Supporting information

Mild and Efficient Synthesis of Indoles and Isoquinolones via a Nickel-Catalyzed Larock-Type Heteroannulation Reaction

Wei-Zhi Weng, Jian Xie, and Bo Zhang*

State Key Laboratory of Natural Medicines, China Pharmaceutical University, 24 Tongjia Xiang, Nanjing 210009, China

E-mail: zb3981444@cpu.edu.cn

Table of contents

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Physical data of the compounds</td>
<td>S2</td>
</tr>
<tr>
<td>Gram-scale reactions</td>
<td>S31</td>
</tr>
<tr>
<td>Mechanistic studies</td>
<td>S32</td>
</tr>
<tr>
<td>Nickel-catalyzed Larock-type heteroannulation reactions of ortho-bromo substrates with 1,2-diphenylethyne</td>
<td>S37</td>
</tr>
<tr>
<td>References</td>
<td>S38</td>
</tr>
<tr>
<td>NMR spectra</td>
<td>S39</td>
</tr>
</tbody>
</table>
Physical data of the compounds

1-(2,3-Diphenyl-1H-indol-1-yl)ethanone (3)[1]

According to GP1 with N-(2-iodophenyl)acetamide (53.0 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 3 as white solid (56.7 mg, 91%). Mp: 130-132 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.46 (d, J = 8.4 Hz, 1H), 7.56 (dd, J = 7.8, 0.6 Hz, 1H), 7.43-7.20 (m, 12H), 1.99 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.6, 136.7, 134.9, 133.0, 132.9, 130.7, 130.0, 129.2, 128.6, 128.6, 128.2, 126.9, 125.5, 123.4, 123.3, 119.5, 116.2, 27.9; HRMS (ESI) calculated for C₂₂H₁₈NO [M+H]+ m/z 312.1383, found 312.1382.

1-(5-Methyl-2,3-diphenyl-1H-indol-1-yl)ethanone (4)[2]

According to GP1 with N-(2-iodo-4-methylphenyl)acetamide (56.0 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.2 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 4 as white solid (52.6 mg, 81%). Mp: 170-172 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.34 (d, J = 8.4 Hz, 1H), 7.36-7.19 (m, 12H), 2.43 (s, 3H), 1.98 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.4, 135.1, 135.0, 133.4, 133.1, 133.0, 130.7, 130.0, 129.4, 128.5, 128.2, 126.83, 126.76, 123.2, 119.3, 115.9, 27.8, 21.4; HRMS (ESI) calculated for C₂₃H₂₀NO [M+H]+ m/z 326.1539, found 326.1539.
1-(5-Methoxy-2,3-diphenyl-1H-indol-1-yl)ethanone (5)[2]

According to GP1 with N-(2-iodo-4-methoxyphenyl)acetamide (58.7 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl\(_2\) (11.0 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 5 as white solid (58.5 mg, 86%). Mp: 176-178 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.41-8.38 (m, 1H), 7.35-7.18 (m, 10H), 7.02-6.99 (m, 2H), 3.79 (s, 3H), 1.96 (s, 3H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 171.1, 156.6, 135.6, 133.0, 132.9, 131.5, 130.7, 130.1, 129.9, 128.6, 128.5, 128.2, 126.8, 123.2, 117.3, 113.8, 102.0, 55.6, 27.7; HRMS (ESI) calculated for C\(_{23}\)H\(_{20}\)NO \([\text{M+H}]^+\) m/z 342.1489, found 342.1487.

1-(5-(tert-Butyl)-2,3-diphenyl-1H-indol-1-yl)ethanone (6)

According to GP1 with N-(4-(tert-butyl)-2-iodophenyl)acetamide (63.7 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl\(_2\) (16.3 mg, 0.03 mmol, 0.15 equiv), and Et\(_3\)N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 6 as white solid (69.8 mg, 95%). Mp: 147-149 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.40 (d, \(J = 8.7\) Hz, 1H), 7.54 (d, \(J = 1.8\) Hz, 1H), 7.49 (dd, \(J = 8.7, 2.1\) Hz, 1H), 7.35-7.21 (m, 10H), 1.98 (s, 3H), 1.36 (s, 9H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 171.3, 147.0, 135.2, 134.9, 133.2, 133.1, 130.8, 130.0, 129.0, 128.5, 128.2, 126.8, 123.6, 123.4, 115.8, 115.4, 34.7, 31.7, 27.8; HRMS (ESI) calculated for C\(_{26}\)H\(_{26}\)NO \([\text{M+H}]^+\) m/z 368.2009, found 368.2007.
1-(5-Fluoro-2,3-diphenyl-1H-indol-1-yl)ethanone (7)[1]

According to GP1 with N-(4-fluoro-2-iodophenyl)acetamide (55.9 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (53.9 mg, 0.3 mmol, 1.5 equiv), Ni(dppe)Cl₂ (11.2 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 µL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 7 as white solid (56.5 mg, 86%). Mp: 175-177 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.44 (dd, J = 9.1, 4.7 Hz, 1H), 7.39-7.31 (m, 5H), 7.29-7.24 (m, 3H), 7.22-7.16 (m, 3H), 7.11 (td, J = 9.1, 2.7 Hz, 1H), 1.98 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.3, 159.9 (d, J = 239.0 Hz), 136.4, 133.1, 132.6, 132.5, 130.3 (d, J = 9.3 Hz), 129.8, 128.9, 128.7, 128.3, 127.1, 123.0 (d, J = 3.8 Hz), 117.5 (d, J = 8.8 Hz), 113.0 (d, J = 24.8 Hz), 105.1 (d, J = 24.2 Hz), 27.8; HRMS (ESI) calculated for C₂₂H₁₇FNO [M+H]+ m/z 330.1289, found 330.1290.

1-(5-Chloro-2,3-diphenyl-1H-indol-1-yl)ethanone (8)[2]

According to GP1 with N-(4-chloro-2-iodophenyl)acetamide (59.3 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppe)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 µL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 8 as white solid (37.0 mg, 54%). Mp: 197-199 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.39 (d, J = 8.7 Hz, 1H), 7.51 (d, J = 2.1 Hz, 1H), 7.38-7.25 (m, 9H), 7.19-7.16 (m, 2H), 1.98 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.4, 136.1, 135.1, 132.4, 132.3, 130.7, 130.5, 129.9, 129.3, 128.9, 128.7, 128.4, 127.2, 125.5, 122.6, 119.1, 117.4, 27.8; HRMS (ESI) calculated for C₂₂H₁₇ClNO [M+H]+ m/z 346.0993, found 346.1000.
1-(2,3-Diphenyl-5-(trifluoromethyl)-1H-indol-1-yl)ethanone (9)

According to GPI with N-(2-iodo-4-(trifluoromethyl)phenyl)acetamide (65.9 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 9 as white solid (33.1 mg, 44%).

Mp: 153-155 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.55 (d, J = 8.7 Hz, 1H), 7.83-7.80 (m, 1H), 7.64 (dd, J = 8.8, 1.5 Hz, 1H), 7.40-7.28 (m, 8H), 7.22-7.19 (m, 2H), 2.01 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.6, 138.2 (q, J = 1.1 Hz), 136.6, 132.2, 132.1, 130.7, 129.9, 129.1, 129.0, 128.8, 128.5, 127.3, 126.0 (q, J = 13.9 Hz), 124.7 (q, J = 270.3 Hz), 123.1, 122.1 (q, J = 3.7 Hz), 117.0 (q, J = 4.0 Hz), 116.5, 27.9; HRMS (ESI) calculated for C₂₃H₁₇F₃NO [M+H]⁺ m/z 380.1257, found 380.1255.

1-(2,3,5-Triphenyl-1H-indol-1-yl)ethanone (10)

According to GPI with N-(3-iodo-[1,1'-biphenyl]-4-yl)acetamide (68.0 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 10 as white solid (57.0 mg, 74%).

Mp: 152-154 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.39 (d, d, J = 8.7, 0.3 Hz, 1H), 7.76-7.20 (m, 1H), 7.67-7.60 (m, 3H), 7.44-7.23 (m, 13H), 2.01 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.5, 141.5, 137.2, 136.2, 135.6, 132.9, 132.8, 130.7, 130.0, 129.7, 128.7, 128.6, 128.3, 127.4, 127.0, 126.9, 124.9, 123.5, 117.9, 116.5, 27.9; HRMS (ESI) calculated for C₂₈H₂₂NO [M+H]⁺ m/z 388.1696, found 388.1698.
1-(6-Methyl-2,3-diphenyl-1H-indol-1-yl)ethanone (11)[2]

\[\text{Ph} \quad \text{Ph} \quad \text{N} \quad \text{O} \quad \text{Me} \quad \text{Me} \]

According to GP1 with N-(2-iodo-5-methylphenyl)acetamide (55.6 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (53.7 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (15.9 mg, 0.03 mmol, 0.15 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 11 as white solid (60.8 mg, 93%). Mp: 118-120 °C; \(^1\text{H NMR}\) (300 MHz, CDCl₃) δ 8.30 (s, 1H), 7.43 (d, \(J = 8.1\) Hz, 1H), 7.36-7.19 (m, 10H), 7.13 (dd, \(J = 8.0, 0.8\) Hz, 1H), 2.52 (s, 3H), 1.98 (s, 3H); \(^{13}\text{C NMR}\) (75 MHz, CDCl₃) δ 171.7, 137.1, 135.6, 134.3, 133.2, 133.0, 130.7, 130.0, 128.54, 128.46, 128.2, 127.0, 126.8, 125.1, 123.2, 119.1, 116.3, 28.0, 22.0; HRMS (ESI) calculated for C\(_{23}\)H\(_{20}\)NO \([\text{M}+\text{H}]^+\) m/z 326.1539, found 326.1538.

1-(6-Fluoro-2,3-diphenyl-1H-indol-1-yl)ethanone (12)

\[\text{F} \quad \text{Ph} \quad \text{Ph} \quad \text{N} \quad \text{O} \quad \text{Me} \]

According to GP1 with N-(5-fluoro-2-iodophenyl)acetamide (56.1 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.2 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 12 as white solid (25.3 mg, 38%). Mp: 145-147 °C; \(^1\text{H NMR}\) (300 MHz, CDCl₃) δ 8.23 (dd, \(J = 10.7, 2.4\) Hz, 1H), 7.46 (dd, \(J = 8.7, 5.5\) Hz, 1H), 7.38-7.31 (m, 5H), 7.29-7.24 (m, 3H), 7.22-7.18 (m, 2H), 7.05 (td, \(J = 8.9, 2.4\) Hz, 1H), 1.98 (s, 3H); \(^{13}\text{C NMR}\) (75 MHz, CDCl₃) δ 171.5, 161.2 (d, \(J = 239.6\) Hz), 136.9 (d, \(J = 12.7\) Hz), 135.1 (d, \(J = 3.8\) Hz), 132.7 (d, \(J = 2.3\) Hz), 130.7, 129.9, 128.8, 128.7, 128.3, 127.1, 125.6 (d, \(J = 1.7\) Hz), 123.0 (d, \(J = 1.1\) Hz), 120.2 (d, \(J = 9.9\) Hz), 111.9 (d, \(J = 24.2\) Hz), 103.7 (d, \(J = 28.6\) Hz), 27.8; HRMS (ESI) calculated for C\(_{22}\)H\(_{16}\)FNOna \([\text{M}+\text{Na}]^+\) m/z 352.1108, found
Methyl 1-acetyl-2,3-diphenyl-1H-indole-6-carboxylate (13)

According to GP1 with methyl 3-acetamido-4-iodobenzoate (63.9 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl$_2$ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 30/1) to afford the desired product 13 as white solid (64.2 mg, 87%).

$\text{MeO}_2\text{C}$

$\text{Ph}$

$\text{Me}$

$\text{N}$

$\text{Ph}$

$\text{Mp: 163-165 °C; }^1\text{H NMR (300 MHz, CDCl}_3\text{)} \delta 9.11 (d, \text{J} = 0.9 \text{ Hz}, 1\text{H}), 8.00 (dd, \text{J} = 8.3, 1.4 \text{ Hz}, 1\text{H}), 7.58 (d, \text{J} = 8.4 \text{ Hz}, 1\text{H}), 7.40-7.32 (m, 5\text{H}), 7.22-7.18 (m, 2\text{H}), 3.96 (s, 3\text{H}); ^13\text{C NMR (75 MHz, CDCl}_3\text{)} \delta 171.3, 167.6, 137.8, 136.1, 132.7, 132.4, 132.3, 130.6, 129.9, 129.0, 128.7, 128.3, 127.1, 127.0, 125.0, 122.9, 119.2, 117.8, 52.1, 27.9; \text{HRMS (ESI) calculated for C}_{24}\text{H}_{20}\text{NO}_3 [\text{M+H}]^+ m/z 370.1438, found 370.1438.}$

1-(6,7-Diphenyl-5H-[1,3]dioxolo[4,5-f]indol-5-yl)ethanone (14)[3]

$\text{Ph}$

$\text{Me}$

$\text{N}$

$\text{Ph}$

According to GP1 with N-(6-iodobenzo[d][1,3]dioxol-5-yl)acetamide (61.5 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl$_2$ (11.2 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 14 as white solid (37.9 mg, 53%).

$\text{Mp: 194-196 °C; }^1\text{H NMR (300 MHz, CDCl}_3\text{)} \delta 8.04 (s, 1\text{H}), 7.35-7.29 (m, 5\text{H}), 7.28-7.23 (m, 3\text{H}), 7.18-7.15 (m, 2\text{H}), 6.91 (s, 1\text{H}), 5.99 (s, 2\text{H}), 1.96 (s, 3\text{H}); ^13\text{C NMR (75 MHz, CDCl}_3\text{)} \delta 171.6, 146.8, 145.1, 133.74, 133.72, 133.10, 133.08, 131.6, 130.8, 129.9, 128.5, 128.4, 128.2, 126.9, 123.5, 101.2, 98.4, 98.0, 27.9; \text{HRMS (ESI)}}$
calculated for C_{23}H_{18}NO_{3} [M+H]^+ m/z 356.1281, found 356.1281.

1-(4-Methyl-2,3-diphenyl-1H-indol-1-yl)ethanone (15)

![Chemical structure of 15](attachment:structure_15.png)

According to GP1 with N-(2-iodo-3-methylphenyl)acetamide (55.1 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.2 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl\(_2\) (11.1 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 15 as white solid (24.8 mg, 38%).

MP: 171-173 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.35 (d, \(J = 8.4\) Hz, 1H), 7.29-6.99 (m, 11H), 7.00 (d, \(J = 7.2\) Hz, 1H), 2.01 (s, 3H), 1.97 (s, 3H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 171.5, 136.5, 135.6, 135.3, 133.0, 131.10, 131.07, 130.4, 128.2, 128.1, 127.5, 127.0, 125.6, 125.2, 124.7, 113.7, 27.9, 20.1; HRMS (ESI) calculated for C_{23}H_{20}NO [M+H]^+ m/z 326.1539, found 326.1540.

1-(2,3-Diphenyl-1H-benzo[f]indol-1-yl)ethanone (16)

![Chemical structure of 16](attachment:structure_16.png)

According to GP1 with N-(3-iodonaphthalen-2-yl)acetamide (62.9 mg, 0.2 mmol, 1.0 equiv), 1,2-diphenylethyne (54.2 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl\(_2\) (11.2 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 16 as white solid (14.3 mg, 20%).

MP: 226-228 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.56 (d, \(J = 9.0\) Hz, 1H), 7.90 (d, \(J = 8.4\) Hz, 1H), 7.80 (d, \(J = 9.3\) Hz, 1H), 7.56 (d, \(J = 8.7\) Hz, 1H), 7.40-7.31 (m, 6H), 7.28 (s, 5H), 7.24-7.18 (m, 1H), 2.06 (s, 3H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 172.2, 135.4, 134.7, 133.5, 132.9, 131.2, 131.0, 130.6, 128.5, 128.34, 128.30, 128.2, 127.6, 127.4, 126.1, 125.7, 125.0, 124.3, 123.7, 122.9, 115.7, 28.2; HRMS (ESI) calculated
for $C_{26}H_{20}NO [M+H]^+ m/z$ 362.1539, found 362.1540.

1-(2,3-Di-p-tolyl-1H-indol-1-yl)ethanone (17)$^{[2]}$

According to GP1 with $N$-(2-iodophenyl)acetamide (52.2 mg, 0.2 mmol, 1.0 equiv), 1,2-di-p-tolylethyn (62.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl$_2$ (11.2 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 17 as white solid (62.8 mg, 93%). Mp: 156-158 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.45 (d, $J = 8.1$ Hz, 1H), 7.54 (d, $J = 7.5$ Hz, 1H), 7.40-7.34 (m, 1H), 7.29-7.07 (m, 9H), 2.35 (s, 3H), 2.31 (s, 3H), 1.98 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 171.7, 138.4, 136.7, 136.4, 134.9, 130.6, 130.0, 129.9, 129.8, 129.4, 129.3, 128.9, 125.2, 123.6, 122.9, 119.5, 116.1, 27.9, 21.3, 21.2; HRMS (ESI) calculated for $C_{26}H_{20}NO [M+H]^+ m/z$ 340.1696, found 340.1692.

1-(2,3-Bis(4-methoxyphenyl)-1H-indol-1-yl)ethanone (18)$^{[2]}$

According to GP1 with $N$-(2-iodophenyl)acetamide (52.3 mg, 0.2 mmol, 1.0 equiv), 1,2-bis(4-methoxyphenyl)ethyn (71.6 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl$_2$ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 18 as white solid (65.8 mg, 89%). Mp: 139-141 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.45 (d, $J = 8.1$ Hz, 1H), 7.54 (d, $J =$
7.8 Hz, 1H), 7.41-7.36 (m, 1H), 7.31-7.22 (m, 3H), 7.18-7.13 (m, 2H), 6.91-6.82 (m, 4H), 3.83 (s, 3H), 3.79 (s, 3H), 2.01 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 171.7, 159.7, 158.4, 136.6, 134.6, 132.0, 131.1, 129.4, 125.4, 125.2, 125.1, 123.6, 122.6, 119.4, 116.2, 114.1, 113.7, 55.2, 55.1, 27.9; HRMS (ESI) calculated for C$_{24}$H$_{22}$NO$_3$ [M+H]$^+$ m/z 372.1594, found 372.1594.

1-(2,3-Bis(4-fluorophenyl)-1H-indol-1-yl)ethanone (19)

![Chemical Structure](image)

According to GP1 with N-(2-iodophenyl)acetamide (53.2 mg, 0.2 mmol, 1.0 equiv), 1,2-bis(4-fluorophenyl)ethyne (65.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl$_2$ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 19 as white solid (56.0 mg, 81%). Mp: 154-156 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.44 (d, $J = 8.1$ Hz, 1H), 7.50 (d, $J = 7.8$ Hz, 1H), 7.44-7.38 (m, 1H), 7.33-7.23 (m, 3H), 7.18-7.14 (m, 2H), 7.10-6.96 (m, 4H), 2.03 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 171.1, 162.7 (d, $J = 248.4$ Hz), 161.8 (d, $J = 245.0$ Hz), 136.6, 133.9, 132.4 (d, $J = 8.3$ Hz), 131.5 (d, $J = 8.3$ Hz), 129.0, 128.8 (d, $J = 5.0$ Hz), 128.7 (d, $J = 4.4$ Hz), 125.7, 123.9, 122.7, 119.3, 116.2, 115.9 (d, $J = 21.5$ Hz), 115.4 (d, $J = 21.5$ Hz), 28.0; HRMS (ESI) calculated for C$_{22}$H$_{16}$F$_2$NO [M+H]$^+$ m/z 348.1195, found 348.1191.

1-(2,3-Bis(4-chlorophenyl)-1H-indol-1-yl)ethanone (20)[2]

![Chemical Structure](image)
According to GP1 with N-(2-iodophenyl)acetamide (52.3 mg, 0.2 mmol, 1.0 equiv), 1,2-bis(4-chlorophenyl)ethyne (74.5 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 20 as white solid (36.3 mg, 48%).

Mp: 192-194 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.41 (d, J = 8.1 Hz, 1H), 7.50 (d, J = 7.8 Hz, 1H), 7.45-7.39 (m, 1H), 7.38-7.23 (m, 7H), 7.15-7.10 (m, 2H), 2.06 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.0, 136.7, 135.0, 133.8, 133.1, 131.8, 131.2, 131.1, 129.1, 128.8, 128.7, 125.9, 124.0, 122.6, 119.4, 116.1, 28.1; HRMS (ESI) calculated for C₂₂H₁₆Cl₂NO [M+H]+ m/z 380.0603, found 380.0601.

¹-(2,3-Di-m-tolyl-1H-indol-1-yl)ethanone (21)[²]

According to GP1 with N-(2-iodophenyl)acetamide (52.6 mg, 0.2 mmol, 1.0 equiv), 1,2-di-m-tolylethyne (62.1 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.2 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 21 as pale yellow oil (51.5 mg, 76%). ¹H NMR (300 MHz, CDCl₃) δ 8.45 (d, J = 8.1 Hz, 1H), 7.55 (d, J = 7.8 Hz, 1H), 7.40-7.35 (m, 1H), 7.30-6.98 (m, 9H), 2.31 (s, 3H), 2.27 (s, 3H), 1.99 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.7, 138.2, 137.6, 136.7, 135.0, 132.9, 132.8, 131.3, 130.7, 129.3, 128.4, 128.0, 127.9, 127.6, 127.1, 125.3, 123.6, 123.2, 119.6, 116.1, 27.9, 21.4, 21.3; HRMS (ESI) calculated for C₂₄H₂₂NO [M+H]+ m/z 340.1696, found 340.1692.
1-(2,3-Di(thiophen-3-yl)-1H-indol-1-yl)ethanone (22)

According to GP1 with N-(2-iodophenyl)acetamide (53.2 mg, 0.2 mmol, 1.0 equiv), 1,2-di(thiophen-3-yl)ethyne (57.3 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.2 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 22 as yellow solid (54.2 mg, 84%).

Mp: 123-125 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.46 (d, J = 8.4 Hz, 1H), 7.67 (d, J = 7.2 Hz, 1H), 7.43-7.24 (m, 5H), 7.17 (dd, J = 3.0, 1.2 Hz, 1H), 7.17 (dd, J = 4.8, 1.2 Hz, 1H), 7.17 (dd, J = 4.8, 1.2 Hz, 1H), 7.17 (dd, J = 4.8, 1.2 Hz, 1H), 2.08 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.2, 136.7, 133.0, 132.8, 129.8, 129.5, 128.7, 128.3, 126.53, 126.48, 125.6, 125.0, 123.8, 123.4, 119.5, 119.2, 116.4, 27.0; HRMS (ESI) calculated for C₁₈H₁₄NO₂S [M+H]⁺ m/z 324.0511, found 324.0513.

1-(2,3-Diethyl-1H-indol-1-yl)ethanone (23)

According to GP1 with N-(2-iodophenyl)acetamide (53.0 mg, 0.2 mmol, 1.0 equiv), hex-3-yne (25.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 23 as yellow oil (35.8 mg, 83%). ¹H NMR (300 MHz, CDCl₃) δ 7.77-7.34 (m, 1H), 7.51-7.48 (m, 1H), 7.27-7.21 (m, 2H), 3.03 (q, J = 7.3 Hz, 2H), 2.77 (s, 3H), 2.68 (q, J = 7.6 Hz, 2H), 1.25-1.19 (m, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 170.0, 139.3, 135.7, 130.6, 123.4, 122.6, 121.2, 118.6, 114.6, 27.7, 20.3, 17.0, 15.0; HRMS (ESI) calculated for C₁₄H₁₈NO [M+H]⁺ m/z 216.1383, found
1-(2,3-Dibutyl-1H-indol-1-yl)ethanone (24)\[^{[2]}\]

According to GP1 with N-(2-iodophenyl)acetamide (52.8 mg, 0.2 mmol, 1.0 equiv), dec-5-yne (42.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl\(_2\) (11.0 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 \(\mu\)L, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 24 as yellow oil (40.3 mg, 74%). \(^{1}\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.75-7.72 (m, 1H), 7.49-7.45 (m, 1H), 7.25-7.19 (m, 2H), 3.01-2.96 (m, 2H), 2.74 (s, 3H), 2.67-2.61 (m, 2H), 1.63-1.51 (m, 4H), 1.47-1.34 (m, 4H), 0.95 (q, \(J\) = 7.2 Hz, 6H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 170.0, 138.2, 135.6, 130.9, 123.3, 122.5, 120.0, 118.6, 114.4, 32.43, 32.38, 27.6, 26.7, 23.6, 22.83, 22.76, 14.0, 13.9; HRMS (ESI) calculated for C\(_{18}\)H\(_{26}\)NO \([M+H]^+\) m/z 272.2009, found 272.2002.

1-(2,3-Bis(methoxymethyl)-1H-indol-1-yl)ethanone (25)

According to GP1 with N-(2-iodophenyl)acetamide (52.6 mg, 0.2 mmol, 1.0 equiv), 1,4-dimethoxybut-2-yne (35.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl\(_2\) (11.1 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 \(\mu\)L, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10/1) to afford the desired product 25 as yellow oil (36.9 mg, 75%). \(^{1}\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.11 (d, \(J\) = 8.4 Hz, 1H), 7.66 (d, \(J\) = 7.2 Hz, 1H), 7.36-7.24 (m, 2H), 4.79 (s, 2H), 4.66 (s, 2H), 3.39 (s, 3H), 3.36 (s, 3H), 2.79 (s, 3H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 175.0, 136.4, 133.7, 128.8, 125.4, 123.2, 120.1, 119.4, 115.7, 64.5, 64.3, 57.9, 57.7, 26.2; HRMS (ESI) calculated for C\(_{14}\)H\(_{18}\)NO\(_3\) \([M+H]^+\) m/z 248.1281, found 248.1284.
According to GP1 with N-(2-iodophenyl)acetamide (53.1 mg, 0.2 mmol, 1.0 equiv), prop-1-yn-1-ylbenzene (35.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl$_2$ (10.9 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 26 as yellow solid (11.3 mg, 23%). Mp: 74-76 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.42 (d, $J = 8.1$ Hz, 1H), 7.54-7.43 (m, 4H), 7.40-7.29 (m, 4H), 2.14 (s, 3H), 1.96 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 171.1, 136.8, 134.9, 133.6, 130.2, 128.7, 128.4, 125.3, 123.4, 118.6, 118.1, 116.3, 27.6, 9.2; HRMS (ESI) calculated for C$_{17}$H$_{16}$NO [M+H]$^+$ m/z 250.1226, found 250.1225.

1-(3-Hexyl-2-methyl-1H-indol-1-yl)ethanone (27) and 1-(3-hexyl-2-methyl-1H-indol-1-yl)ethanone (27’)[5]

According to GP1 with N-(2-iodophenyl)acetamide (52.7 mg, 0.2 mmol, 1.0 equiv), non-2-yn (38.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl$_2$ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 27+27’ as yellow oil (45.6 mg, 89%, 27/27’ = 1.3:1). $^1$H NMR (300 MHz, CDCl$_3$) δ 27 7.97-7.91 (m, 1H), 7.45-7.42 (m, 1H), 7.26-7.19 (m, 2H), 2.69 (s, 3H), 2.63 (t, $J = 7.5$ Hz, 2H), 2.55 (s, 3H), 1.60-1.53 (m, 2H), 1.35-1.25 (m, 6H), 0.90-0.86 (m, 3H); 27’ 7.77-7.71 (m, 1H), 7.45-7.42 (m, 1H), 7.26-7.19 (m, 2H), 2.98 (t, $J = 7.7$ Hz, 2H), 2.73 (s, 3H), 2.18 (s, 3H), 1.60-1.53 (m, 2H), 1.35-1.25
According to GP1 with N-(2-iodophenyl)acetamide (52.5 mg, 0.2 mmol, 1.0 equiv), ethynylbenzene (31.0 mg, 0.3 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 28 as yellow oil (10.6 mg, 23%). ¹H NMR (300 MHz, CDCl₃) δ 8.36 (d, J = 8.1 Hz, 1H), 7.56 (d, J = 7.5 Hz, 1H), 7.48-7.42 (m, 5H), 7.39-7.26 (m, 2H), 6.63 (s, 1H), 2.08 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 171.4, 139.7, 137.7, 134.2, 129.0, 128.7, 128.6, 125.1, 123.7, 120.4, 116.0, 111.5, 27.9; HRMS (ESI) calculated for C₁₇H₂₄NO [M+H]⁺ m/z 258.1852, found 258.1852.

1-(2-Phenyl-1H-indol-1-yl)ethanone (28)[6]

According to GP2 with 2-iodo-N-(p-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethynyl (43.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 30 as white solid (73.7 mg, 95%). Mp: 216-218 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.57 (d, J = 7.5 Hz, 1H), 7.54-7.12 (m, 6H), 6.99 (s, 4H), 6.89 (s, 5H), 2.21 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 162.7, 141.2, 137.6, 137.2, 136.8, 136.4, 134.9, 132.4, 131.6, 131.0, 129.2, 129.1, 128.2,
127.9, 127.1, 127.0, 126.8, 126.7, 125.5, 118.6, 21.0; HRMS (ESI) calculated for C_{28}H_{22}NO [M+H]^+ m/z 388.1696, found 388.1704.

7-Methyl-3,4-diphenyl-2-(p-tolyl)isoquinolin-1(2H)-one (31)

According to GP2 with 2-iodo-5-methyl-N-(p-tolyl)benzamide (70.6 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl\textsubscript{2} (10.9 mg, 0.02 mmol, 0.1 equiv), and Et\textsubscript{3}N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 31 as white solid (77.9 mg, 97%).

Mp: 204-206 °C; \textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}) δ 8.36 (s, 1H), 7.39 (d, J = 8.7 Hz, 1H), 7.22-7.10 (m, 6H), 7.01-6.95 (m, 4H), 6.89 (s, 5H), 2.50 (s, 3H), 2.22 (s, 3H); \textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3}) δ 162.7, 143.3, 137.1, 136.9, 136.8, 136.6, 135.3, 135.0, 133.8, 131.6, 129.2, 129.1, 127.8, 127.02, 126.98, 126.7, 125.5, 125.4, 118.6, 21.3, 21.0; HRMS (ESI) calculated for C_{29}H_{24}NO [M+H]^+ m/z 402.1852, found 402.1853.

7-Methoxy-3,4-diphenyl-2-(p-tolyl)isoquinolin-1(2H)-one (32)\textsuperscript{7}

According to GP2 with 2-iodo-5-methoxy-N-(p-tolyl)benzamide (73.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl\textsubscript{2} (11.2 mg, 0.02 mmol, 0.1 equiv), and Et\textsubscript{3}N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 8/1) to afford the desired product 32 as white solid (82.0 mg, 98%).

Mp: 206-208 °C; \textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}) δ 7.98 (s, 1H), 7.24-7.10 (m, 7H), 6.99-6.96 (m, 4H), 6.88 (s, 5H), 3.92 (s, 3H), 2.22 (s, 3H); \textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3}) δ 162.4, 158.8, 138.9, 137.1, 137.0, 136.6, 134.9, 131.6, 131.5, 131.2, 129.2,
129.1, 127.8, 127.3, 127.0, 126.7, 22.7, 118.6, 108.2, 55.6, 21.0; **HRMS** (ESI) calculated for C\textsubscript{29}H\textsubscript{24}NO\textsubscript{2} [M+H]\textsuperscript{+} m/z 418.1802, found 418.1804.

**7-Fluoro-3,4-diphenyl-2-(p-tolyl)isoquinolin-1(2H)-one (33)**

![Chemical structure of 7-Fluoro-3,4-diphenyl-2-(p-tolyl)isoquinolin-1(2H)-one (33)](chart)

According to GP\textsubscript{2} with 5-fluoro-2-iodo-N-(p-tolyl)benzamide (71.3 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.8 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl\textsubscript{2} (11.4 mg, 0.02 mmol, 0.1 equiv), and Et\textsubscript{3}N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 33 as white solid (78.2 mg, 96%).

Mp: 187-189 °C; \textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}) δ 8.19 (d, J = 9.0 Hz, 1H), 7.31-7.10 (m, 7H), 7.01-6.98 (m, 4H), 6.89 (s, 5H), 2.21 (s, 3H); \textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3}) δ 161.9 (d, J = 3.8 Hz), 161.5 (d, J = 246.8 Hz), 140.5 (d, J = 3.0 Hz), 137.4, 136.5, 136.2, 134.5, 134.2 (d, J = 2.3 Hz), 131.2 (d, J = 36.8 Hz), 129.1 (d, J = 27.8 Hz), 128.2, 128.1, 128.0, 127.2, 127.13, 127.07, 127.03, 126.9, 121.0 (d, J = 23.3 Hz), 118.2, 113.2 (d, J = 22.5 Hz), 21.1; **HRMS** (ESI) calculated for C\textsubscript{28}H\textsubscript{21}FNO [M+H]\textsuperscript{+} m/z 406.1602, found 406.1601.

**Methyl 1-oxo-3,4-diphenyl-2-(p-tolyl)-1,2-dihydroisoquinoline-7-carboxylate (34)**

![Chemical structure of Methyl 1-oxo-3,4-diphenyl-2-(p-tolyl)-1,2-dihydroisoquinoline-7-carboxylate (34)](chart)

According to GP\textsubscript{2} with methyl 4-iodo-3-(p-tolylcarbamoyl)benzoate (79.2 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl\textsubscript{2} (11.1 mg, 0.02 mmol, 0.1 equiv), and Et\textsubscript{3}N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product 34 as white solid (72.8 mg, 82%).

Mp: 217-219 °C; \textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}) δ 9.21 (dd, J = 1.8, 0.6 Hz, 1H), 8.17 (dd, J = 8.4, 1.8 Hz, 1H), 8.17 (dd, J = 8.7, 0.6 Hz, 1H), 7.24-7.16 (m, 3H), 7.14-7.09
(m, 2H), 7.03-6.96 (m, 4H), 6.93-6.87 (m, 5H), 3.94 (s, 3H), 2.23 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 166.5, 162.4, 143.7, 140.9, 137.5, 136.4, 135.9, 134.5, 132.5, 131.5, 130.7, 130.5, 129.3, 129.0, 128.2, 128.1, 127.4, 127.1, 127.0, 125.8, 125.2, 118.4, 52.2, 21.0; HRMS (ESI) calculated for C$_{30}$H$_{24}$NO $[\text{M}+\text{H}]^+$ m/z 446.1751, found 446.1754.

5,7-Dimethyl-3,4-diphenyl-2-(p-tolyl)isoquinolin-1(2H)-one (35)

According to GP2 with 2-ido-3,5-dimethyl-N-(p-tolyl)benzamide (73.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.1 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl$_2$ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product 35 as white solid (40.5 mg, 49%). Mp: 159-161 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.34 (s, 1H), 7.25 (s, 1H), 7.07 (s, 5H), 6.99-6.91 (m, 4H), 6.88-6.79 (m, 5H), 2.45 (s, 3H), 2.21 (s, 3H), 1.75 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 162.9, 140.8, 139.9, 138.3, 137.1, 137.0, 136.6, 135.2, 135.0, 133.1, 132.0, 131.3, 129.2, 129.0, 127.3, 126.9, 126.74, 126.66, 126.5, 118.3, 23.7, 21.1; HRMS (ESI) calculated for C$_{30}$H$_{26}$NO $[\text{M}+\text{H}]^+$ m/z 416.2009, found 416.2009.

5-Methyl-3,4-diphenyl-2-(p-tolyl)isoquinolin-1(2H)-one (36)

According to GP2 with 2-ido-3-methyl-N-(p-tolyl)benzamide (70.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.4 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl$_2$ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 36 as white solid (60.5 mg, 75%).
Mp: 186-188 °C; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 8.55-8.52 (m, 1H), 7.40-7.39 (m, 2H), 7.08 (s, 5H), 6.99-6.92 (m, 4H), 6.84 (s, 5H), 2.20 (s, 3H), 1.78 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 162.9, 141.8, 139.8, 137.2, 136.9, 136.7, 135.5, 135.12, 135.10, 132.1, 131.2, 129.2, 129.0, 127.3, 127.1, 127.0, 126.80, 126.76, 126.6, 126.5, 118.3, 23.7, 21.0; HRMS (ESI) calculated for C$_{29}$H$_{24}$NO $[M+H]^+$ m/z 402.1852, found 402.1857.

6,7-Dimethoxy-3,4-diphenyl-2-(p-tolyl)isoquinolin-1(2H)-one (37)[7]

![Chemical structure of 6,7-Dimethoxy-3,4-diphenyl-2-(p-tolyl)isoquinolin-1(2H)-one (37)](image)

According to GP2 with 2-iodo-4,5-dimethoxy-N-(p-tolyl)benzamide (79.6 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.1 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl$_2$ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 $\mu$L, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 12/1) to afford the desired product 37 as white solid (78.9 mg, 88%). Mp: 293-295 °C; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.95 (s, 1H), 7.23-7.12 (m, 5H), 7.02-6.95 (m, 4H), 6.89 (s, 1H), 6.61 (s, 1H), 4.01 (s, 3H), 3.72 (s, 3H), 2.22 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 161.9, 153.3, 149.1, 139.8, 137.0, 136.9, 136.6, 134.9, 132.9, 131.4, 131.0, 129.1, 129.0, 127.9, 126.9, 126.7, 119.4, 118.2, 108.1, 105.9, 56.1, 55.7, 21.0; HRMS (ESI) calculated for C$_{30}$H$_{26}$NO$_3$ [M+H]$^+$ m/z 448.1907, found 448.1910.

7,8-Diphenyl-6-(p-tolyl)-[1,3]dioxolo[4,5-g]isoquinolin-5(6H)-one (38)

![Chemical structure of 7,8-Diphenyl-6-(p-tolyl)-[1,3]dioxolo[4,5-g]isoquinolin-5(6H)-one (38)](image)

According to GP2 with 6-iodo-N-(p-tolyl)benzo[d][1,3]dioxole-5-carboxamide (76.5 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.3 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl$_2$ (11.2 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 $\mu$L, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography
(petroleum ether/EtOAc = 10/1) to afford the desired product 38 as white solid (78.5 mg, 91%). Mp: 235-237 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 7.91 (s, 1H), 7.22-7.08 (m, 5H), 7.01-6.95 (m, 4H), 6.88 (s, 5H), 6.59 (s, 1H), 6.00 (s, 2H), 2.21 (s, 3H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 161.8, 152.0, 147.6, 140.0, 137.1, 136.9, 136.7, 135.0, 134.9, 131.5, 131.0, 129.13, 129.05, 127.9, 127.02, 126.96, 126.8, 121.0, 118.5, 106.0, 103.7, 101.6, 21.0; HRMS (ESI) calculated for C\(_{29}\)H\(_{22}\)NO\(_3\) [M+H]\(^+\) m/z 432.1594, found 432.1592.

3,4-Diphenyl-2-(p-tolyl)-6-(trifluoromethyl)isoquinolin-1(2H)-one (39)

![Chemical Structure of 39](attachment:image.png)

According to GP2 with 2-iodo-N-(p-tolyl)-4-(trifluoromethyl)benzamide (81.0 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.2 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl\(_2\) (10.8 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 39 as white solid (33.0 mg, 36%). Mp: 250-252 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.67 (d, \(J = 8.4\) Hz, 1H), 7.71 (dd, \(J = 8.4, 1.2\) Hz, 1H), 7.54-7.51 (m, 1H), 7.26-7.19 (m, 3H), 7.24 (s, 3H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 161.9, 142.8, 137.7, 137.6, 136.3, 135.3, 134.3, 134.0 (q, \(J = 31.9\) Hz), 131.4, 130.8, 129.39, 129.36, 128.9, 128.2, 127.6 (q, \(J = 1.1\) Hz), 127.4, 127.24, 127.15, 123.7 (q, \(J = 271.5\) Hz), 122.7 (q, \(J = 3.8\) Hz), 121.9, 21.0; HRMS (ESI) calculated for C\(_{29}\)H\(_{21}\)F\(_3\)NO [M+H]\(^+\) m/z 456.1570, found 456.1572.

3,4-Diphenyl-2-(p-tolyl)benzo[g]isoquinolin-1(2H)-one (40)

![Chemical Structure of 40](attachment:image.png)

According to GP2 with 3-iodo-N-(p-tolyl)-2-naphthamide (77.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (43.1 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl\(_2\) (11.1 mg,
0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 40 as white solid (86.0 mg, 98%). Mp: 236-238 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 9.17 (s, 1H), 8.09-8.06 (m, 1H), 7.77-7.34 (m, 1H), 7.67 (s, 1H), 7.53-7.46 (m, 2H), 7.27-7.19 (m, 5H), 7.02 (s, 2H), 6.94-6.86 (m, 5H), 2.23 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 163.2, 140.1, 137.1, 136.8, 136.7, 135.3, 135.0, 133.9, 131.7, 131.6, 131.0, 129.6, 129.3, 129.2, 128.0, 127.02, 126.98, 126.8, 126.0, 124.3, 124.0, 118.6, 21.0; HRMS (ESI) calculated for C$_{32}$H$_{24}$NO [M+H]$^+$ m/z 438.1852, found 438.1858.

2,3,4-Triphenylisoquinolin-1(2H)-one (41)[8]

![2,3,4-Triphenylisoquinolin-1(2H)-one (41)](image)

According to GP2 with 2-iodo-N-phenylbenzamide (64.7 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl$_2$ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product 41 as white solid (70.0 mg, 94%). Mp: 201-203 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.57 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.61-7.49 (m, 2H), 7.24-7.09 (m, 11H), 6.89 (s, 5H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 162.6, 141.1, 139.5, 137.6, 136.4, 134.8, 132.5, 131.6, 131.0, 129.5, 128.5, 128.2, 127.9, 127.5, 127.2, 127.1, 126.85, 126.83, 125.6, 118.8; HRMS (ESI) calculated for C$_{27}$H$_{20}$NO [M+H]$^+$ m/z 374.1539, found 374.1540.

2-(4-Methoxyphenyl)-3,4-diphenylisoquinolin-1(2H)-one (42)[9]

![2-(4-Methoxyphenyl)-3,4-diphenylisoquinolin-1(2H)-one (42)](image)

According to GP2 with 2-iodo-N-(4-methoxyphenyl)benzamide (71.2 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl$_2$ (11.1
mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 µL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 42 as white solid (73.9 mg, 92%).

Mp: 209-211 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.56 (dd, $J = 7.7$, 1.1 Hz, 1H), 7.58-7.47 (m, 2H), 7.26-7.11 (m, 6H), 7.02-6.99 (m, 2H), 6.95-6.84 (m, 5H), 6.73-6.70 (m, 2H), 3.68 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 162.7, 158.3, 141.3, 137.5, 136.3, 134.8, 132.3, 132.1, 131.5, 130.8, 130.2, 128.1, 127.8, 127.1, 127.0, 126.7, 125.4, 118.5, 113.7, 55.1; HRMS (ESI) calculated for C$_{28}$H$_{22}$NO$_2$ [M+H]$^+$ m/z 404.1645, found 404.1650.

2-(4-Fluorophenyl)-3,4-diphenylisoquinolin-1(2H)-one (43)$^{[9]}$

![Chemical structure of 2-(4-Fluorophenyl)-3,4-diphenylisoquinolin-1(2H)-one (43)](image)

According to GP2 with N-(4-fluorophenyl)-2-iodobenzamide (68.2 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyn (42.8 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl$_2$ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 µL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product 43 as white solid (71.1 mg, 91%).

Mp: 190-192 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.56 (d, $J = 7.8$ Hz, 1H), 7.61-7.50 (m, 2H), 7.27-7.06 (m, 8H), 6.91-6.86 (m, 7H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 162.7, 161.4 (d, $J = 245.3$ Hz), 140.8, 137.5, 136.1, 135.3 (d, $J = 3.8$ Hz), 134.5, 132.6, 131.5, 131.1, 131.0, 128.2, 128.0, 127.2, 127.2 (d, $J = 30.0$ Hz), 126.9, 125.6, 125.3, 119.0, 115.5 (d, $J = 22.5$ Hz); HRMS (ESI) calculated for C$_{27}$H$_{19}$FNO [M+H]$^+$ m/z 392.1445, found 392.1449.

2-Methyl-3,4-diphenylisoquinolin-1(2H)-one (44)$^{[9]}$

![Chemical structure of 2-Methyl-3,4-diphenylisoquinolin-1(2H)-one (44)](image)

According to GP2 with 2-iodo-N-methylbenzamide (52.2 mg, 0.20 mmol, 1.0 equiv),
1,2-diphenylethyne (43.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product 44 as white solid (26.6 mg, 43%). Mp: 152-154 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.58-8.55 (m, 1H), 7.56-7.46 (m, 2H), 7.26-7.11 (m, 9H), 7.08-7.05 (m, 2H), 3.36 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 162.7, 141.2, 137.1, 136.4, 135.0, 132.0, 131.5, 129.9, 128.2, 127.9, 127.8, 126.8, 126.6, 125.3, 124.9, 118.9, 34.3; HRMS (ESI) calculated for C₂₂H₁₈NO [M+H]+ m/z 312.1383, found 312.1389.

2-Benzyl-3,4-diphenylisoquinolin-1(2H)-one (45)⁹

According to GP2 with N-benzyl-2-iodobenzamide (67.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne (42.8 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (10.8 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 45 as white solid (15.8 mg, 20%). Mp: 228-230 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.63-8.60 (m, 1H), 7.56-7.47 (m, 2H), 7.18-7.08 (m, 8H), 7.07-7.01 (m, 4H), 6.90-6.87 (m, 4H), 5.22 (s, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 162.6, 141.3, 137.7, 137.3, 136.3, 134.3, 132.2, 131.4, 130.4, 128.2, 128.1, 128.0, 127.8, 127.5, 126.9, 126.8, 126.70, 126.65, 125.4, 125.1, 119.4, 49.0; HRMS (ESI) calculated for C₂₈H₂₂NO [M+H]+ m/z 388.1696, found 388.1697.

2,3,4-Tri-p-tolylisoquinolin-1(2H)-one (46)
According to GP2 with 2-iodo-\(N-(p\)-tolyl)benzamide (67.7 mg, 0.20 mmol, 1.0 equiv), 1,2-di-\(p\)-tolylethyne (49.8 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl\(_2\) (10.9 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 46 as white solid (78.5 mg, 94%). Mp: 84-86 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.54 (d, \(J = 7.8\) Hz, 1H), 7.56-7.45 (m, 2H), 7.25-7.22 (m, 1H), 7.00-6.97 (m, 8H), 6.78-6.68 (m, 4H), 2.27 (s, 3H), 2.22 (s, 3H), 2.07 (s, 3H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 162.8, 141.2, 137.9, 137.0, 136.9, 136.6, 136.1, 133.5, 132.2, 132.0, 131.4, 130.8, 129.2, 129.1, 128.6, 128.1, 127.7, 126.5, 125.5, 125.4, 118.6, 21.1, 21.03, 21.01; HRMS (ESI) calculated for C\(_{30}\)H\(_{26}\)NO \([\text{M+H}]^+\) m/z 416.2009, found 416.2014.

3,4-Bis(4-methoxyphenyl)-2-(p-tolyl)isoquinolin-1(2H)-one (47)

![Chemical structure of 3,4-Bis(4-methoxyphenyl)-2-(p-tolyl)isoquinolin-1(2H)-one (47)](image)

According to GP2 with 2-iodo-\(N-(p\)-tolyl)benzamide (67.7 mg, 0.20 mmol, 1.0 equiv), 1,2-bis(4-methoxy phenyl)ethyne (58.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl\(_2\) (11.1 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10/1) to afford the desired product 47 as white solid (80.7 mg, 90%). Mp: 245-247 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.54 (d, \(J = 7.5\) Hz, 1H), 7.57-7.44 (m, 2H), 7.27-7.24 (m, 1H), 7.04-6.94 (m, 6H), 6.80-6.73 (m, 4H), 6.48-6.38 (m, 2H), 3.73 (s, 3H), 3.59 (s, 3H), 2.23 (s, 3H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 162.8, 158.1, 158.0, 141.2, 138.0, 137.1, 137.0, 132.6, 132.3, 132.1, 129.3, 129.0, 128.8, 128.2, 127.4, 126.6, 125.5, 125.4, 118.4, 113.4, 112.5, 55.1, 54.9, 21.0; HRMS (ESI) calculated for C\(_{30}\)H\(_{26}\)NO\(_3\) \([\text{M+H}]^+\) m/z 448.1907, found 448.1910.
3,4-Bis(4-fluorophenyl)-2-(p-tolyl)isoquinolin-1(2H)-one (48)

According to GP2 with 2-iodo-N-(p-tolyl)benzamide (67.8 mg, 0.20 mmol, 1.0 equiv), 1,2-bis(4-fluorophenyl)ethyne (51.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (10.8 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 12/1) to afford the desired product 48 as white solid (82.3 mg, 97%). Mp: 186-188 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.58-8.54 (m, 1H), 7.62-7.50 (m, 2H), 7.22-7.19 (m, 1H), 7.10-7.02 (m, 4H), 6.97-6.82 (m, 6H), 6.67-6.59 (m, 2H), 2.25 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 161.7 (d, J = 245.3 Hz), 162.6, 161.4 (d, J = 246.8 Hz), 140.5, 137.6, 137.4, 136.6, 133.1 (d, J = 8.3 Hz), 132.7 (d, J = 8.3 Hz), 132.6, 132.2 (d, J = 3.8 Hz), 130.9 (d, J = 3.8 Hz), 129.5, 129.0, 128.4, 127.1, 125.6, 125.3, 118.0, 115.2 (d, J = 21.0 Hz), 114.4 (d, J = 21.8 Hz), 21.1; HRMS (ESI) calculated for C₂₉H₂₀F₂NO [M+H]⁺ m/z 424.1508, found 424.1506.

3,4-Bis(4-chlorophenyl)-2-(p-tolyl)isoquinolin-1(2H)-one (49)

According to GP2 with 2-iodo-N-(p-tolyl)benzamide (67.4 mg, 0.20 mmol, 1.0 equiv), 1,2-bis(4-chlorophenyl)ethyne (59.7 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude
reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 49 as white solid (84.9 mg, 93%). Mp: 145-147 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.55 (d, $J$ = 7.5 Hz, 1H), 7.60-7.49 (m, 2H), 7.24-7.17 (m, 3H), 7.06-7.02 (m, 4H), 6.96-6.91 (m, 4H), 6.83-6.81 (m, 2H), 2.25 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 162.5, 140.1, 137.7, 137.0, 136.3, 134.6, 133.4, 133.1, 133.0, 132.8, 132.6, 132.1, 129.5, 128.9, 128.43, 128.36, 127.6, 127.2, 125.5, 125.2, 117.7, 21.1; HRMS (ESI) calculated for C$_{28}$H$_{20}$Cl$_2$NO [M+H]$^+$ m/z 456.0917, found 456.0919.

3,4-Di-$m$-tolyl-2-(p-tolyl)isoquinolin-1(2H)-one (50)

According to GP2 with 2-iodo-N-(p-tolyl)benzamide (67.4 mg, 0.20 mmol, 1.0 equiv), 1,2-di-$m$-tolylethyne (50.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl$_2$ (10.9 mg, 0.02 mmol, 0.1 equiv), and Et$_3$N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 50 as white solid (53.1 mg, 64%). Mp: 83-85 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 8.55 (d, $J$ = 7.8 Hz, 1H), 7.58-7.50 (m, 2H), 7.27-7.24 (m, 1H), 7.14-7.06 (m, 1H), 6.98-6.90 (m, 7H), 6.80-6.67 (m, 4H), 2.22 (s, 6H), 2.02 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 162.7, 141.2, 137.7, 137.3, 137.1, 136.8, 136.3, 134.6, 132.3, 132.2, 131.8, 131.7, 129.0, 128.60, 128.57, 128.14, 128.06, 128.04, 127.72, 127.65, 127.4, 126.78, 126.74, 126.6, 125.6, 125.4, 118.7, 21.2, 21.02, 20.96; HRMS (ESI) calculated for C$_{30}$H$_{26}$NO [M+H]$^+$ m/z 416.2009, found 416.2015.

3,4-Di(thiophen-3-yl)-2-(p-tolyl)isoquinolin-1(2H)-one (51)
According to GP2 with 2-iodo-N-(p-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), 1,2-di(thiophen-3-yl)ethyne (46.2 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product 51 as white solid (71.7 mg, 90%). Mp: 207-209 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.53 (d, J = 7.8 Hz, 1H), 7.63-7.58 (m, 1H), 7.53-7.49 (m, 1H), 7.40 (d, J = 7.8 Hz, 1H), 7.25-7.21 (m, 1H), 7.07-6.98 (m, 5H), 6.89-6.85 (m, 2H), 6.73-6.72 (m, 1H), 6.53-6.52 (m, 1H), 2.28 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 162.6, 137.4, 137.3, 137.0, 136.8, 136.2, 134.8, 132.5, 130.1, 129.3, 129.2, 128.6, 126.9, 126.6, 125.4, 125.3, 125.1, 124.9, 124.1, 114.2, 21.1; HRMS (ESI) calculated for C₂₄H₁₈NO₂ [M+H]⁺ m/z 400.0824, found 400.0831.

3,4-Diethyl-2-(p-tolyl)isoquinolin-1(2H)-one (52)

According to GP2 with 2-iodo-N-(p-tolyl)benzamide (68.0 mg, 0.20 mmol, 1.0 equiv), hex-3-yne (22.6 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 52 as white solid (56.4 mg, 97%). Mp: 111-113 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.45 (d, J = 7.8 Hz, 1H), 7.74-7.66 (m, 2H), 7.46-7.42 (m, 1H), 7.36-7.26 (m, 2H), 7.20-7.09 (m, 2H), 2.81 (q, J = 7.5 Hz, 2H), 2.50-2.43 (m, 5H), 1.28 (t, J = 7.2 Hz, 3H), 1.00 (t, J = 7.4 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 163.2, 141.2, 138.2, 136.9, 132.4, 130.0, 128.6, 125.7, 125.4, 122.6, 114.6, 23.1, 21.2, 20.4, 14.7, 13.9; HRMS (ESI) calculated for C₂₀H₂₂NO [M+H]⁺ m/z 292.1696, found 292.1700.
3,4-Dipropyl-2-(p-tolyl)isoquinolin-1(2H)-one (53)[7]

According to GP2 with 2-iodo-N-(p-tolyl)benzamide (67.4 mg, 0.20 mmol, 1.0 equiv), oct-4-yne (26.9 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 53 as yellow oil (58.8 mg, 92%). ¹H NMR (300 MHz, CDCl₃) δ 8.44 (d, J = 7.8 Hz, 1H), 7.69-7.63 (m, 2H), 7.44-7.39 (m, 1H), 7.31-7.28 (m, 2H), 7.13-7.10 (m, 2H), 2.75-2.69 (m, 2H), 2.42 (s, 3H), 2.39-2.33 (m, 2H), 1.70-1.62 (m, 2H), 1.45-1.35 (m, 2H), 1.09 (t, J = 7.4 Hz, 3H), 0.71 (t, J = 7.4 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 163.1, 140.4, 138.1, 137.1, 136.9, 132.3, 129.9, 128.53, 128.50, 125.7, 125.3, 122.7, 113.5, 32.2, 29.7, 23.6, 22.8, 21.2, 14.4, 14.1; HRMS (ESI) calculated for C₂₂H₂₆NO [M+H]+ m/z 320.2009, found 320.2011.

3,4-Dibutyl-2-(p-tolyl)isoquinolin-1(2H)-one (54)[8]

According to GP2 with 2-iodo-N-(p-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), dec-5-yne (34.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 54 as yellow oil (62.3 mg, 90%). ¹H NMR (300 MHz, CDCl₃) δ 8.44 (d, J = 7.8 Hz, 1H), 7.69-7.62 (m, 2H), 7.43-7.38 (m, 1H), 7.30-7.27 (m, 2H), 7.13-7.10 (m, 2H), 2.76-2.71 (m, 2H), 2.41 (s, 3H), 2.41-2.36 (m, 2H), 1.67-1.47 (m, 4H), 1.45-1.33 (m, 2H), 1.16-1.07 (m, 2H), 1.00 (t, J = 7.1 Hz, 3H), 0.70 (t, J = 7.4 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 163.0, 140.2, 138.0, 137.0,
According to GP2 with 2-iodo-N-(p-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), 1,4-dimethoxybut-2-yn-2-ylbenzene (29.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 10/1) to afford the desired product 55 as white solid (55.9 mg, 86%). Mp: 166-168 °C; 

\[ ^{1}H\ NMR\ (300\ MHz,\ CDCl_{3})\ \delta\ 8.43\ (d,\ J = 8.1\ Hz,\ 1H),\ 7.86\ (d,\ J = 8.1\ Hz,\ 1H),\ 7.74-7.69\ (m,\ 1H),\ 7.52-7.47\ (m,\ 1H),\ 7.32-7.26\ (m,\ 2H),\ 7.19-7.16\ (m,\ 2H),\ 4.69\ (s,\ 2H),\ 4.11\ (s,\ 2H),\ 3.50\ (s,\ 3H),\ 3.04\ (s,\ 3H),\ 2.43\ (s,\ 3H);\ ^{13}C\ NMR\ (75\ MHz,\ CDCl_{3})\ \delta\ 162.9,\ 138.6,\ 138.4,\ 136.3,\ 135.8,\ 132.6,\ 129.7,\ 128.7,\ 128.2,\ 127.2,\ 126.2,\ 123.7,\ 113.2,\ 67.3,\ 67.1,\ 58.2,\ 58.1,\ 21.1;\ HRMS\ (ESI)\ calculated\ for\ C_{20}H_{22}NO_{3} [M+H]^+ m/z 324.1594,\ found\ 324.1601.\]

According to GP1 with 2-iodo-N-(p-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), prop-1-yn-1-ylbenzene (29.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (10.8 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc =
20/1) to afford the desired product 56+56' as white solid (60.3 mg, 93%, 56/56' = 1.1:1). 1H NMR (300 MHz, CDCl3) δ 56+56' 8.45 (d, J = 7.8 Hz, 1H), 8.37 (dd, J = 8.0, 1.1 Hz, 1H), 7.65-7.63 (m, 2H), 7.45-7.27 (m, 6H), 7.23-7.19 (m, 4H), 7.09-7.05 (m, 5H), 6.97-6.95 (m, 3H), 6.89-6.86 (m, 2H), 6.82-6.79 (m, 2H), 2.32 (s, 3H), 2.11 (s, 3H), 1.98 (s, 3H), 1.70 (s, 3H); 13C NMR (75 MHz, CDCl3) δ 56 162.5, 140.3, 137.5, 137.0, 136.9, 135.4, 132.4, 130.4, 129.08, 129.06, 128.4, 127.7, 127.6, 126.5, 125.7, 123.2, 110.1, 20.9, 14.7; 56' 163.0, 138.2 137.7, 137.3, 136.8, 132.1, 130.9, 130.1, 128.7, 128.1, 127.9, 127.4, 125.8, 124.8, 117.4, 21.1, 19.4; HRMS (ESI) calculated for C23H20NO [M+H]+ m/z 326.1539, found 326.1542.

3-Phenyl-2-(p-tolyl)isoquinolin-1(2H)-one (57) and 4-phenyl-2-(p-tolyl)isoquinolin-1(2H)-one (57')[10]

According to GP1 with 2-iodo-N-(p-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), ethynylbenzene (25.2 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl2 (11.0 mg, 0.02 mmol, 0.1 equiv), and Et3N (56 μL, 0.4 mmol, 2.0 equiv). The crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 20/1) to afford the desired product 57+57' as yellow oil (56.7 mg, 91%, 57/57' = 1:1). 1H NMR (300 MHz, CDCl3) δ 57+57' 8.57 (d, J = 7.8 Hz, 1H), 8.46 (dd, J = 8.1, 1.1 Hz, 1H), 7.70-7.40 (m, 11H), 7.37-7.25 (m, 4H), 7.17 (s, 6H), 7.07-6.98 (m, 4H), 6.58 (s, 1H), 2.41 (s, 3H), 2.27 (s, 3H); 13C NMR (75 MHz, CDCl3) δ 57 163.2, 143.7, 137.4, 136.7, 136.4, 136.2, 132.7, 129.3, 129.2, 129.0, 128.3, 127.8, 127.7, 126.8, 126.0, 125.4, 107.8, 21.1; 57' 161.7, 138.7, 138.0, 136.4, 136.3, 132.4, 131.3, 129.92, 129.86, 128.7, 128.6, 127.9, 127.1, 126.5, 126.3, 124.7, 119.5, 21.1; HRMS (ESI) calculated for C22H18NO [M+H]+ m/z 312.1383, found 312.1389.
**Gram-scale reactions**

\[
\text{N-(2-iodophenyl)acetamide } 1 \ (1.31 \text{ g}, \ 5.0 \text{ mmol}, \ 1.0 \text{ equiv}), \ 1,2\text{-diphenylethyne } 2 \ (1.34 \text{ g}, \ 7.5 \text{ mmol}, \ 1.5 \text{ equiv}), \ \text{Ni(dppp)Cl}_2 \ (0.27 \text{ g}, \ 0.5 \text{ mmol}, \ 0.1 \text{ equiv}), \ \text{and Et}_3\text{N} \ (1.40 \text{ mL}, \ 10.0 \text{ mmol}, \ 2.0 \text{ equiv}) \ \text{were placed in a dry 100 mL Schlenk tube under a nitrogen atmosphere. Dry CH}_3\text{CN (15.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h monitored with TLC. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH}_2\text{Cl}_2. \ \text{The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 3 (1.27 g, 82%).}
\]

\[
\text{2-Iodo-N-}(p\text{-tolyl})\text{benzamide } 29 \ (1.35 \text{ g}, \ 4.0 \text{ mmol}, \ 1.0 \text{ equiv}), \ 1,2\text{-diphenylethyne } 2 \ (0.86 \text{ g}, \ 4.8 \text{ mmol}, \ 1.2 \text{ equiv}), \ \text{Ni(dppp)Cl}_2 \ (0.22 \text{ g}, \ 0.4 \text{ mmol}, \ 0.1 \text{ equiv}), \ \text{and Et}_3\text{N} \ (1.10 \text{ mL}, \ 8.0 \text{ mmol}, \ 2.0 \text{ equiv}) \ \text{were placed in a dry 100 mL Schlenk tube under a nitrogen atmosphere. Dry CH}_3\text{CN (30.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h monitored with TLC. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH}_2\text{Cl}_2. \ \text{The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 30 (1.39 g, 90%).}
\]
Mechanistic studies

Radical inhibition experiments:

\[
\begin{align*}
\text{2-Iodo-}N\text{-}(p\text{-tolyl})\text{benzamide} & : \quad \text{Ni(dppp)}\text{Cl}_2 (10\,\text{mol}%) \\
& \quad \text{Et}_3\text{N} (2.0\,\text{equiv}) \\
& \quad \text{CH}_3\text{CN, }40\,^\circ\text{C, }6\,\text{h} \\
& \quad \text{TEMPO} (2.0\,\text{equiv}) \\
& \quad 30, 0\% \\
\end{align*}
\]

\[
\begin{align*}
N\text{-}(2\text{-iodophenyl})\text{acetamide} & : \quad \text{Ni(dppp)}\text{Cl}_2 (10\,\text{mol}%) \\
& \quad \text{Et}_3\text{N} (2.0\,\text{equiv}) \\
& \quad \text{CH}_3\text{CN, }40\,^\circ\text{C, }12\,\text{h} \\
& \quad \text{TEMPO} (2.0\,\text{equiv}) \\
& \quad 3, 76\% \\
\end{align*}
\]

N-(2-iodophenyl)acetamide 1 (53.2 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne 2 (54.3 mg, 0.30 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.2 mg, 0.02 mmol, 0.1 equiv), Et₃N (56 μL, 0.40 mmol, 2.0 equiv), and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (63.0 mg, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH₃CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. The formation of 3 was completely suppressed.
(54.0 mg, 0.30 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.2 mg, 0.02 mmol, 0.1 equiv), Et₃N (56 μL, 0.40 mmol, 2.0 equiv), and 2,6-di-tert-butyl-4-methylphenol (BHT) (88.5 mg, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH₃CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH₂Cl₂. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 3 (47.4 mg, 76%).

2-Iodo-N-(p-tolyl)benzamide 29 (67.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne 2 (43.0 mg, 0.24 mmol, 1.2 equiv), Ni(dppp)Cl₂ (10.9 mg, 0.02 mmol, 0.1 equiv), Et₃N (56 μL, 0.4 mmol, 2.0 equiv), and 2,6-di-tert-butyl-4-methylphenol (BHT) (88.2 mg, 0.4 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH₃CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH₂Cl₂. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 30 (72.3 mg, 93%).

**Radical clock experiments:**

*N-(2-iodophenyl)acetamide* (53.1 mg, 0.20 mmol, 1.0 equiv), (1-cyclopropylvinyl)benzene (59.2 mg, 0.40 mmol, 2.0 equiv), Ni(dppp)Cl₂ (11.0 mg,
0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH₃CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no ring-opening product was observed.

2-Iodo-N-(p-tolyl)benzamide (67.9 mg, 0.20 mmol, 1.0 equiv), (1-cyclopropylvinyl)benzene (44.9 mg, 0.30 mmol, 1.5 equiv), Ni(dppp)Cl₂ (11.0 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH₃CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. In this reaction, no ring-opening product was observed.

**Radical trapping experiments:**

\[
\text{N-(2-iodophenyl)acetamide (52.8 mg, 0.20 mmol, 1.0 equiv), ethene-1,1-diyl dibenzene (72.3 mg, 0.40 mmol, 2.0 equiv), Ni(dppp)Cl₂ (11.1 mg, 0.02 mmol, 0.1 equiv), and Et₃N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH₃CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no radical trapping product was observed.} \]
2-Iodo-\( N\)-(p-tolyl)benzamide (67.6 mg, 0.20 mmol, 1.0 equiv), ethene-1,1-diyl dibenzene (36.1 mg, 0.20 mmol, 1.0 equiv), Ni(dppp)Cl\(_2\) (11.0 mg, 0.02 mmol, 0.1 equiv), and Et\(_3\)N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH\(_3\)CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. In this reaction, no radical trapping product was observed.

**Reactions with Ni(0) catalyst:**

\[ \text{N-(2-iodophenyl)acetamide 1 (52.8 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne 2 (54.2 mg, 0.30 mmol, 1.5 equiv), Ni(cod)\(_2\) (5.6 mg, 0.02 mmol, 0.1 equiv), dppp (12.7 mg, 0.03 mmol, 0.15 equiv), and Et\(_3\)N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH\(_3\)CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH\(_2\)Cl\(_2\). The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 40/1) to afford the desired product 3 (60.3 mg, 97%).} \]

2-Iodo-\( N\)-(p-tolyl)benzamide 29 (67.1 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne 2 (43.6 mg, 0.24 mmol, 1.2 equiv), Ni(cod)\(_2\) (5.5 mg, 0.02 mmol, 0.1 equiv), dppp
(12.4 mg, 0.03 mmol, 0.15 equiv), and Et₃N (56 μL, 0.40 mmol, 2.0 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH₃CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. After completion of the reaction, it was transferred to a round-bottomed flask after dilution with CH₂Cl₂. The solvent was removed under reduced pressure and the crude reaction mixture was purified by flash silica gel column chromatography (petroleum ether/EtOAc = 15/1) to afford the desired product 30 (73.5 mg, 95%).

**Role of Et₃N:**

\[
\begin{align*}
\text{Ph} & \quad \text{Ph} \\
\text{N} & \quad \text{I} \\
\text{NHAc} & \quad \text{Ph} \\
\text{1} & \quad \text{2} \\
\end{align*}
\]

\[
\begin{align*}
\text{Ni(dppp)}\text{Cl}_2 (10 \text{ mol\%}) \\
\text{CH}_3\text{CN, 40 °C, 12 h} \\
\end{align*}
\]

\[
\text{Ph} \\
\text{N} \\
\text{Ac} \\
3, 0 \%
\]

\[\text{N-(2-iodophenyl)acetamide 1 (53.1 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne 2 (54.1 mg, 0.30 mmol, 1.5 equiv), and Ni(dppp)Cl}_2 (11.2 mg, 0.02 mmol, 0.1 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH}_3\text{CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 12 h. In this reaction, no product 3 was observed.}

\[
\begin{align*}
\text{Ph} & \quad \text{Ph} \\
\text{N} & \quad \text{Me} \\
\text{H} & \quad \text{I} \\
\text{29} & \quad \text{2} \\
\end{align*}
\]

\[
\begin{align*}
\text{Ni(dppp)}\text{Cl}_2 (10 \text{ mol\%}) \\
\text{CH}_3\text{CN, 40 °C, 6 h} \\
\end{align*}
\]

\[
\text{Ph} \\
\text{N} \\
\text{Ph} \\
\text{Me} \\
30, 0 \%
\]

2-Iodo-N-(p-tolyl)benzamide 29 (67.7 mg, 0.20 mmol, 1.0 equiv), 1,2-diphenylethyne 2 (42.8 mg, 0.24 mmol, 1.2 equiv), and Ni(dppp)Cl₂ (11.2 mg, 0.02 mmol, 0.1 equiv) were placed in a dry 10 mL Schlenk tube under a nitrogen atmosphere. Dry CH₃CN (2.0 mL) was added with a syringe and the reaction mixture was stirred at room temperature for 6 h. In this reaction, no product 30 was observed.
Nickel-catalyzed Larock-type heteroannulation reactions of ortho-bromo substrates with 1,2-diphenylethyne
References:

$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
\[ \text{\textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3})} \]

![NMR Spectrum Image](image-url)
$^{13}$C NMR (75 MHz, CDCl$_3$)
\[ ^1H \text{NMR (300 MHz, CDCl}_3 \]
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
\textbf{\textsuperscript{1}H NMR} (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}\text{C NMR} \ (75 \text{ MHz, CDCl}_3)$
$^{1}$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}$$H$ NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}\text{H NMR}$ (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl$_3$)
$^{13}C$ NMR (75 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
\[1H\text{ NMR (300 MHz, CDCl}_3\text{)}\]
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl$_3$)
13C NMR (75 MHz, CDCl₃)
$^{1}$H NMR (300 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$\text{S77}$
22

$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$1^\text{H} \text{NMR (300 MHz, CDCl}_3)$
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{13}\text{C NMR}$ (75 MHz, CDCl$_3$)
$^{1}$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
\[ ^{13}\text{C NMR (75 MHz, CDCl}_3 \text{)} \]
$^1\text{H NMR}$ (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}{H}$ NMR (300 MHz, CDCl$_3$)
13C NMR (75 MHz, CDCl₃)
34

$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}$H NMR (300 MHz, CDCl$_3$)

![Chemical Structure](image)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl₃)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}\text{H NMR (300 MHz, CDCl}_3\text{)}$
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}\text{H NMR (300 MHz, CDCl}_3\text{)}$
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)


$\text{^1H NMR (300 MHz, CDCl}_3\text{)}$

46
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^1$H NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)

48

![Chemical Structure](image)
$^1$H NMR (300 MHz, CDCl$_3$)

49
$^{13}$C NMR (75 MHz, CDCl$_3$)
\[ \text{\textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3})} \]
\[^{1}\text{H NMR} (300\text{ MHz, CDCl}_3)\]
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{1}H$ NMR (300 MHz, CDCl$_3$)

55
1H NMR (300 MHz, CDCl₃)

S143
$^{13}$C NMR (75 MHz, CDCl$_3$)
$^{13}$C NMR (75 MHz, CDCl$_3$)