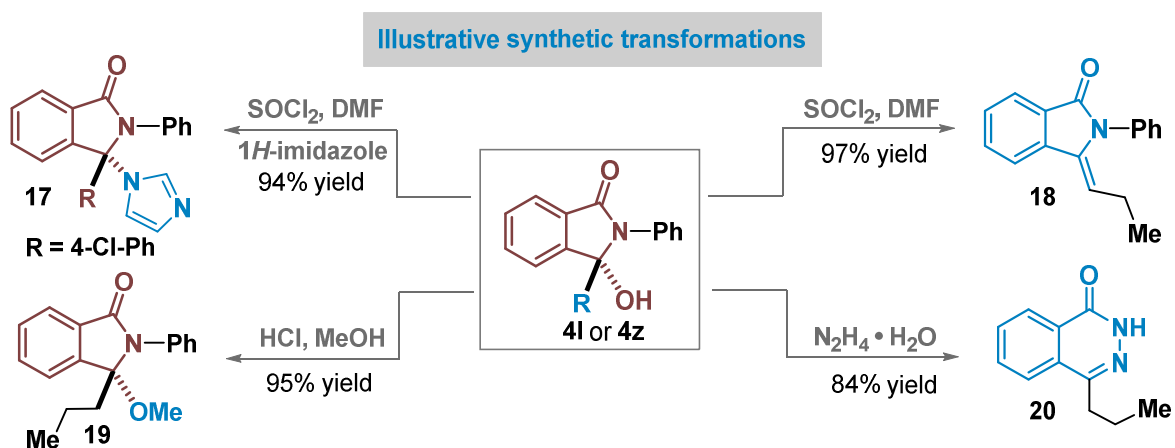


**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl (tert-butoxycarbonyl)-L-isoleucinate (15):**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.73-7.68 (m, 1H), 7.40-7.34 (m, 4H), 7.25-7.21 (m, 3H), 7.19-7.10 (m, 4H), 7.03-7.00 (dd,  $J_1 = 10.4$  Hz,  $J_2 = 2.0$  Hz, 1H), 5.04-5.02 (d,  $J = 8.8$  Hz, 1H), 4.43-4.40 (m, 1H), 1.97 (s, 1H), 1.55-1.47 (m, 1H), 1.45-1.43 (d,  $J = 7.2$  Hz, 9H), 1.30-1.22 (m, 1H), 1.03-1.00 (m, 3H), 0.99-0.94 (m, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  176.4, 170.9, 166.6, 154.4, 150.4, 138.2, 135.7, 135.6, 128.6, 128.5, 127.7, 127.6, 126.5, 126.4, 126.2, 125.7, 125.6, 125.3, 123.5, 116.3, 116.2, 92.6, 80.4, 58.3, 38.0, 28.4, 25.3, 15.8, 11.7; HRMS calculated for  $C_{31}H_{34}N_2O_6Na$  ( $M + Na^+$ ): 553.2315, found: 553.2309. (Yellow oil, 26.8 mg, 87% isolated yield)

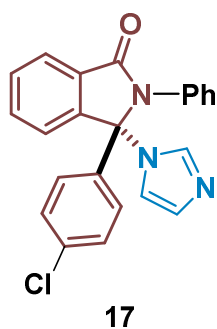


**3-hydroxy-1-oxo-2,3-diphenylisoindolin-5-yl 2-(4-(4-chlorobenzoyl)phenoxy)-2-methylpropanoate (16):**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.76-7.68 (m, 5H), 7.47-7.44 (m, 2H), 7.37-7.31 (m, 4H), 7.24-7.15 (m, 5H), 7.12-7.04 (m, 2H), 6.95-6.91 (m, 3H), 4.51 (s, 1H), 1.78 (s, 6H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  194.3, 172.3, 166.4, 159.3, 154.3, 150.5, 138.7, 138.1, 136.2, 135.6, 132.3, 131.3, 131.0, 128.8, 128.7, 128.6, 128.5, 127.9, 126.5, 126.2, 125.7, 125.4, 123.3, 117.5, 116.0, 92.6, 79.5, 25.6, 25.3; HRMS calculated for  $C_{37}H_{28}ClNO_6Na$  ( $M + Na^+$ ): 640.1503, found: 610.1497. (Colourless oil, 27.9 mg, 91% isolated yield)

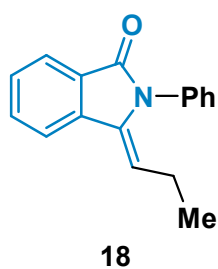
## Illustrative synthetic transformations



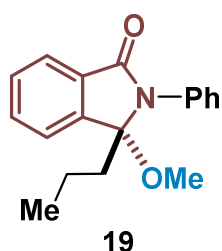
## $^1\text{H}$ and $^{13}\text{C}$ spectra data of compounds 17-20



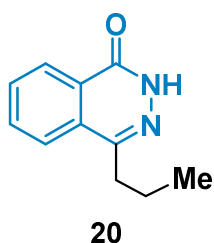
**3-(4-chlorophenyl)-3-(1H-imidazol-1-yl)-2-phenylisoindolin-1-one (17):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.05-8.03 (m, 1H), 7.65-7.63 (m, 2H), 7.40-7.34 (m, 5H), 7.32-7.26 (m, 4H), 7.01 (s, 1H), 6.87-6.85 (m, 2H), 6.63 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.8, 145.7, 137.5, 136.0, 135.4, 134.6, 133.8, 132.8, 130.8, 130.2, 129.6, 129.5, 129.3, 129.0, 128.7, 125.2, 123.5, 119.1, 83.9; HRMS calculated for  $\text{C}_{23}\text{H}_{16}\text{ClN}_3\text{ONa}$  ( $\text{M} + \text{Na}^+$ ): 408.0880, found: 408.0874. (Yellow oil, 32.4 mg, 94% isolated yield)



**2-phenyl-3-propylideneisoindolin-1-one (18):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.79-7.97 (d,  $J = 8.0$  Hz, 1H), 7.91-7.89 (d,  $J = 7.6$  Hz, 1H), 7.67-7.63 (m, 1H), 7.56-7.49 (m, 3H), 7.45-7.40 (m, 1H), 7.35-7.32 (m, 2H), 5.33-5.29 (t,  $J = 8.0$  Hz, 1H), 2.69-2.61 (m, 2H), 1.16-1.13 (t,  $J = 7.6$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.1, 136.7, 135.5, 135.0, 132.2, 130.2, 129.5, 129.0, 128.9, 128.2, 123.9, 123.4, 115.7, 20.8, 14.5; HRMS calculated for  $\text{C}_{17}\text{H}_{16}\text{NONa}$  ( $\text{M} + \text{H}^+$ ): 250.1232, found: 250.1226. (Yellow oil, 27.2 mg, 97% isolated yield)

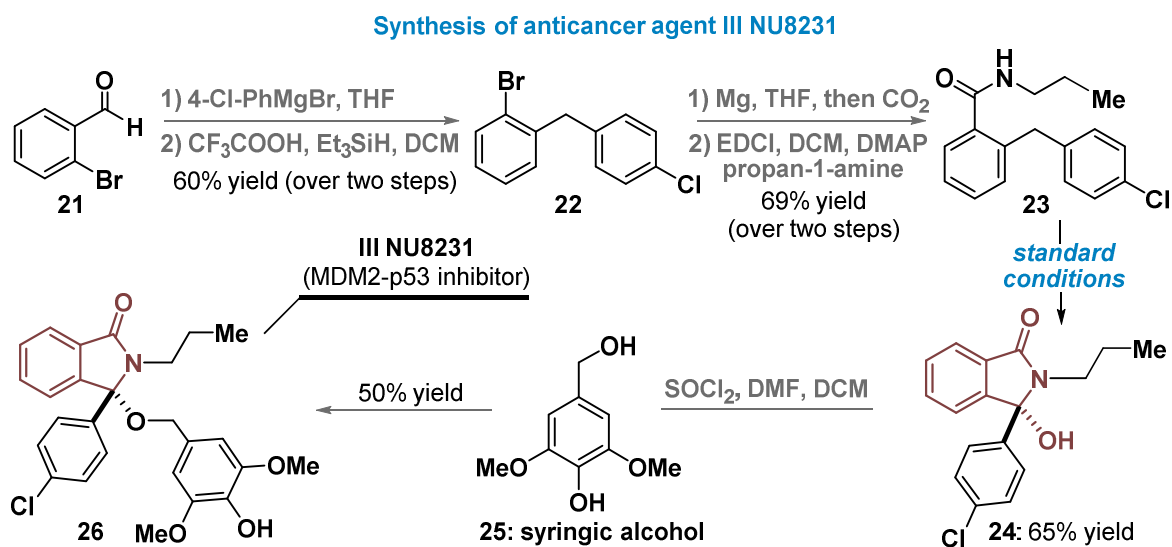


**3-methoxy-2-phenyl-3-propylisoindolin-1-one (19):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.94-7.92 (d,  $J = 8.0$  Hz, 1H), 7.68-7.64 (m, 1H), 7.62-7.59 (m, 2H), 7.58-7.54 (m, 1H), 7.50-7.43 (m, 3H), 7.31-7.26 (m, 1H), 3.01 (s, 3H), 2.05-1.98 (m, 2H), 1.06-0.96 (m, 1H), 0.78-0.69 (m, 1H), 0.66-0.63 (t,  $J = 7.2$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.4, 143.1, 136.1, 132.9, 132.6, 129.9, 129.1, 126.5, 125.1, 124.0, 122.1, 97.7, 50.2, 38.0, 16.6, 13.8; HRMS calculated for  $\text{C}_{18}\text{H}_{19}\text{NO}_2\text{Na}$  ( $\text{M} + \text{Na}^+$ ): 304.1313, found: 304.1308. (White solid, 29.9 mg, 95% isolated yield)

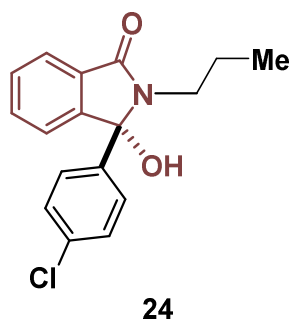


**4-propylphthalazin-1(2H)-one (20):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  10.42 (s, 1H), 8.49-8.47 (d,  $J = 7.6$  Hz, 1H), 7.85-7.83 (m, 2H), 7.80-7.76 (m, 1H), 2.95-2.91 (t,  $J = 7.2$  Hz, 2H), 1.87-1.77 (m, 2H), 1.08-1.04 (t,  $J = 7.6$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.5, 147.8, 133.5, 131.3, 130.0, 128.2, 127.1, 124.8, 34.2, 21.3, 14.1; HRMS calculated for  $\text{C}_{11}\text{H}_{13}\text{N}_2\text{O}$  ( $\text{M} + \text{H}^+$ ): 189.1028, found: 189.1022. (Yellow oil, 16.9 mg, 84% isolated yield)

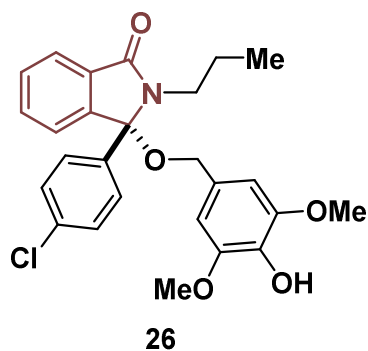
## Synthesis of anticancer agent III NU8231



### $^1\text{H}$ and $^{13}\text{C}$ spectra data of compounds 24 and 26

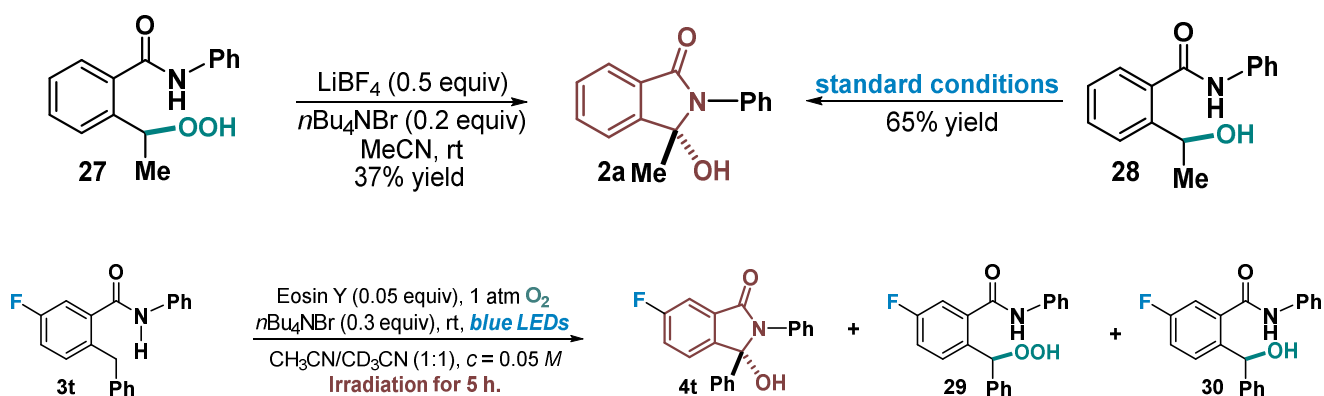


**3-(4-chlorophenyl)-3-hydroxy-2-propylisoindolin-1-one (24):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.73-7.71 (d,  $J = 7.2$  Hz, 1H), 7.59-7.50 (m, 2H), 7.42-7.40 (d,  $J = 7.6$  Hz, 1H), 7.34-7.32 (d,  $J = 8.2$  Hz, 1H), 7.26-7.24 (d,  $J = 7.2$  Hz, 1H), 7.19-7.18 (m, 1H), 3.35 (s, 1H), 3.34-3.28 (m, 1H), 2.91-2.84 (m, 1H), 1.48-1.38 (m, 1H), 0.78-0.75 (t,  $J = 8.0$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.1, 149.7, 140.0, 133.2, 133.0, 131.1, 129.9, 129.0, 128.4, 123.2, 123.0, 90.6, 41.2, 22.2, 12.1. These data are consistent with literature values, see: R. K. Dempster and F. A. Luzzio. *Tetrahedron Lett.* **2011**, 52, 4992. (White solid, 20.4 mg, 65% isolated yield)

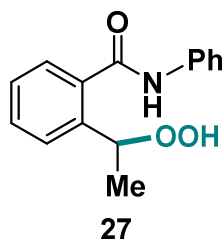


**3-(4-chlorophenyl)-3-((4-hydroxy-3,5-dimethoxybenzyl)oxy)-2-propylisoindolin-1-one (26):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92-7.90 (dd,  $J_1 = 5.2$  Hz,  $J_2 = 1.2$  Hz, 1H), 7.53-7.46 (m, 2H), 7.36-7.28 (m, 4H), 7.12-7.11 (dd,  $J_1 = 5.2$  Hz,  $J_2 = 1.2$  Hz, 1H), 6.46 (s, 2H), 4.17-4.14 (d,  $J = 7.6$  Hz, 1H), 3.93-3.90 (d,  $J = 7.6$  Hz, 1H), 3.87 (s, 6H), 3.34-3.07 (m, 2H), 1.57-1.34 (m, 2H), 0.83-0.80 (t,  $J = 7.6$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.4, 147.0, 145.3, 137.7, 134.6, 134.4, 132.6, 132.0, 130.0, 128.7, 128.5, 128.0, 123.6, 123.3, 104.5, 94.9, 65.4, 56.4, 41.6, 21.7, 11.9. These data are consistent with literature values, see: I. R. Hardcastle, S. U. Ahmed and H. Atkins. *J. Med. Chem.* **2006**, *49*, 6209. (White solid, 23.3 mg, 50% isolated yield)

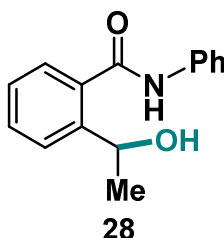
### Experiments on reaction mechanism



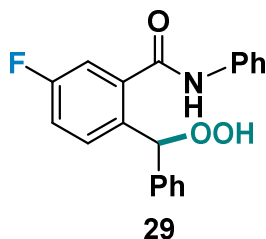
$^1\text{H}$ ,  $^{13}\text{C}$  and  $^{19}\text{F}$  spectra data of compounds 27-30



**2-(1-hydroperoxyethyl)-N-phenylbenzamide (27):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.12 (s, 1H), 8.24 (s, 1H), 7.60-7.58 (d,  $J = 8.0$  Hz, 2H), 7.52-7.45 (m, 3H), 7.34-7.31 (m, 3H), 7.16-7.13 (t,  $J = 7.6$  Hz, 1H), 5.46-5.41 (m, 1H), 1.47-1.46 (d,  $J = 6.4$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 139.1, 137.8, 136.3, 130.8, 129.2, 128.4, 127.8, 127.1, 124.9, 120.2, 80.5, 29.9; HRMS calculated for  $\text{C}_{15}\text{H}_{15}\text{NO}_3$  ( $\text{M} + \text{Na}^+$ ): 280.0950, found: 280.0944. (White solid, 15.1 mg, 44% isolated yield)

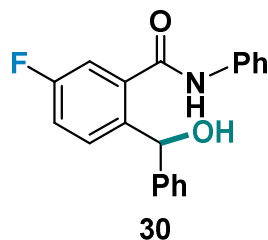


**2-(1-hydroxyethyl)-N-phenylbenzamide (28):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.26 (s, 1H), 7.64-7.61 (t,  $J = 7.2$  Hz, 3H), 7.57-7.48 (m, 2H), 7.40-7.36 (t,  $J = 8.4$  Hz, 3H), 7.20-7.16 (t,  $J = 7.6$  Hz, 1H), 5.11-5.06 (m, 1H), 3.92 (s, 1H), 1.60-1.59 (d,  $J = 6.4$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.4, 143.4, 137.8, 131.4, 129.3, 128.0, 127.9, 127.0, 125.8, 125.0, 120.3, 68.0, 22.1; HRMS calculated for  $\text{C}_{15}\text{H}_{15}\text{NO}_2$  ( $\text{M} + \text{Na}^+$ ): 264.1000, found: 264.0995. (White solid, 15.4 mg, 82% isolated yield)



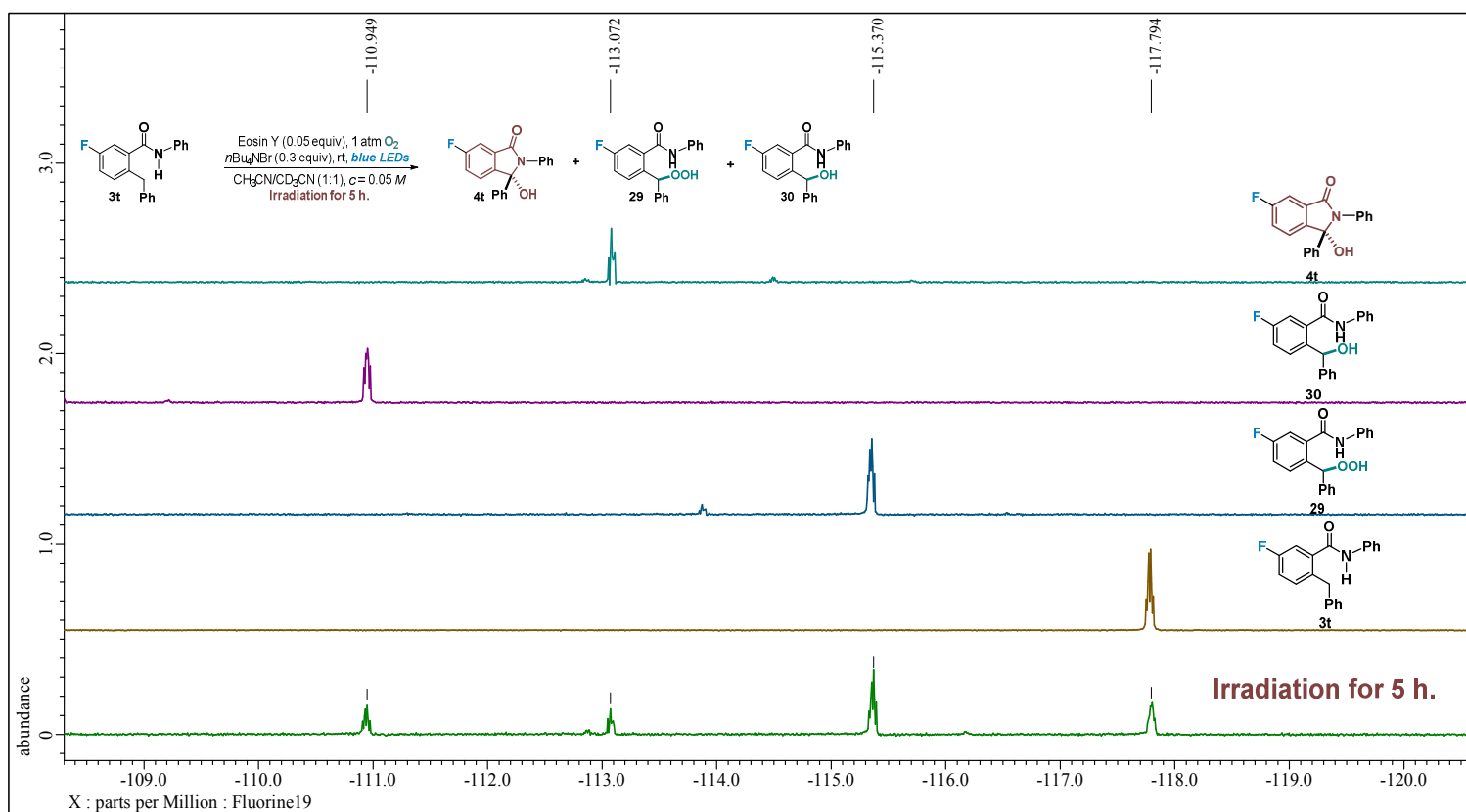
**5-fluoro-2-(hydroperoxy(phenyl)methyl)-N-phenylbenzamide (29):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.06 (s, 1H), 7.60-7.58 (d,  $J = 8.0$  Hz, 2H), 7.41-7.34 (m, 8H), 7.31-7.28 (m, 1H), 7.19-7.10 (m, 2H), 6.50 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.2, 139.1,

137.9, 137.5, 131.5, 131.4, 129.2, 129.0, 128.8, 128.7, 128.6, 127.4, 126.8, 126.1, 125.1, 120.3, 117.7, 117.5, 115.8, 115.6, 85.8;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -115.4; HRMS calculated for  $\text{C}_{20}\text{H}_{16}\text{FNO}_3$  ( $\text{M} + \text{Na}^+$ ): 360.1012, found: 360.1006. (White solid, 13.3 mg, 40% isolated yield)



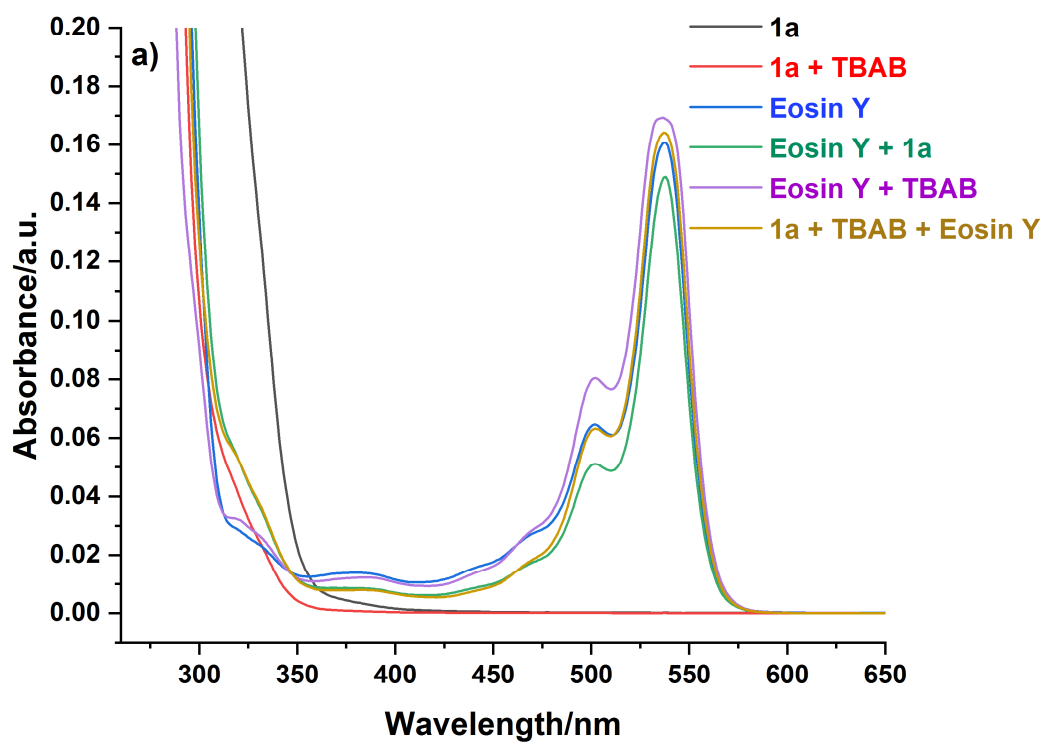
**5-fluoro-2-(hydroxy(phenyl)methyl)-N-phenylbenzamide (30):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.94 (s, 1H), 7.42-7.38 (m, 2H), 7.36-7.27 (m, 8H), 7.25-7.18 (m, 2H), 7.17-7.10 (m, 1H), 6.06 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.1, 142.0, 138.7, 137.1, 131.9, 131.8, 129.2, 128.8, 128.6, 128.3, 127.5, 126.5, 126.1, 125.3, 124.8, 124.7, 120.6, 117.9, 117.7, 115.3, 115.1, 73.6;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -110.9; HRMS calculated for  $\text{C}_{20}\text{H}_{16}\text{FNO}_2$  ( $\text{M} + \text{Na}^+$ ): 344.1063, found: 344.1057. (White solid, 15.2 mg, 80% isolated yield)

Reaction progress of **3t** monitored by *in situ*  $^{19}\text{F}$ -NMR





## UV-Vis absorption experiments



**Figure 1.** UV-Vis absorption spectra of the individual reaction components and the reaction mixtures

## Fluorescence quenching experiments

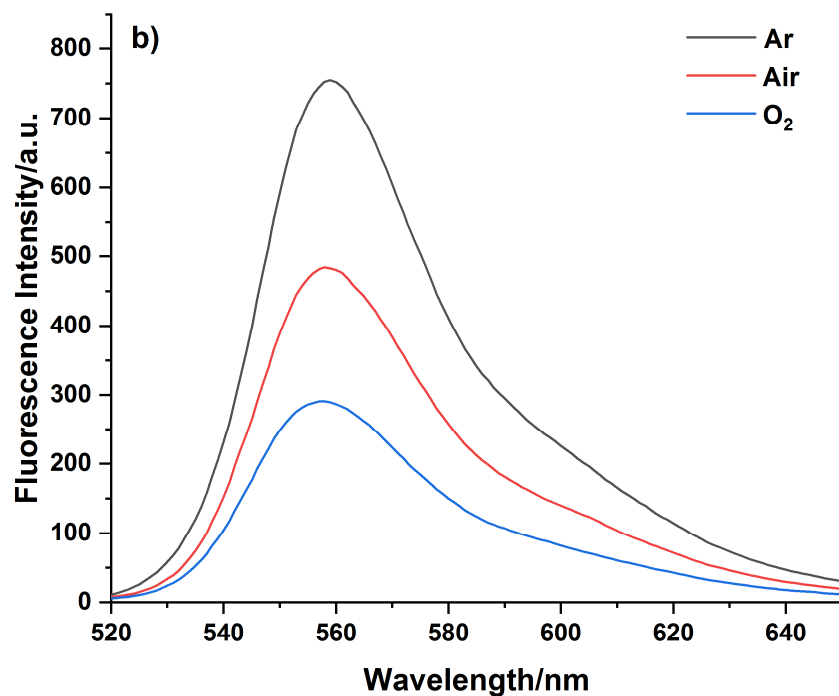


Figure 2. Fluorescence spectra of Eosin Y with different concentrations of O<sub>2</sub>

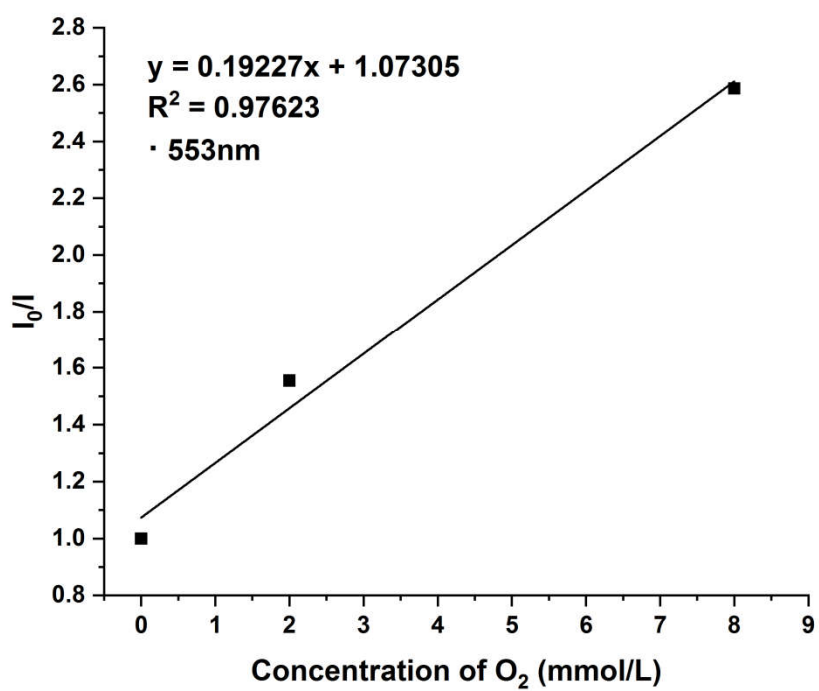


Figure 3. Stern Volmer plots of O<sub>2</sub>

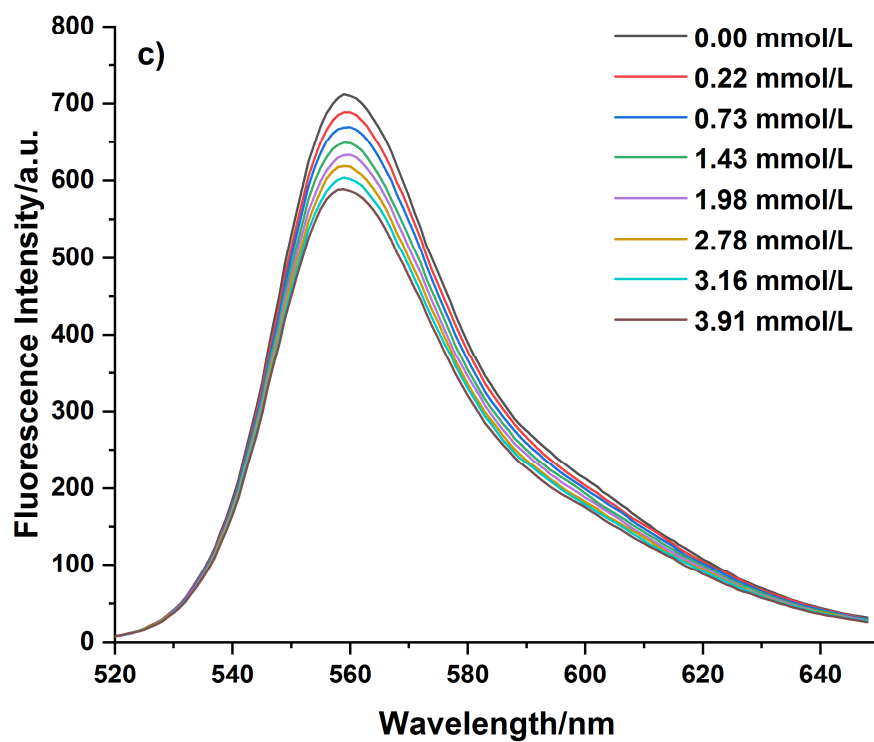


Figure 4. Fluorescence spectra of Eosin Y with different concentrations of **1a**

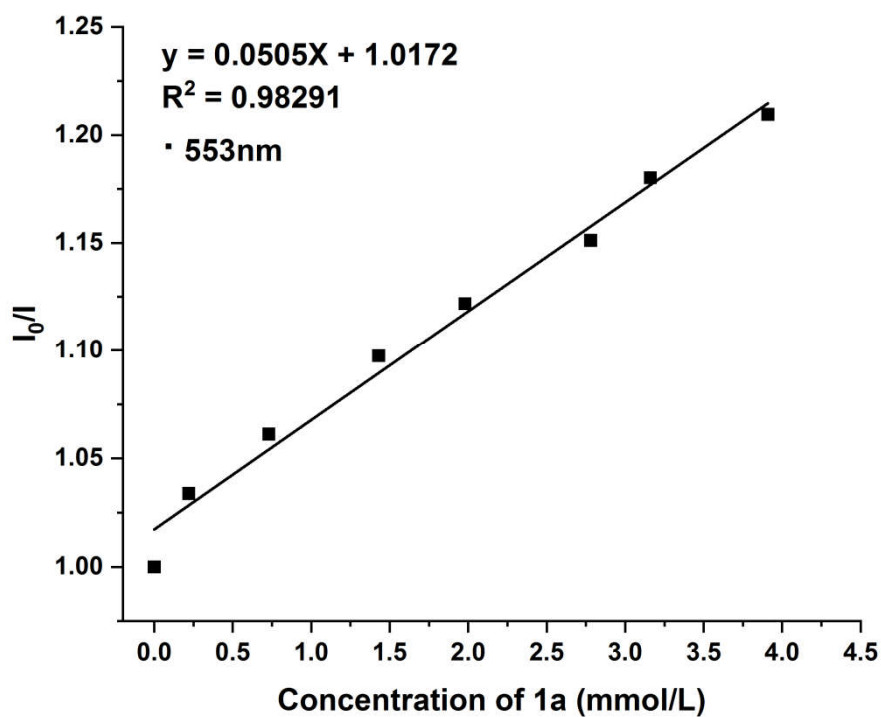


Figure 5. Stern Volmer plots of the substrate **1a**

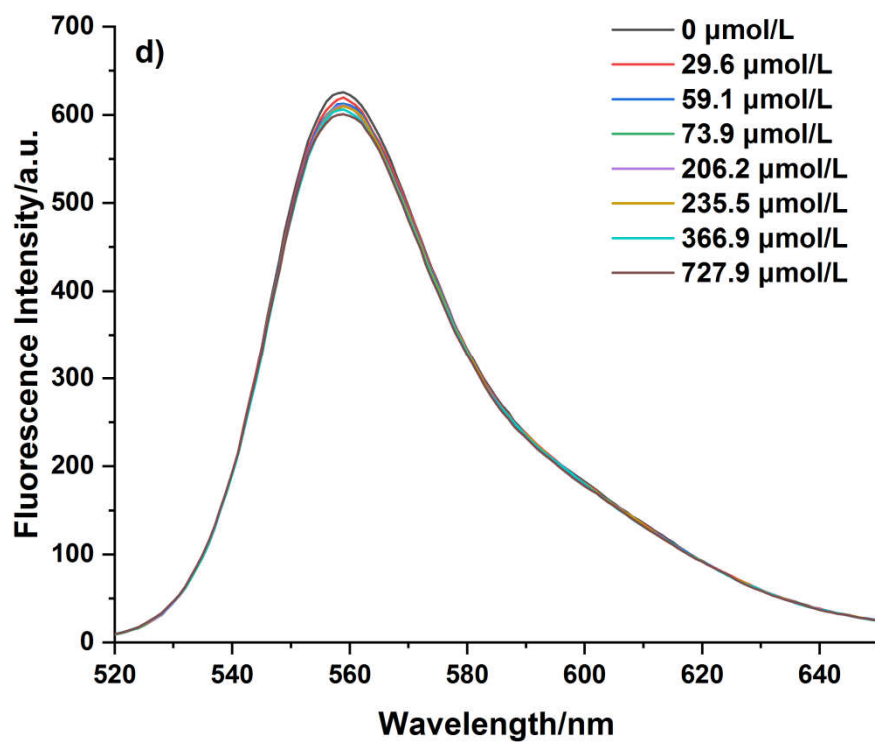


Figure 6. Fluorescence spectra of Eosin Y with different concentrations of  $n\text{Bu}_4\text{NBr}$

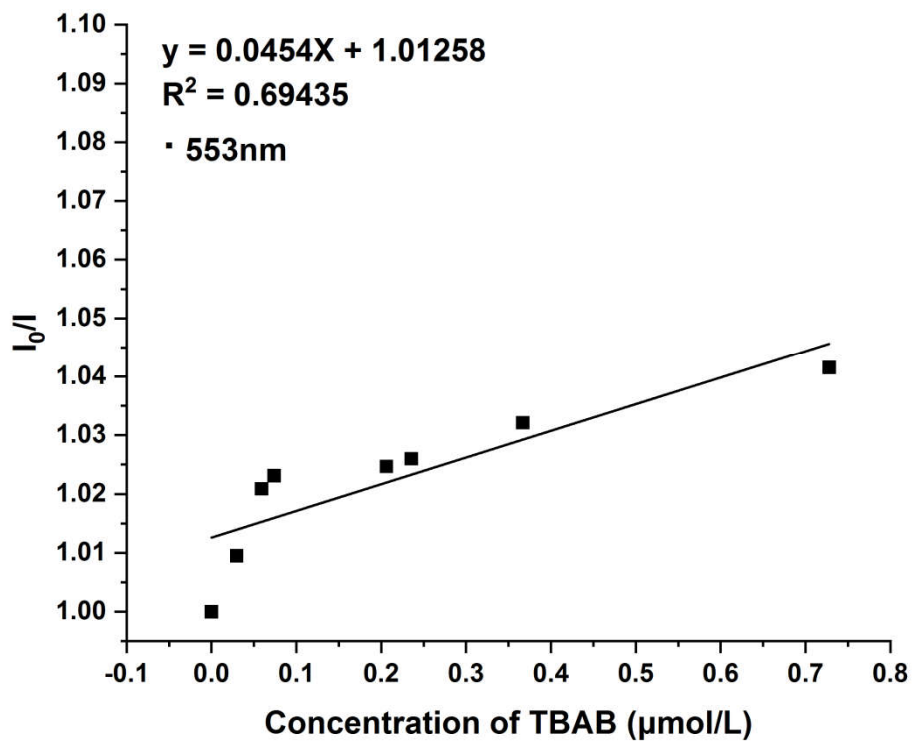


Figure 7. Stern Volmer plots of the TBAB

### CV curve of the substrate 1a

Cyclic voltammetry (CV) was performed using an Epsilon electrochemical workstation (a BASi three-electrode cell system): glassy carbon electrode as the working electrode, Pt wire as the counter electrode, Ag/AgCl (KCl, 3 M) electrode as the reference electrode, and ferrocenium/ferrocene ( $\text{Fc}^+/\text{Fc}$ ) as the internal standard. Scan rate:  $100 \text{ mV s}^{-1}$  (in the range  $-0.7$  to  $+2.3 \text{ V}$ ).  $n\text{Bu}_4\text{NPF}_6$  (0.1 M MeCN) was used as the supporting electrolyte.

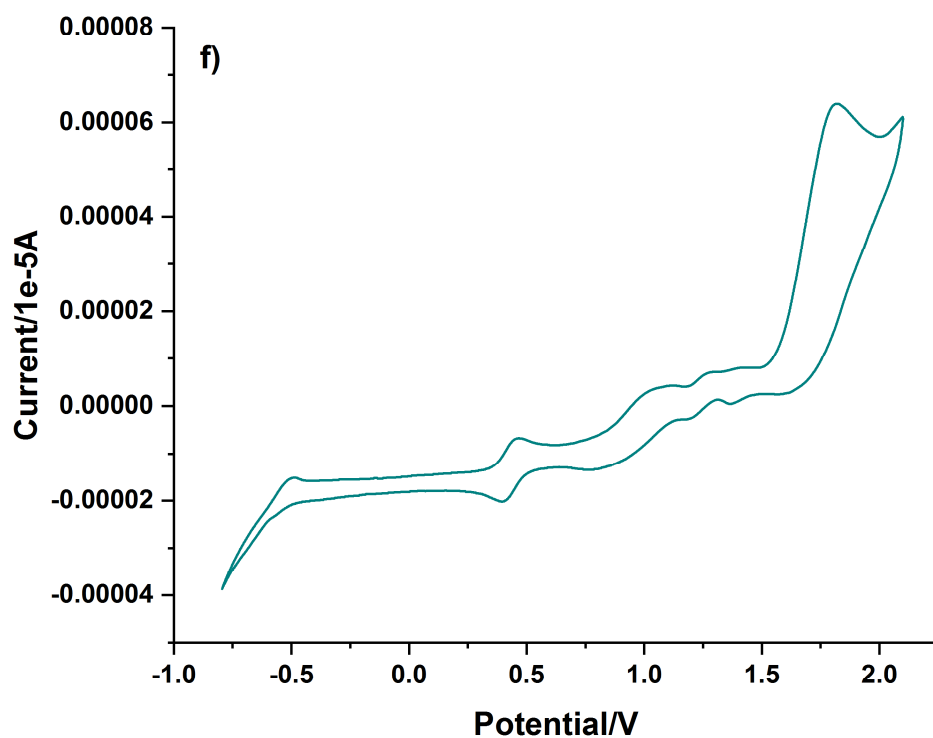


Figure 8. Cyclic voltammetry of the substrate 1a

## EPR experiments

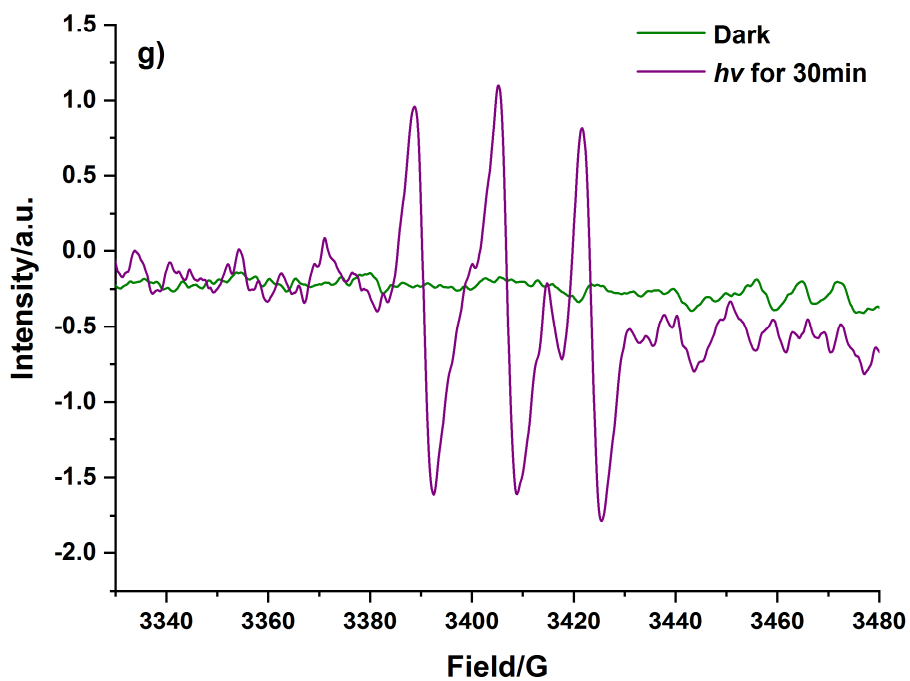


Figure 9. EPR experiments on reaction of **1a** by adding TEMP

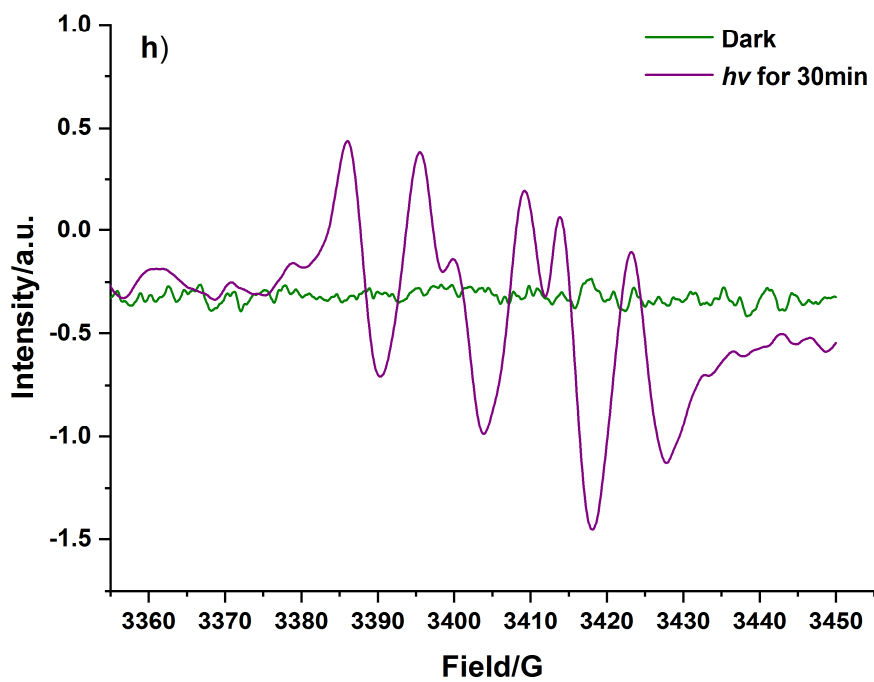


Figure 10. EPR experiments on reaction of **1a** by adding DMPO

# Copies of $^1\text{H}$ NMR, $^{13}\text{C}$ NMR and $^{19}\text{F}$ NMR spectra

