Supporting information

Ni(acac)$_2$ catalyzed Addition Reactions of Aryl- and Alkylboronic Acids to Tryptanthrins

Wei-Long Chen, Chong-Xing Liu, Hong-Ping Xiao,* and Jun Jiang*

College of Chemistry and Materials Science, Wenzhou University, Wenzhou 325035, PR China.

E-mail: hp_xiao@126.com; junjiang@wzu.edu.cn

General: NMR spectra were recorded on Brucker-500 MHz spectrometers. HRMS (Micromass GCT-MS) spectra were recorded on BRUKER micrOTOF-Q. Infrared spectra were recorded on a Nicolet MX-1E FT-IR spectrometer. Solvents for the column chromatography were distilled before use. Tryptanthrin and its derivatives were synthesized according to literature.$^{[1]}$ Ni(acac)$_2$ was purchased from Strem and used without further purification. All other chemicals were purchased from J&K or Afla Aesar and used as received.

Catalytic Addition Reactions:

General Procedure for the Ni(acac)$_2$ catalyzed 1,2-addition involving arylboronic acids

Tryptanthrins (0.2 mmol), aryl boronic acids (1 mmol), Ni(acac)$_2$ (0.01 mmol), potassium carbonate (0.4 mmol) and 2 mL cyclopentyl methyl ether (CPME) were added to a Schlenk tube. The resulting mixture was stirred at the corresponding temperature for specific time until the reaction was completed, then the solvent was evaporated under reduced pressure. The crude mixture was purified through flash column chromatography.
on a silica gel (Rf= 0.2-0.4, CH2Cl2; Eluent: Petroleum ether: dichloromethane= 1:3 to dichloromethane: ethyl acetate= 25:1) to yield the targeting products.

**Table 1 Ligand-screening of Catalytic Addition of Arylboronic Acids to Tryptanthrin.**

<table>
<thead>
<tr>
<th>L1</th>
<th>50% yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>L2</td>
<td>51% yield</td>
</tr>
<tr>
<td>L3</td>
<td>60% yield</td>
</tr>
<tr>
<td>L4</td>
<td>58% yield</td>
</tr>
<tr>
<td>L5</td>
<td>62% yield</td>
</tr>
<tr>
<td>L6</td>
<td>51% yield</td>
</tr>
<tr>
<td>L7</td>
<td>trace</td>
</tr>
<tr>
<td>L8</td>
<td>NR</td>
</tr>
<tr>
<td>L9</td>
<td>63% yield</td>
</tr>
</tbody>
</table>

Conditions: The reaction of 1a (0.2 mmol), 2a (1.0 mmol) and K2CO3 (0.4 mmol) was performed in 2mL CPME for 5 hours at 100 °C in the presence of 5 mol % Ni(acac)2 and 6 mol % ligand; given are isolated yields.

**General Procedure for the Ni(acac)2 catalyzed 1,2-addition of alkylboronic acids**
Tryptanthrins (0.2 mmol), alkyl boric acids (1 mmol), Ni(acac)$_2$ (0.03 mmol), potassium carbonate (0.4 mmol) and 2 mL chlorobenzene were added to a Schlenk tube. Then the resulting mixture was stirred at 120 °C for specific time until the reaction was completed. The reaction mixture was purified through flash column chromatography on a silica gel (R$_f$= 0.2-0.3, CH$_2$Cl$_2$; Eluent: Petroleum ether: dichloromethane= 1:20 to pure dichloromethane) to yield the targeting products.

### Table 2 Ligand-screening of Catalytic Addition of Alkylboronic Acids to Tryptanthrin.

<table>
<thead>
<tr>
<th>Ligand</th>
<th>Isolated Yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>L1</td>
<td>41% yield</td>
</tr>
<tr>
<td>L2</td>
<td>53% yield</td>
</tr>
<tr>
<td>L3</td>
<td>56% yield</td>
</tr>
<tr>
<td>L4</td>
<td>62% yield</td>
</tr>
<tr>
<td>L5</td>
<td>25% yield</td>
</tr>
<tr>
<td>L6</td>
<td>36% yield</td>
</tr>
<tr>
<td>L7</td>
<td>28% yield</td>
</tr>
<tr>
<td>L8</td>
<td>11% yield</td>
</tr>
<tr>
<td>L9</td>
<td>55% yield</td>
</tr>
</tbody>
</table>

Conditions: The reaction of 1a (0.2 mmol), 4a (1.0 mmol) and K$_2$CO$_3$ (0.4 mmol) was performed in 2mL PhCl for 24 hours at 120 °C in the presence of 15 mol % Ni(acac)$_2$ and 18 mol % ligand; given are isolated yields.

**Physical data:**

3
6-Hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3a): white solid; 90% yield; 
$^1$H NMR (500 MHz, DMSO) $\delta$ (ppm) 7.10 (s, 1H), 7.29-7.43 (m, 7H), 7.55 (m, 1H), 7.62 (m, 1H), 7.72 (m, 1H), 7.84-7.87 (m, 1H), 8.32 (d, $J= 7.7$ Hz, 1H), 8.53 (d, $J= 7.9$ Hz, 1H); $^{13}$C NMR (126 MHz, DMSO) $\delta$ (ppm) 78.98, 116.34, 121.44, 125.00, 125.63, 126.41, 127.02, 127.54, 127.65, 127.68, 128.17, 129.85, 134.75, 135.94, 138.56, 141.79, 147.08, 158.96, 161.68. IR (KBr) γ 3338, 1655, 1466, 1358, 1186, 757, 699 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{15}$N$_2$O$_2$)$^+$ requires m/z 327.1128, found m/z 327.1124.

7-Bromo-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3b): white solid; 80% yield; $^1$H NMR (500 MHz, DMSO) $\delta$ (ppm) 7.06 (s, 1H), 7.28-7.33 (m, 3H), 7.38-7.40 (m, 2H), 7.52-7.54 (m, 2H), 7.59-7.62 (m, 1H), 7.68-7.69 (m, 1H), 7.83-7.86 (m, 1H), 8.30 (d, $J= 7.7$ Hz, 1H), 8.61(d, $J= 5.6$ Hz, 1H); $^{13}$C NMR (126 MHz, DMSO) $\delta$(ppm) 80.02, 115.54, 119.42, 121.22, 125.94, 126.48, 127.64, 127.95, 131.09, 131.88, 133.61, 134.97, 139.23, 140.83, 146.98, 158.84, 161.17; IR (KBr) γ 3370, 1655, 1452, 1357, 1169, 771, 709 cm$^{-1}$; HRMS exact mass calcd for (C$_{22}$H$_{14}$BrN$_2$O$_2$)$^+$ requires m/z 405.0233, found m/z 405.0230.
**8-Fluoro-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3c):** white solid; 85% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.23-7.43 (m, 8H), 7.63-7.73 (m, 2H), 7.86 (m, 1H), 8.31-8.32 (m, 1H), 8.53 (m, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 78.84, 112.14, 112.34, 116.48, 116.67, 118.02, 118.08, 121.37, 125.63, 126.38, 127.67, 127.91, 128.27, 134.81, 138.26, 138.32, 141.17, 146.98, 158.78, 159.68, 161.51, 161.62; IR (KBr) γ 3370, 1656, 1477, 1364, 1183, 772, 695 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{14}$FN$_2$O$_2$)$^+$ requires m/z 345.1034, found m/z 345.1043.

![8-Fluoro-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3c) structure](image)

**8-Chloro-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3d):** white solid; 74% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.23-7.43 (m, 7H), 7.64-7.74 (m, 3H), 7.87 (m, 1H), 8.32 (d, J= 6.9 Hz, 1H), 8.51 (d, J=7.8 Hz,1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 78.80, 117.99, 121.35, 124.83, 125.63, 126.43, 127.72, 127.93, 128.29, 129.88, 131.01, 134.91, 137.31, 138.10, 141.10, 146.99, 158.86, 161.16; IR (KBr) γ 3358, 1659, 1644, 1355, 1182, 775, 701 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{14}$ClN$_2$O$_2$)$^+$ requires m/z 361.0738, found m/z 361.0744.

![8-Chloro-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3d) structure](image)

**8-Bromo-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3e):** white solid; 65% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.23-7.47 (m, 7H), 7.63-7.87 (m, 4H), 8.32-8.45 (m, 2H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 78.75, 118.37, 119.05, 121.36, 125.63, 126.44, 127.62, 127.72, 127.74, 127.94, 128.30, 132.77, 134.92, 137.72, 138.35, 141.12, 147.00, 158.88, 161.05; IR (KBr) γ 3352, 1660, 1463, 1352, 1182, 774, 700 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{14}$BrN$_2$O$_2$)$^+$ requires m/z 405.0233, found m/z 405.0239.
6-Hydroxy-8-methyl-6H-indolo[2,1-b]quinazolin-12-one (3f): white solid; 82% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 2.33 (s, 3H), 7.07 (s, 1H), 7.16 (s, 1H), 7.28-7.41 (m, 6H), 7.61-7.63 (m, 1H), 7.70-7.72 (m, 1H), 7.83-7.85 (m, 1H), 8.31 (d, J = 7.3 Hz, 1H), 8.39 (d, J = 7.7 Hz, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 20.77, 78.99, 116.10, 121.42, 125.24, 125.57, 126.34, 127.49, 127.63, 128.17, 130.20, 134.64, 136.04, 136.27, 136.58, 141.86, 147.08, 158.76, 161.88; IR (KBr) γ 3355, 1658, 1465, 1358, 1190, 776, 704 cm$^{-1}$; HRMS exact mass calcd for (C$_{22}$H$_{17}$N$_2$O$_2$)$^+$ requires m/z 341.1285, found m/z 341.1298.

6-Hydroxy-8-methoxy-6H-indolo[2,1-b]quinazolin-12-one (3g): white solid; 76% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 3.76 (s, 3H), 6.87 (m, 1H), 7.13 (m, 2H), 7.33-7.40 (m, 5H), 7.61-7.71 (m, 2H), 7.84 (m, 1H), 8.30-8.43 (m, 2H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 55.66, 79.07, 79.07, 110.50, 114.87, 117.42, 121.39, 125.58, 126.25, 127.46, 127.61, 127.74, 128.21, 131.86, 134.55, 137.57, 141.70, 147.01, 158.33, 158.54, 161.81; IR (KBr) γ 3336, 1656, 1482, 1466, 1301, 1187, 779, 703 cm$^{-1}$; HRMS exact mass calcd for (C$_{22}$H$_{17}$N$_2$O$_3$)$^+$ requires m/z 357.1234, found m/z 357.1237.
6-Hydroxy-8-nitro-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3h): white solid; 84% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.38 (m, 4H), 7.51 (m, 2H), 7.66 (m, 1H), 7.75-7.76 (m, 1H), 7.90 (m, 1H), 8.07 (m, 1H), 8.34-8.35 (m, 1H), 8.49-8.50 (m, 1H), 8.72-8.73 (m, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 78.49, 117.10, 119.97, 121.29, 125.80, 126.42, 126.66, 127.84, 128.03, 128.15, 128.36, 135.31, 137.37, 140.62, 143.31, 145.75, 146.75, 159.14, 161.07; IR (KBr) γ 3368, 1669, 1600, 1463, 1361, 1175, 777, 703 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{14}$N$_3$O$_4$)$^+$ requires m/z 372.0979, found m/z 372.0981.

9-Chloro-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3i): white solid; 89% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.15 (s, 1H), 7.29-7.37 (m, 4H), 7.43-7.44 (m, 3H), 7.62-7.65 (m, 1H), 7.72-7.73 (m, 1H), 7.86-7.89 (m, 1H), 8.32 (m, 1H), 8.52 (m, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 78.65, 116.22, 121.27, 125.68, 126.47, 126.51, 126.86, 127.74, 127.77, 127.85, 128.21, 133.80, 134.86, 135.01, 139.55, 141.19, 146.94, 158.98, 161.31; IR (KBr) γ 3360, 1666, 1468, 1298, 1183, 779, 693 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{14}$ClN$_2$O$_2$)$^+$ requires m/z 361.0738, found m/z 361.0732.

9-Bromo-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3j): white solid; 86% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.15 (m, 1H), 7.31-7.33 (m, 4H), 7.43 (m, 2H), 7.56-7.73 (m, 3H), 7.87 (m, 1H), 8.31-8.32 (m, 1H), 8.66 (m, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 78.71, 119.00, 121.27, 122.09, 125.67, 126.47, 126.83,
10-Fluoro-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3k): white solid; 67\% yield; \(^1\)H NMR (500 MHz, DMSO) \(\delta\) (ppm) 7.14-7.17 (m, 2H), 7.28-7.35 (m, 3H), 7.37-7.43 (m, 4H), 7.59-7.62 (m, 1H), 7.67-7.69 (m, 1H), 7.83-7.86 (m, 1H), 8.30 (d, J = 7.8 Hz, 1H); \(^{13}\)C NMR (126 MHz, DMSO) \(\delta\) (ppm) 79.20, 118.62, 118.80, 121.13, 121.16, 121.69, 121.71, 124.42, 124.51, 125.60, 126.81, 127.29, 127.65, 127.79, 128.20, 128.97, 129.02, 134.81, 139.63, 141.61, 146.76, 147.75, 149.79, 156.86, 161.46; IR (KBr) \(\gamma\) 3364, 1675, 1473, 1354, 1190, 772, 693 cm\(^{-1}\); HRMS exact mass calcd for (C\(_{21}\)H\(_{14}\)BrN\(_2\)O\(_2\))\(^{+}\) requires \(m/z\) 405.0233, found \(m/z\) 405.0233.

\[ \text{R} \]

10-Chloro-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3l): white solid; 76\% yield; \(^1\)H NMR (500 MHz, DMSO) \(\delta\) (ppm) 7.16 (s, 1H), 7.32-7.44 (m, 7H), 7.62-7.65 (m, 1H), 7.72-7.73 (m, 1H), 7.83-7.86 (m, 1H), 8.31-8.32 (m, 1H), 8.52 (m, 1H); \(^{13}\)C NMR (126 MHz, DMSO) \(\delta\) (ppm) 78.65, 116.22, 121.27, 125.68, 126.47, 126.51, 126.86, 127.74, 127.78, 127.86, 128.21, 133.79, 134.86, 135.02, 139.55, 141.19, 146.93, 158.99, 161.32; IR (KBr) \(\gamma\) 3363, 1653, 1455, 1298, 1150, 756, 695 cm\(^{-1}\); HRMS exact mass calcd for (C\(_{21}\)H\(_{14}\)ClN\(_2\)O\(_2\))\(^{+}\) requires \(m/z\) 361.0738, found \(m/z\) 361.0742.
3-Chloro-6-hydroxy-6-phenyl-6H-indolo[2,1-b]quinazolin-12-one (3m): white solid; 83% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.11 (s, 1H), 7.28-7.39 (m, 5H), 7.42-7.44 (m, 2H), 7.54-7.57 (m, 1H), 7.63-7.65 (m, 1H), 7.81 (m, 1H), 8.30 (d, J = 8.5 Hz, 1H), 8.50 (d, J = 8.0 Hz, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 79.09, 116.37, 120.37, 125.00, 125.65, 126.89, 127.19, 127.76, 128.17, 128.36, 129.91, 135.89, 138.35, 139.36, 141.48, 148.22, 158.38, 163.21; IR (KBr) γ 3370, 1660, 1466, 1314, 1177, 760, 705 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{14}$ClN$_2$O$_2$)$^+$ requires m/z 361.0738, found m/z 361.0739.

6-(4-Fluoro-phenyl)-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (3n): white solid; 85% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.15 (m, 3H), 7.37-7.86 (m, 8H), 8.32 (m, 1H), 8.52 (m, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 78.56, 114.84, 115.01, 116.38, 121.49, 124.96, 126.40, 127.04, 127.57, 127.65, 127.89, 127.95, 129.98, 134.74, 135.59, 137.91, 138.58, 147.04, 158.95, 160.69, 161.42, 162.63; IR (KBr) γ 3385, 1667, 1460, 1316, 1156, 842, 756, 695 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{14}$FN$_2$O$_2$)$^+$ requires m/z 345.1034, found m/z 345.1048.
6-(4-Chloro-phenyl)-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (3o): white solid; 53% yield; \(^1\)H NMR (500 MHz, DMSO) \(\delta\) (ppm) 7.20 (s, 1H), 7.35-7.40 (m, 4H), 7.44-7.46 (m, 2H), 7.55-7.58 (m, 1H), 7.61-7.64 (m, 1H), 7.71-7.73 (m, 1H), 7.85-7.88 (m, 1H), 8.32 (d, \(J = 7.9\) Hz, 1H), 8.52 (d, \(J = 8.0\) Hz, 1H); \(^1\)C NMR (126 MHz, DMSO) \(\delta\) (ppm) 78.57, 116.40, 121.50, 124.96, 126.41, 127.08, 127.65, 127.72, 128.16, 130.07, 132.47, 134.76, 135.38, 138.63, 140.71, 147.00, 158.94, 161.25; IR (KBr) \(\gamma\) 3392, 1664, 1466, 1294, 1184, 828, 714, 693 cm\(^{-1}\); HRMS exact mass calcd for \((C_{21}H_{14}ClN_2O_2)^+\) requires \(m/z\) 361.0738, found \(m/z\) 361.0729.

![Image of 6-(4-Chloro-phenyl)-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (3o)](image)

6-(4-Bromo-phenyl)-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (3p): white solid; 48% yield; \(^1\)H NMR (500 MHz, DMSO) \(\delta\) (ppm) 7.20 (s, 1H), 7.35-7.40 (m, 4H), 7.52-7.63 (m, 4H), 7.71-7.72 (m, 1H), 7.86 (m, 1H), 8.32 (d, \(J = 7.0\) Hz, 1H), 8.51 (d, \(J = 7.4\) Hz, 1H); \(^1\)C NMR (126 MHz, DMSO) \(\delta\) (ppm) 78.62, 116.40, 121.06, 121.50, 124.96, 126.41, 127.09, 127.65, 128.05, 130.07, 131.08, 134.77, 135.33, 138.63, 141.15, 147.00, 158.93, 161.19; IR (KBr) \(\gamma\) 3297, 1655, 1463, 1314, 1074, 831, 759, 694 cm\(^{-1}\); HRMS exact mass calcd for \((C_{21}H_{14}BrN_2O_2)^+\) requires \(m/z\) 405.0233, found \(m/z\) 405.0234.

![Image of 6-(4-Bromo-phenyl)-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (3p)](image)

6-Hydroxy-6-(4-iodo-phenyl)-6H-indolo[2,1-b]quinazolin-12-one (3q): white solid; 48% yield; \(^1\)H NMR (500 MHz, DMSO) \(\delta\) (ppm) 7.16 (s, 1H), 7.22-7.23 (m, 2H), 7.34-7.39 (m, 2H), 7.55-7.58 (m, 1H), 7.61-7.64 (m, 1H), 7.69-7.72 (m, 1H), 7.84-7.88 (m, 1H), 8.31 (d, \(J = 7.9\) Hz, 1H), 8.51 (d, \(J = 8.0\) Hz, 1H); \(^1\)C NMR (126 MHz, DMSO) \(\delta\) (ppm) 78.71, 94.06, 116.39, 121.48, 124.95, 126.41, 127.08, 127.62, 127.65, 128.09,
130.05, 134.76, 135.35, 136.94, 138.63, 141.60, 147.01, 158.92, 161.21; IR (KBr) \( \gamma \) 3338, 1657, 1463, 1314, 832, 758, 693 cm\(^{-1}\); HRMS exact mass calcd for \((C_{21}H_{14}N_2O_2)^+\) requires \(m/z\) 453.0095, found \(m/z\) 453.0096.

6-Hydroxy-6-(4-trifluoromethyl-phenyl)-6H-indolo[2,1-b]quinazolin-12-one \( (3r) \):
white solid; 30\% yield; \(^1\)H NMR (500 MHz, DMSO) \( \delta \) (ppm) 7.33-7.38 (m, 3H), 7.57-7.71 (m, 7H), 7.85-7.88 (m, 1H), 8.33 (d, \( J = 7.5 \) Hz, 1H), 8.54 (d, \( J = 7.8 \) Hz, 1H); \(^{13}\)C NMR (126 MHz, DMSO) \( \delta \) (ppm) 78.75, 116.46, 121.55, 123.06, 124.99, 125.12, 125.15, 126.42, 126.67, 127.13, 127.68, 128.24, 128.49, 130.20, 134.77, 135.16, 138.73, 146.26, 146.98, 158.93, 161.06; IR (KBr) \( \gamma \) 3296, 1656, 1468, 1325, 1121, 844, 758 cm\(^{-1}\); HRMS exact mass calcd for \((C_{22}H_{14}F_3N_2O_2)^+\) requires \(m/z\) 395.1002, found \(m/z\) 395.1010.

6-Hydroxy-6-(4-methoxy-phenyl)-6H-indolo[2,1-b]quinazolin-12-one \( (3s) \):
white solid; 50\% yield; \(^1\)H NMR (500 MHz, DMSO) \( \delta \) (ppm) 3.72 (s, 3H), 6.87-6.89 (m, 2H), 6.99-7.00 (m, 1H), 7.31-7.33 (m, 2H), 7.36-7.39 (m, 2H), 7.52-7.57 (m, 1H), 7.60-7.63 (m, 1H), 7.71-7.73 (m, 1H), 7.84-7.87 (m, 1H), 8.30-8.32 (m, 1H), 8.50-8.52 (m, 1H); \(^{13}\)C NMR (126 MHz, DMSO) \( \delta \) (ppm) 55.08, 78.67, 113.56, 116.30, 121.39, 125.00, 126.39, 126.97, 127.47, 127.62, 129.76, 133.70, 134.72, 136.02, 138.49, 147.11, 158.83, 158.97, 161.79; IR (KBr) \( \gamma \) 3353, 1655, 1465, 1366, 1307, 1255, 1175, 839, 755, 695 cm\(^{-1}\); HRMS exact mass calcd for \((C_{22}H_{17}N_2O_3)^+\) requires \(m/z\) 357.1234, found \(m/z\) 357.1236.
6-(4-tert-Butyl-phenyl)-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (3t): white solid; 82% yield; \(^1\)H NMR (500 MHz, DMSO) \(\delta\) (ppm) 1.24 (s, 9H), 7.01 (s, 1H), 7.31-7.37 (m, 6H), 7.55 (m, 1H), 7.60-7.63 (m, 1H), 7.71-7.73 (m, 1H), 7.84-7.87 (m, 1H), 8.32 (d, J = 7.6 Hz, 1H), 8.52 (d, J = 7.8 Hz, 1H); \(^{13}\)C NMR (126 MHz, DMSO) \(\delta\) (ppm) 31.04, 34.19, 78.84, 116.29, 121.39, 124.95, 125.06, 125.35, 126.40, 126.99, 127.52, 127.64, 129.78, 134.74, 135.98, 138.48, 138.82, 147.07, 150.05, 158.95, 161.74; IR (KBr) \(\gamma\) 3392, 1658, 1463, 1361, 1181, 819, 756, 695 cm\(^{-1}\); HRMS exact mass calcd for (C\(_{25}\)H\(_{23}\)N\(_2\)O\(_2\))^+ requires \(m/z\) 383.1754, found \(m/z\) 383.1758.

6-Hydroxy-6-o-tolyl-6H-indolo[2,1-b]quinazolin-12-one (3u): white solid; 86% yield; \(^1\)H NMR (500 MHz, DMSO) \(\delta\) (ppm) 1.44 (s, 3H), 7.03-7.05 (m, 1H), 7.10 (m, 1H), 7.13-7.15 (m, 1H), 7.25-7.28 (m, 1H), 7.33-7.41 (m, 2H), 7.56-7.59 (m, 1H), 7.63-7.66 (m, 1H), 7.71-7.72 (m, 1H), 7.86-7.89 (m, 1H), 8.15-8.17 (m, 1H), 8.35 (d, J = 7.8 Hz, 1H), 8.52 (d, J = 8.0 Hz, 1H); \(^{13}\)C NMR (126 MHz, DMSO) \(\delta\) (ppm) 18.73, 78.16, 116.18, 121.27, 124.53, 125.74, 126.50, 126.55, 127.19, 127.79, 127.97, 130.04, 131.22, 134.09, 134.33, 134.94, 138.92, 139.07, 146.99, 158.95, 160.87; IR (KBr) \(\gamma\) 3342, 1659, 1464, 1312, 1174, 762, 694 cm\(^{-1}\); HRMS exact mass calcd for (C\(_{22}\)H\(_{17}\)N\(_2\)O\(_2\))^+ requires \(m/z\) 341.1285, found \(m/z\) 341.1299.
6-Hydroxy-6-naphthalen-2-yl-6H-indolo[2,1-b]quinazolin-12-one (3v): white solid; 81% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 7.25 (s, 1H), 7.38-7.40 (m, 3H), 7.51-7.70 (m, 5H), 7.82-7.94 (m, 4H), 8.10 (m, 1H), 8.34 (d, J = 7.4 Hz, 1H), 8.57 (d, J = 7.5 Hz, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 79.12, 116.42, 121.51, 123.94, 124.18, 125.07, 126.24, 126.31, 126.42, 127.07, 127.41, 127.58, 127.66, 127.87, 128.11, 129.97, 132.40, 132.51, 134.75, 135.86, 138.70, 139.13, 147.10, 159.02, 161.65; IR (KBr) γ 3327, 1654, 1465, 1317, 1183, 819, 780, 691 cm$^{-1}$; HRMS exact mass calcd for (C$_{25}$H$_{17}$N$_2$O$_2$)$^+$ requires m/z 377.1285, found m/z 377.1287.

6-Cyclohexyl-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (5a): white solid; 68% yield; $^1$H NMR (500 MHz, DMSO) δ (ppm) 0.73-0.77 (m, 1H), 0.93-0.95 (m, 1H), 1.08-1.21 (m, 3H), 1.33-1.35 (m, 1H), 1.55 (m, 2H), 1.70-1.72 (m, 1H), 1.92-1.94 (m, 1H), 2.17-2.22 (m, 1H), 6.30 (s, 1H), 7.40-7.43 (m, 1H), 7.52-7.55 (m, 1H), 7.60-7.64 (m, 2H), 7.82-7.90 (m, 2H), 8.30 (d, J = 7.4 Hz, 1H), 8.47 (d, J = 7.7 Hz, 1H); $^{13}$C NMR (126 MHz, DMSO) δ (ppm) 25.50, 25.61, 25.73, 25.94, 46.94, 79.82, 115.90, 121.19, 124.87, 126.36, 126.46, 127.33, 127.66, 129.43, 133.61, 134.77, 138.69, 146.96, 158.78, 161.69; IR (KBr) γ 3379, 2925, 2856, 1652, 1468, 1317, 774, 694 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{21}$N$_2$O$_2$)$^+$ requires m/z 333.1598, found m/z 333.1599.
6-Cyclopentyl-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (5b): white solid; 50% yield; $^1$H NMR (500 MHz, DMSO) $\delta$ (ppm) 1.36-1.51 (m, 7H), 1.63-1.66 (m, 1H), 2.72-2.76 (m, 1H), 6.32 (s, 1H), 7.39-7.42 (m, 1H), 7.52-7.55 (m, 1H), 7.61-7.67 (m, 2H), 7.80-7.82 (m, 1H), 7.88-7.91 (m, 1H), 8.30 (d, J = 7.8 Hz, 1H), 8.47 (d, J = 8.0 Hz, 1H); $^{13}$C NMR (126 MHz, DMSO) $\delta$ (ppm) 25.03, 25.13, 26.03, 26.46, 48.35, 79.98, 115.91, 121.21, 124.74, 126.34, 126.56, 127.35, 127.65, 129.50, 134.11, 134.78, 138.52, 146.95, 158.84, 161.48; IR (KBr) $\gamma$ 3387, 2914, 2858, 1656, 1468, 1317, 774, 695 cm$^{-1}$; HRMS exact mass calcd for (C$_{20}$H$_{19}$N$_2$O$_2$)$^+$ requires m/z 319.1441, found m/z 319.1446.

6-Hexyl-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (5c): white solid; 51% yield; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm) 0.75 (t, J = 7.0 Hz, 3H), 0.86-0.91 (m, 2H), 1.05-1.17 (m, 6H), 2.18-2.24 (m, 1H), 2.27-2.33 (m, 1H), 4.45 (s,1H), 7.06-7.13 (m,2H), 7.45-7.52 (m, 2H), 7.72-7.79 (m, 2H), 8.16 (d, J = 7.8 Hz, 1H), 8.21 (d, J = 7.2 Hz, 1H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ (ppm) 13.92, 22.40, 23.24, 29.10, 31.34, 39.85, 78.37, 116.53, 121.38, 123.69, 126.99, 127.06, 127.27, 127.68, 129.97, 132.57, 134.47, 138.43, 147.14, 159.81, 160.82; IR (KBr) $\gamma$ 3392, 2919, 2851, 1656, 1468, 1317, 774, 693 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{23}$N$_2$O$_2$)$^+$ requires m/z 335.1754, found m/z 335.1764.

6-Butyl-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (5d): white solid; 41% yield; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm) 0.71 (t, J = 7.3 Hz, 3H), 0.80-0.87 (m, 2H), 1.14-1.19 (m, 2H), 2.18-2.24 (m, 1H), 2.28-2.34 (m, 1H), 4.68 (s, 1H), 7.03-7.08 (m, 2H),
7.44-7.51 (m, 2H), 7.71-7.78 (m, 2H), 8.11-8.17 (m, 2H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ (ppm) 13.72, 22.56, 25.40, 39.57, 78.33, 116.43, 121.28, 123.67, 126.98, 127.07, 127.24, 127.66, 129.90, 132.63, 134.47, 138.32, 147.12, 159.83, 160.80; IR (KBr) γ 3367, 2926, 2855, 1661, 1464, 1319, 752, 698 cm$^{-1}$; HRMS exact mass calcd for (C$_{19}$H$_{19}$N$_2$O$_2$)$^+$ requires m/z 307.1441, found m/z 307.1441.

6-Cyclohexyl-8-fluoro-6-hydroxy-6H-indolo[2,1-b]quinazolin-12-one (5e): white solid; 40% yield; $^1$H NMR (500 MHz, DMSO) $\delta$ (ppm) 0.78-0.85 (m, 1H), 0.93-1.23 (m, 4H), 1.38-1.41 (m, 1H), 1.55-1.57 (m, 2H), 1.69-1.72 (m, 1H), 1.85-1.87 (m, 1H), 2.17-2.22 (m, 1H), 6.44 (s, 1H), 7.37-7.41 (m, 1H), 7.44-7.47 (m, 1H), 7.62-7.65 (m, 1H), 7.83-7.84 (m, 1H), 7.89-7.93 (m, 1H), 8.29-8.30 (m, 1H), 8.45-8.48 (m, 1H); $^{13}$C NMR (126 MHz, DMSO) $\delta$ (ppm) 25.47, 25.52, 25.65, 25.78, 25.86, 46.78, 79.75, 112.22, 112.42, 115.97, 116.15, 117.42, 117.49, 121.10, 126.34, 127.50, 127.71, 134.88, 136.22, 136.28, 146.83, 158.60, 159.41, 161.43; IR (KBr) γ 3376, 2929, 2858, 1659, 1467, 1316, 778, 663 cm$^{-1}$; HRMS exact mass calcd for (C$_{21}$H$_{20}$FN$_2$O$_2$)$^+$ requires m/z 351.1503, found m/z 351.1509.

6-Cyclohexyl-6-hydroxy-8-methyl-6H-indolo[2,1-b]quinazolin-12-one (5f): white solid; 60% yield; $^1$H NMR (500 MHz, DMSO) $\delta$ (ppm) 0.72-0.79 (m, 1H), 0.94-0.99 (m, 1H), 1.06-1.21 (m, 3H), 1.31-1.34 (m, 1H), 1.55 (m, 2H), 1.70-1.73 (m, 1H), 1.91-1.94 (m, 1H), 2.16-2.20 (m, 1H), 2.42 (s, 3H), 6.28 (s, 1H), 7.33-7.34 (m, 1H), 7.42 (m, 1H), 7.60-7.63 (m, 1H), 7.82-7.83 (m, 1H), 7.88-7.91 (m, 1H), 8.28-8.34 (m, 2H); $^{13}$C NMR (126 MHz, DMSO) $\delta$ (ppm) 20.96, 25.49, 25.58, 25.72, 25.93, 46.91, 79.83, 115.64, 151.1509.
121.17, 125.31, 126.29, 127.29, 127.64, 129.77, 133.67, 134.68, 135.94, 136.43, 146.95, 158.57, 161.86; IR (KBr) γ 3383, 2926, 2855, 1654, 1476, 1310, 766, 688 cm⁻¹; HRMS exact mass calcd for (C_{22}H_{23}N_{2}O_{2})⁺ requires m/z 347.1754, found m/z 347.1758.

6-Hexyl-6-hydroxy-8-methyl-6H-indolo[2,1-b]quinazolin-12-one (5g): white solid; 41% yield; 1H NMR (500 MHz, CDCl₃) δ (ppm) 0.75 (t, J = 6.9 Hz, 3H), 0.82-0.88 (m, 2H), 1.07-1.15 (m, 6H), 2.14-2.20(m, 1H), 2.22 (s, 3H), 2.27-2.35 (m, 1H), 4.49 (s, 1H), 6.77-6.79 (m, 1H), 7.42-7.49 (m, 1H), 7.72-7.75 (m, 1H), 7.78-7.80 (m, 1H), 7.99-8.01(m, 1H), 8.08-8.09 (m, 1H); ¹³C NMR (126 MHz, CDCl₃) δ (ppm) 13.91, 21.45, 22.42, 23.21, 29.13, 31.35, 39.68, 78.27, 116.11, 121.26, 124.26, 126.76, 127.02, 127.75, 130.43, 132.68, 134.17, 136.09, 137.06, 147.30, 159.76, 160.87; IR (KBr) γ 3385, 2932, 2860, 1662, 1474, 1315, 767, 667 cm⁻¹; HRMS exact mass calcd for (C_{22}H_{25}N_{2}O_{2})⁺ requires m/z 349.1911, found m/z 349.1902.

6-Cyclohexyl-6-hydroxy-8-methoxy-6H-indolo[2,1-b]quinazolin-12-one (5h): white solid; 37% yield; ¹H NMR (500 MHz, DMSO) δ (ppm) 0.79-0.87 (m, 1H), 0.95-1.23 (m, 4H), 1.37-1.39 (m, 1H), 1.55-1.57 (m, 2H), 1.69-1.71 (m, 1H), 1.84-1.87 (m, 1H), 2.16-2.20 (m, 1H), 3.84 (s, 3H), 6.33 (s, 1H), 7.08-7.11 (m, 2H), 7.60-7.63 (m, 1H), 7.81-7.83 (m, 1H), 7.87-7.90 (m, 1H), 8.28 (d, J = 7.8 Hz, 1H), 8.36 (d, J = 8.7 Hz, 1H); ¹³C NMR (126 MHz, DMSO) δ (ppm) 25.50, 25.55, 25.71, 25.83, 25.90, 46.94, 55.63, 79.88, 110.76, 114.06, 116.87, 121.17, 126.20, 127.27, 127.64, 131.99, 134.57, 135.45, 146.88, 157.91, 158.35, 161.71; IR (KBr) γ 3392, 2923, 2853, 1653, 1472, 1314, 1281,
768, 691 cm⁻¹; HRMS exact mass calcd for (C₂₂H₂₃N₂O₃)⁺ requires m/z 363.1703, found m/z 363.1693.


**X-ray analysis data of (3a)**

![Diagram of (3a)]
Datablock: mo_20150322c_0m

Bond precision:  C-C = 0.0051 A  Wavelength=0.71073

Cell:  
a=7.079(2)  b=9.403(3)  c=23.106(7)  
alpha=90  beta=91.430(5)  gamma=90

Temperature:  298 K

Calculated  Reported
Volume  1535.6(8)  1535.5(8)
Space group  P 21/c  P 21/c
Hall group  -P 2ybc  -P 2ybc
Moiety formula  C21 H14 N2 O2  C21 H14 N2 O2
Sum formula  C21 H14 N2 O2  C21 H14 N2 O2
Mr  326.34  326.34
Dx,g cm^-3  1.412  1.412
Z  4  4
Mu (mm^-1)  0.092  0.092
F000  680.0  680.0
F000’  680.29
h,k,lmax  8,11,26  8,11,26
Nref  3021  2947
 Tmin,Tmax  0.982,0.989  0.971,0.989
Tmin’  0.971

Correction method= # Reported T Limits: Tmin=0.971 Tmax=0.989
AbsCorr = MULTI-SCAN

Data completeness= 0.976  Theta(max)= 26.000
R(reflections)= 0.0820(2570)  wR2(reflections)= 0.1544(2947)
S = 1.124  Npar= 227

The following ALERTS were generated. Each ALERT has the format
<test-name ALERT-type ALERT-level>.
Click on the hyperlinks for more details of the test.
**X-ray** analysis data of (5a)

![Chemical structure](image)

**Datablock: mo_20151008b_0m**

<table>
<thead>
<tr>
<th>Bond precision: C-C</th>
<th>0.0019 Å</th>
<th>Wavelength</th>
<th>0.71073</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell:</td>
<td>a=11.6035(16)</td>
<td>b=7.1139(11)</td>
<td>c=19.725(3)</td>
</tr>
<tr>
<td></td>
<td>alpha=90°</td>
<td>beta=97.765(2)</td>
<td>gamma=90°</td>
</tr>
<tr>
<td>Temperature:</td>
<td>298 K</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Calculated</th>
<th>Reported</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume</td>
<td>1641.1(4)</td>
</tr>
<tr>
<td>Space group</td>
<td>P 21/c</td>
</tr>
<tr>
<td>Hall group</td>
<td>-P 2ybc</td>
</tr>
<tr>
<td>Moiety formula</td>
<td>C21 H20 N2 O2</td>
</tr>
<tr>
<td>Sum formula</td>
<td>C21 H20 N2 O2</td>
</tr>
<tr>
<td>Mr</td>
<td>332.39</td>
</tr>
<tr>
<td>Dx, g cm-3</td>
<td>1.345</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>Mu (mm-1)</td>
<td>0.087</td>
</tr>
<tr>
<td>F000</td>
<td>704.0</td>
</tr>
<tr>
<td>F000’</td>
<td>704.29</td>
</tr>
<tr>
<td>h,k,l max</td>
<td>14,8,24</td>
</tr>
<tr>
<td>Nref</td>
<td>3216</td>
</tr>
<tr>
<td>Tmin, Tmax</td>
<td>0.977, 0.983</td>
</tr>
<tr>
<td>Tmin’</td>
<td>0.970</td>
</tr>
</tbody>
</table>

Correction method= # Reported T Limits: Tmin=0.970 Tmax=0.983
AbsCorr = MULTI-SCAN

Data completeness= 0.997  θ(max) = 26.000

R(reflections) = 0.0387 (2711)  wR2(reflections) = 0.1223 (3206)

S = 1.081  Npar = 227

The following ALERTs were generated. Each ALERT has the format **test-name ALERT alert-type alert-level**.

Click on the hyperlinks for more details of the test.
**Alert level C**

An exptl absorpt correction type has been given without a literature citation. This should be contained in the _exptl_absorpt_process_details field.

Absorption correction given as Multi-scan

**Alert level G**

<table>
<thead>
<tr>
<th>ALERT level</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>ALERT level A = Most likely a serious problem - resolve or explain</td>
</tr>
<tr>
<td>0</td>
<td>ALERT level B = A potentially serious problem, consider carefully</td>
</tr>
<tr>
<td>1</td>
<td>ALERT level C = Check. Ensure it is not caused by an omission or oversight</td>
</tr>
<tr>
<td>5</td>
<td>ALERT level G = General information/check it is not something unexpected</td>
</tr>
</tbody>
</table>

2 | ALERT type 1 CIP construction/grammar error, inconsistent or missing data |
2 | ALERT Type 2 Indicator that the structure model may be wrong or deficient |
2 | ALERT Type 3 Indicator that the structure quality may be low |
2 | ALERT Type 4 Improvement, methodology, query or suggestion |
2 | ALERT Type 5 Informative message, check |
NMR data of products
3a's $^1$H NMR (500 MHz, DMSO)

3a's $^{13}$C NMR (126 MHz, DMSO)
$3b$'s $^1$H NMR (500 MHz, DMSO)

$3b$'s $^{13}$C NMR (126 MHz, DMSO)
3c's 1H NMR (500 MHz, DMSO)

3c's 13C NMR (126 MHz, DMSO)
3d's $^1$H NMR (500 MHz, DMSO)

3d's $^{13}$C NMR (126 MHz, DMSO)
3e's $^1$H NMR (500 MHz, DMSO)

3e's $^{13}$C NMR (126 MHz, DMSO)
$^{1}H$ NMR (500 MHz, DMSO)

$^{13}C$ NMR (126 MHz, DMSO)
3g’s $^1$H NMR (500 MHz, DMSO)

3g’s $^{13}$C NMR (126 MHz, DMSO)
$3h$'s $^1$H NMR (500 MHz, DMSO)

$3h$'s $^{13}$C NMR (126 MHz, DMSO)
3i's $^1$H NMR (500 MHz, DMSO)

3i's $^{13}$C NMR (126 MHz, DMSO)
3j's $^1$H NMR (500 MHz, DMSO)

![$^1$H NMR spectrum](image)

3j's $^{13}$C NMR (126 MHz, DMSO)

![$^{13}$C NMR spectrum](image)
3k’s $^1$H NMR (500 MHz, DMSO)

3k’s $^{13}$C NMR (126 MHz, DMSO)
3I's $^1$H NMR (500 MHz, DMSO)

3I's $^{13}$C NMR (126 MHz, DMSO)
3m’s $^1$H NMR (500 MHz, DMSO)

3m’s $^{13}$C NMR (126 MHz, DMSO)
$3n's$ $^1H$ NMR (500 MHz, DMSO)

$3n's$ $^{13}C$ NMR (126 MHz, DMSO)
3o's $^1$H NMR (500 MHz, DMSO)

3o's $^{13}$C NMR (126 MHz, DMSO)
3p’s $^1$H NMR (500 MHz, DMSO)

3p’s $^{13}$C NMR (126 MHz, DMSO)
$3q$'s $^1$H NMR (500 MHz, DMSO)

$3q$'s $^{13}$C NMR (126 MHz, DMSO)
3r’s $^1$H NMR (500 MHz, DMSO)

3r’s $^{13}$C NMR (126 MHz, DMSO)
3s’s $^1$H NMR (500 MHz, DMSO)

3s’s $^{13}$C NMR (126 MHz, DMSO)
3t’s $^1$H NMR (500 MHz, DMSO)

3t’s $^{13}$C NMR (126 MHz, DMSO)
3u's \(^1\)H NMR (500 MHz, DMSO)

3u's \(^{13}\)C NMR (126 MHz, DMSO)
3v's $^1$H NMR (500 MHz, DMSO)

3v's $^{13}$C NMR (126 MHz, DMSO)
5a’s $^1$H NMR (500 MHz, DMSO)

5a’s $^{13}$C NMR (126 MHz, DMSO)
$5b$'s $^1$H NMR (500 MHz, DMSO)

$5b$'s $^{13}$C NMR (126 MHz, DMSO)
5c's $^1$H NMR (500 MHz, CDCl$_3$)

5c's $^1$C NMR (500 MHz, CDCl$_3$)
5d's $^1$H NMR (500 MHz, CDCl$_3$)

5d's $^1$C NMR (500 MHz, CDCl$_3$)
5e’s $^1$H NMR (500 MHz, DMSO)

5e’s $^1$C NMR (500 MHz, DMSO)
$5\sigma$'s $^1$H NMR (500 MHz, DMSO)

$5\sigma$'s $^1$C NMR (500 MHz, DMSO)
5g's $^1$H NMR (500 MHz, CDCl$_3$)

5g's $^1$C NMR (500 MHz, CDCl$_3$)
5h's $^1$H NMR (500 MHz, DMSO)

5h's $^1$C NMR (500 MHz, DMSO)