SUPPORTING INFORMATION

An expedient approach to 1, 2-dihydroisoquinoline derivatives via Cobalt catalysed 6-endo dig cyclization followed by Mannich condensation of o-alkynylarylaldimines

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<thead>
<tr>
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1. X-ray crystal structure and data of 14i

The single crystal X-ray data were collected on an Oxford Xcalibur CCD diffractometer using graphite monochromated MoKα radiation (λ = 0.71073 Å) at 293(2) K. The multi-scan correction was applied. The structure was solved by the direct method using SIR-92 and refined by full-matrix least-square refinement technique on F₁ using SHELXL97. The hydrogen atom were placed into the calculated position and included in the last cycle of the refinement. All calculations were done using Wingx software package. The main crystallographic data and structural refinement details are given in Table 1. Atomic coordinates, bond lengths, bond angles, and thermal parameters for compound 14i have been deposited at the Cambridge Crystallographic Data Centre (CCDC, deposit no. 868792).

![Fig. 1. X-ray crystallographic ORTEP diagram of compound 14i](image)

![Fig. 2. Hydrogen bonding in the crystal lattice of (14i).](image)

**Table 1. Crystal data of 14i**
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<th><strong>Empirical Formula</strong></th>
<th>$C_{25}H_{20}ClF_2NO_2$</th>
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<td><strong>Wavelength</strong></td>
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<td><strong>Space group</strong></td>
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<td><strong>b</strong></td>
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<td><strong>Crystal size</strong></td>
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<td>Final R indices [I&gt;2sigma (I)]&lt;sup&gt;a,b&lt;/sup&gt;</td>
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<td>R indices (all data)</td>
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<td>Largest diff. peak and hole</td>
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</table>

<sup>a</sup>R = Σ(‖Fo‖ - |Fc|)/Σ |Fo|;  <sup>b</sup>R<sub>W</sub> = (Σ[w(Fo<sup>2</sup> - Fc<sup>2</sup>)<sup>2</sup>]/Σ[w(Fo<sup>2</sup>)])<sup>1/2</sup>

**References:**

Typical experimental procedure: Synthesis of 1a-l:
To the solution of the 2-(1-alkynyl)benzaldehyde (5.0 mmol) in ethanol was added R-NH$_2$ (1.2 equiv.). The reaction mixture was then stirred at room temperature under Argon atmosphere for 10 h. The reacting mixture was then filtered. Removal of solvent from filtrate afforded 90-92% yield product.

Typical experimental procedure: Synthesis of 2a-l:
To a mixture of 1a-l (0.5 mmol) and CoCl$_2$ (30 mol%) in dichloroethane (5 mL) was added nitromethane (1 mmol) at room temperature under Ar gas atmosphere. The resulting mixture was stirred for 5-8 hours at 80 °C, and then it was cooled to room temperature. After addition of a saturated aqueous NaHCO$_3$, the mixture was extracted with AcOEt (3x). The combined extracts were washed with brine, dried over MgSO$_4$, filtered, and the organic solvent was evaporated under reduced pressure. The crude product was purified by basic silica gel column chromatography using a mixture of hexane-AcOEt (8:1 to 5:1) as an eluent to give 2a-l in 58-90% yield.

Typical experimental procedure: Synthesis of 3:
To a mixture of 1a (0.5 mmol) and CoCl$_2$ (30 mol%) in dichloroethane (5 mL) was added nitroethane (1 mmol) at room temperature under Ar gas atmosphere. The resulting mixture was stirred for 12 hours at 80 °C, and then it was cooled to room temperature. After addition of a saturated aqueous NaHCO$_3$, the mixture was extracted with AcOEt three times. The combined extracts were washed with brine, dried over MgSO$_4$, filtered, and the organic solvent was evaporated under reduced pressure. The crude product was purified by basic silica gel column chromatography using a mixture of hexane-AcOEt (4:1) as an eluent to get 3 in 65% yield.

Typical experimental procedure: Synthesis of 10:
To a mixture of 1a (0.5 mmol) and CoCl$_2$ (30 mol%) in dichloroethane (5 mL) and triflic acid (1 mmol) was added at room temperature under Ar gas atmosphere. The resulting mixture was stirred for 4 hours at 80 °C, and then it was cooled to room temperature. The solvent was evaporated under reduced pressure. The crude product was crystalized by hexane and ethyl acetate to get 10 in 99% yield.

1,2-Dihydro-1-(nitromethyl)-2,3-diphenylisoquinoline (2a)

152mg, 90% Yield, Light Brown Solid: mp 180-182°C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 7.54-7.52 (m, 2H), 7.33-7.32 (m, 2H), 7.28-7.19 (m, 4H), 7.13-7.06 (m, 3H), 6.91-6.88 (m, 3H), 6.77 (s, 1H), 5.74 (dd, $J$=11, 4.28 Hz, 1H), 4.80 (t, $J$=11.2 Hz, 1H), 4.37 (dd, $J$=11.6, 4.28 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ ppm 146.0, 140.6, 136.4, 132.1, 128.8, 128.75, 128.43, 128.4, 127.5, 127.1, 126.4, 125.7, 124.9, 123.3, 122.9, 111.8, 76.5, 64.2; HRMS (ESI) $m$/z calculated for C$_{22}$H$_{18}$N$_2$O$_2$ (M+H)$^+$, calcd 343.1446, found 343.1252.
2-(4-Fluorophenyl)-1,2-dihydro-1-(nitromethyl)-3-phenyloisoquinoline (2b)

117 mg, 65% Yield, Light Brown Solid: mp 201-202°C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.53-7.50 (m, 2H), 7.34-7.31 (m, 2H), 7.29-7.21 (m, 4H), 7.13 (d, \(J=7.36\) Hz, 1H), 6.88-6.83 (m,2H), 6.79-6.74 (m, 3H), 5.63 (dd, \(J = 11.0, 4.28\) Hz, 1H), 4.77 (t, \(J=11.6\) Hz, 1H), 4.35 (dd, \(J=12.2, 4.28\) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 140.8, 136.2, 132.1, 128.9, 128.6, 128.5, 127.7, 127.4, 126.1, 125.8, 125.0, 124.8, 124.7, 115.8, 115.5, 111.6, 64.6; HRMS (ESI) \(m/z\) calculated for C\(_{22}\)H\(_{17}\)FN\(_2\)O\(_2\)(M+H)\(^+\), calc 361.1352, found 361.1172.

1,2-Dihydro-1-(nitromethyl)-3-phenyl-2-p-tolylisoquinoline (2c)

141 mg, 80% Yield, Dark Brown Solid: mp 130-132°C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.40 (d, \(J = 8.5\) Hz, 2H), 7.35-7.30 (m, 2H), 7.23-7.19 (m, 1H), 7.16-7.11 (m, 2H), 7.07 (d, \(J=7.96\) Hz, 2H), 6.87-6.84 (m, 2H), 6.78-6.72 (m, 3H), 5.62 (dd, \(J=11.0, 4.28\) Hz, 1H), 4.77 (t, \(J=11.0\) Hz, 1H), 4.34 (dd, \(J=11.6, 4.24\) Hz, 1H), 2.29 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 142.4, 140.8, 138.7, 133.4, 132.3, 129.2, 128.9, 127.6, 127.2, 126.1, 125.7, 124.9, 124.7, 115.7, 115.5, 110.9, 76.7, 64.6, 21.2; HRMS (ESI) \(m/z\) calculated for C\(_{23}\)H\(_{20}\)N\(_2\)O\(_2\)(M+H)\(^+\), calc 357.1602, found 357.1234.

1,2-Dihydro-1-(nitromethyl)-2-phenyl-3-p-tolylisoquinoline (2d)

109 mg, 62% yield, Yellow Solid: mp 151-153°C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.55-7.52 (m, 2H), 7.32-7.31 (m, 2H), 7.26-7.25 (m, 1H), 7.24-7.22 (m, 3H), 7.12-7.09 (m, 1H), 6.89-6.87 (m, 2H), 6.81-6.77 (m, 2H), 6.74 (s, 1H), 5.68 (dd, \(J=10.9, 3.6\) Hz, 1H), 4.78 (t, \(J=11.0\) Hz, 1H), 4.35 (dd, \(J=11.6, 4.28\) Hz, 1H), 2.16 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 143.8, 141.1, 136.7, 133.2, 132.4, 129.6, 128.8, 128.5, 127.8, 127.2, 126.5, 125.9, 124.9, 123.2, 111.4, 76.8, 64.6, 20.7; HRMS (ESI) \(m/z\) calculated for C\(_{23}\)H\(_{20}\)N\(_2\)O\(_2\)(M+H)\(^+\), calc 357.1602, found 357.1415.

2-(4-Fluorophenyl)-1,2-dihydro-1-(nitromethyl)-3-p-tolylisoquinoline (2e)

145mg, 78% yield, dark brown solid: mp 130-132°C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.43-7.41 (m, 2H), 7.32-7.30 (m, 2H), 7.22-7.18 (m, 1H), 7.12-7.05 (m, 4H), 6.91-6.86 (m, 3H), 6.74(s, 1H), 5.73 (dd, \(J=11, 4.28\) Hz, 1H), 4.79 (t, \(J=11.6\) Hz, 1H), 4.36 (dd, \(J=11.6, 4.28\) Hz, 1H), 2.28 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 146.2, 140.7, 138.5, 133.6, 132.3, 129.2, 128.8, 128.75, 127.5, 127.0, 126.5, 125.7, 124.8, 123.3, 123.0, 111.1, 76.6, 64.3, 21.2; HRMS (ESI) \(m/z\) calculated for C\(_{23}\)H\(_{19}\)FN\(_2\)O\(_2\)(M+H)\(^+\), calc 375.1508, found 375.1535.

7-Fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)-2-phenyloisoquinoline (2f)

127mg, 66% yield, yellow solid: mp 152-154°C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.45 (d, \(J=8.4\) Hz, 2H), 7.27-7.23 (m, 1H), 7.11-7.07 (m, 2H), 7.04-6.99 (m, 1H), 6.91-6.87 (m, 3H), 6.85-6.83 (dd, \(J=7.6, 2.2\) Hz, 1H), 6.79-6.76 (m, 2H), 6.66 (s, 1H), 5.67 (dd, \(J=10.7, 4.6\) Hz, 1H), 4.78 (t, \(J=11.4, 4.6\) Hz, 1H), 4.36 (dd, \(J=11.4, 3.8\) Hz, 1H), 3.74 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 162.9, 160.0, 146.0, 139.9, 128.9, 128.8, 126.3, 123.5, 123.1, 116.0, 115.8, 113.9, 112.8, 112.6, 109.4, 76.2, 64.0, 55.2; HRMS (ESI) \(m/z\) calculated for C\(_{23}\)H\(_{19}\)FN\(_2\)O\(_3\)(M+H)\(^+\), calc 391.1452, found 391.1304. 

S6
2-(4-Chlorophenyl)-7-fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)isoquinoline (2g)

150 mg, 72% yield, pale yellow solid: mp 163-164°C. 1H NMR (400 MHz, CDCl3) δ ppm 7.42-7.40 (m, 2H), 7.29-7.26 (m, 1H), 7.06-7.01 (m, 3H), 6.88-6.78 (m, 3H), 6.68 (s, 1H), 5.61 (dd, J=11.0, 3.8 Hz, 1H), 4.77 (t, J=11.0 Hz, 1H), 4.36 (dd, J=11.6, 4.28 Hz, 1H), 3.77 (s, 3H); 13C NMR (100 MHz, CDCl3) δ ppm 160.5, 160.1, 144.7, 139.5, 129.9, 128.8, 128.3, 127.8, 126.4, 124.2, 116.2, 115.9, 114.1, 112.8, 112.6, 109.8, 76.1, 63.9, 55.2; HRMS (ESI) m/z calculated for C23H18ClFN3O3 (M+H)+, calcd 425.1068, found 425.0950.

7-Fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)-2-p-tolylisoquinoline (2h)

131 mg, 66% yield, pale yellow solid, mp 149-151°C; 1H NMR (400 MHz, CDCl3) δ ppm 7.74 (d, J=8.5 Hz, 2H), 7.27-7.23 (m, 2H), 7.04-6.99 (m, 1H), 6.90-6.88 (m, 2H), 6.83 (dd, J=8.5, 2.4 Hz, 1H), 6.79-6.76 (m, 3H), 6.63 (s, 1H), 5.61 (dd, J=11.0, 3.68 Hz, 1H), 4.77 (t, J=11.0 Hz, 1H), 4.35 (dd, J=11.6, 4.2 Hz, 1H), 3.75 (s, 3H); 13C NMR (100 MHz, CDCl3) δ ppm 160.4, 159.8, 143.6, 140.2, 143.3, 129.6, 128.9, 127.8, 126.1, 123.1, 115.9, 115.7, 113.9, 112.8, 112.6, 109.0, 76.3, 64.2, 55.2, 20.6; HRMS (ESI) m/z calculated for C24H22FN3O3 (M+H)+, calcd 405.1614, found 405.1483.

2-(4-Chlorophenyl)-1,2-dihydro-7-nitro-1-(nitromethyl)-3-phenylisoquinoline (2i)

146 mg, 70% yield, yellow solid, mp 197-198°C; 1H NMR (400 MHz, CDCl3) δ ppm 8.22 (dd, J=8.5, 2.4 Hz, 1H), 8.07 (d, J=2.4 Hz, 1H), 7.52-7.49 (m, 2H), 7.45 (d, J=8.5 Hz, 1H), 7.32-7.30 (m, 2H), 7.08 (d, J=8.5 Hz, 2H), 6.84-6.79 (m, 3H), 5.78 (dd, J=11.0, 3.68 Hz, 1H), 4.84 (t, J=11.6 Hz, 1H), 4.42 (dd, J=11.6, 4.2 Hz, 1H); 13C NMR (100 MHz, CDCl3) δ ppm 146.4, 145.1, 144.0, 138.1, 135.2, 130.2, 129.9, 129.3, 128.8, 126.1, 125.1, 124.9, 124.5, 121.6, 110.0, 75.8, 64.0; Calcd. Getting decomposed while doing mass spectrometry. Despite several attempts mass could not be obtained.

1,2-Dihydro-3-(4-methoxyphenyl)-7-nitro-1-(nitromethyl)-2-phenylisoquinoline (2j)

129 mg, 62% yield, orange solid: mp 81-83°C. 1H NMR (400 MHz, CDCl3) δ ppm 8.12 (dd, J=7.96, 1.8 Hz, 1H), 7.98 (s, 1H), 7.40 (d, J=8.5 Hz, 2H), 7.35-7.32 (m, 1H), 7.07-7.03 (m, 2H), 6.91-6.87 (m, 1H), 6.82-6.80 (m, 2H), 6.73 (d, J=8.5 Hz, 2H), 6.63 (s, 1H), 5.73 (dd, J=11.0, 4.2 Hz, 1H), 4.77 (t, J=11.0 Hz, 1H), 4.35 (dd, J=11.0, 4.24 Hz, 1H); 13C NMR (100 MHz, CDCl3) δ ppm 160.4, 145.5, 145.2, 145.0, 138.4, 129.3, 128.8, 127.6, 125.5, 124.3, 124.2, 124.0, 123.5, 121.3, 113.8, 107.9, 75.6, 63.7, 54.9; HRMS (ESI) m/z calculated for C23H19N3O3 (M+H)+, calcd 418.1402, found 418.1283.

1,2-Dihydro-3-(4-methoxyphenyl)-7-nitro-1-(nitromethyl)-2-p-tolylisoquinoline (2k)

146 mg, 68% yield, orange solid, mp 64-66°C. 1H NMR (400 MHz, CDCl3) δ ppm 8.18 (d, J=8.6, 2.2 Hz, 1H), 8.03 (d, J=3.2 Hz, 1H), 7.48-7.45 (m, 2H), 7.40-7.37 (m, 1H), 6.98-6.95 (m, 1H), 6.92-6.90 (d, J=8.2 Hz, 2H), 6.81-6.75 (m, 4H), 6.66 (s, 1H), 5.75 (dd, J=10.5, 4.12 Hz, 1H), 4.82 (t, J=11.2 Hz, 1H), 4.40 (dd, J=11.4, 4.16 Hz, 1H), 3.77 (s, 3H), 2.18 (s, 3H); 13C NMR (100 MHz, CDCl3) δ ppm 160.7, 145.8, 145.5, 145.3, 138.7, 129.6,
129.2, 127.9, 125.8, 124.7, 124.5, 124.3, 123.8, 121.6, 114.1, 108.2, 75.9, 64.0, 55.2 29.7; HRMS (ESI) m/z calculated for C$_{24}$H$_{22}$N$_3$O$_5$ (M+H)$^+$, calc. 432.1559, found 432.1450.

1,2-Dihydro-7-methoxy-3-(4-methoxyphenyl)-1-(nitromethyl)-2-phenylisoquinoline (2l)

116, 58% yield, yellow solid, mp 77-78°C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 7.45-7.43 (m, 2H), 7.22 (d, $J$=8.56 Hz, 1H), 7.11-7.07 (m, 2H), 6.90-6.85 (m, 3H), 6.79-6.76 (m, 2H), 6.6-6.65 (m, 2H), 5.66 (dd, $J$=11.0, 4.28 Hz, 1H), 4.78 (t, $J$=11.6 Hz, 1H), 4.37 (dd, $J$=11.6, 4.28 Hz, 1H), 3.77 (s, 3H), 3.75 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ ppm 159.7, 159.0, 146.4, 138.2, 129.1, 128.8, 128.0, 126.1, 125.6, 123.1, 122.9, 114.8, 113.9, 110.9, 110.3, 76.6, 64.4, 55.4, 55.2 HRMS (ESI) m/z calculated for C$_{24}$H$_{22}$N$_3$O$_5$ (M+H)$^+$, calc. 403.1657, found 403.1522.

1-(1-Nitroethyl)-2,3-diphenyl-1,2-dihydroisoquinoline (3)

115 mg, 65% yield, pale brown solid, mp 182-183°C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 7.51 (dd, $J$=7.7, 1.3 Hz, 2H), 7.32-7.32 (m, 1H), 7.26-7.18 (m, 4H), 7.08-7.02 (m, 3H), 6.91-6.88 (m, 3H), 6.81 (s, 1H), 5.23 (d, $J$=10.0 Hz, 1H), 4.92-4.88 (m, 1H), 1.40(d, $J$=6.8 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ ppm 128.7, 128.8, 128.5, 128.4, 127.6, 127.5, 126.6, 125.6, 124.7, 123.4, 123.1, 111.8, 82.7, 69.2. 17.0; HRMS (ESI) m/z calculated for C$_{24}$H$_{22}$N$_3$O$_5$ (M+H)$^+$, calc. 357.1597, found 357.1590.

2,3-Diphenylisoquinolin-2-ium (10)

135 mg, 98% yield, green solid, $^1$H NMR (400 MHz, DMSO-d$_6$) $\delta$ ppm 10.36-10.30 (m, 1H), 8.75 (bs, 1H), 8.59 (bs, 1H), 8.40-8.33 (m, 2H), 8.11 (bs, 1H), 7.63 (m, 2H), 7.48 (m, 3H), 7.21-7.36 (m, 5H); $^{13}$C NMR (100 MHz, DMSO) $\delta$ ppm 150.4, 142.6, 138.9, 135.4, 135.2, 129.4, 128.8, 128.0, 127.7, 127.4, 126.9, 126.6, 125.6, 124.5, 124.1, 123.5; HRMS (ESI) m/z calculated for C$_{21}$H$_{18}$N$_3$+ (M+H)$^+$, calc. 282.1277, found 282.1050.
Typical experimental procedure: Synthesis of 14a-l:

To a solution of the corresponding 2-(1-alkynyl)benzaldehyde (1.08 mmol), aniline (1.08 mmol and ketone (5.38 mmol) in EtOH (5 mL) were added CoCl\(_2\) (0.324 mmol, 30 mol%) and L-proline (0.108 mmol, 10 mol%) and the mixture was stirred at 60°C under a nitrogen atmosphere for 4-16 h. After completion of the reaction, the resulting mixture was concentrated under reduced pressure, quenched with water (30 mL), extracted with EtOAc (2×30 mL) and dried over Na\(_2\)SO\(_4\) (anhydrous). The solvent was evaporated and the reaction mixture was subjected to column chromatography on silica gel using ethyl acetate/hexanes as the eluent.

1-(7-Fluoro-3-(4-methoxyphenyl)-2-(p-tolyl)-1,2-dihydroisoquinolin-1-yl)propan-2-one (14a)

380 mg, 88% yield, yellow solid, mp 122-123.8°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.41 (d, \(J=8.8\) Hz, 2H), 7.19-7.15 (m, 1H), 6.93-6.86 (m, 5H), 6.78-6.73 (m, 3H), 6.62 (s, 1H), 5.44-5.40 (m, 1H), 3.74 (s, 3H), 3.21 (dd, \(J=16.8, 8.8\) Hz, 1H), 2.53 (dd, \(J=16.8, 5.12\) Hz, 1H), 2.15 (s, 3H), 2.12 (s, 3H); \(^{13}\)C NMR (100MHz, CDCl\(_3\)) \(\delta\) 207.0, 162.7, 160.3, 159.5, 144.6, 139.9, 133.6, 131.8, 129.7, 129.2, 128.1, 125.6, 122.4, 114.3, 114.1, 113.8, 112.4, 112.2, 109.6, 60.8, 55.1, 46.9, 31.7, 20.5; HRMS (ESI) \(m/z\) calculated for C\(_{26}\)H\(_{24}\)FNO\(_2\) (M+H)+, calcd 402.1869, found 402.1940.

1-(7-Methoxy-3-(4-methoxyphenyl)-2-(p-tolyl)-1,2-dihydroisoquinolin-1-yl)propan-2-one (14b)

397 mg, 89% yield, yellow solid, mp 153.6-156.2°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.43 (d, \(J=8.8\) Hz, 2H), 7.16 (d, \(J=8.8\) Hz, 1H), 6.95-6.87 (m, 4H), 6.80-6.76 (m, 3H), 6.65 (s, 1H), 6.59 (d, \(J=2.2\) Hz, 1H), 5.44-5.41 (m, 1H), 3.75 (s, 3H), 3.74 (s, 3H), 3.24 (dd, \(J=16.8, 9.5\) Hz, 1H), 2.52 (dd, \(J=16.8, 4.4\) Hz, 1H), 2.16 (s, 3H), 2.13 (s, 3H); \(^{13}\)C NMR (400 MHz, CDCl\(_3\)) \(\delta\) 207.6, 159.2, 158.6, 144.9, 138.2, 133.5, 131.4, 130.1, 129.2, 128.0, 125.5, 125.1, 122.2, 113.8, 113.4, 110.5, 110.4, 61.2, 55.2, 55.1, 47.2, 31.9, 20.6; HRMS (ESI) \(m/z\) calculated for C\(_{27}\)H\(_{26}\)NO\(_3\) (M+H)+, calcd 414.2069, found 414.2077.

1-(2-(4-Hydroxyphenyl)-3-(4-methoxyphenyl)-7-nitro-1,2-dihydroisoquinolin-1-yl)propan-2-one (14c)

295 mg, 64% yield, yellow solid, mp 109-112.5°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 8.09 (d, \(J=8.1\) Hz, 1H), 7.93 (s, 1H), 7.42 (d, \(J=8.8\) Hz, 2H), 7.31 (d, \(J=8.8\) Hz, 1H), 6.93 (d, \(J=8.8\) Hz, 2H), 6.79 (d, \(J=8.8\) Hz, 2H), 6.59-6.57 (m, 3H), 5.49-5.46 (m, 1H), 4.74 (b, 1H), 3.77 (s, 3H), 3.28 (dd, \(J=16.8, 8.8\) Hz, 1H), 2.58 (dd, \(J=16.8, 4.4\) Hz, 1H), 2.17 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 207.3, 160.3, 152.2, 145.7, 145.4, 139.8, 138.6, 130.6, 129.3, 128.8, 125.2, 123.9, 123.1, 121.2, 115.6, 114.0, 107.7, 61.3, 55.2, 46.9, 31.7, 31.5; HRMS (ESI) \(m/z\) calculated for C\(_{25}\)H\(_{22}\)N\(_2\)O\(_3\) (M+H)+, calcd 431.1607, found 431.1604.

1-(2-(4-Chlorophenyl)-7-methoxy-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)propan -2-one (14d)

365 mg, 78% yield, brown solid, mp 70.8-73°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.39 (d, \(J=8.8\) Hz, 2H), 7.18 (d, \(J=8.8\) Hz, 1H), 7.09 (dd, \(J=8.8, 2.2\) Hz, 1H), 7.02 (m, 3H), 6.80-6.78 (m, 3H), 6.69 (s, 1H), 6.60-6.58 (m, 2H), 5.44-5.40 (m, 1H), 3.76 (s, 3H), 3.756 (s, 3H), 3.27 (dd, \(J=16.8,9.5\) Hz, 1H), 2.49 (dd, \(J=16.8, 4.4\) Hz, 1H), 2.13 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 207.4, 159.4, 158.8, 145.9, 137.5, 133.7, 129.5, 129.0, 128.6, 127.9, 126.9, 125.8,
124.8, 123.3, 116.2, 114.0, 113.5, 60.9, 55.3, 55.2, 46.9, 31.8; HRMS (ESI) m/z calculated for C_{26}H_{25}ClNO_{3} (M+H)^+, calcd 434.1523, found 434.1532.

1-(2-(4-Fluorophenyl)-3-(4-methoxyphenyl)-7-nitro-1,2-dihydroisoquinolin-1-yl)propan-2-one (14e)

367 mg, 79% yield, yellow solid, mp 83-84.9°C; ^1H NMR (400 MHz, CDCl$_3$) δ ppm 8.11 (dd, J=8.7, 2.3 Hz, 1H), 7.94 (s, 1H), 7.41 (d, J=9.16 Hz, 2H), 7.33 (d, J=8.0 Hz, 1H), 7.06-7.03 (m, 2H), 6.82-6.77 (m, 4H), 6.66 (s, 1H), 5.52 (dd, J=9.1, 4.5 Hz, 1H), 3.77 (s, 3H), 3.31 (dd, J=17.4, 9.1 Hz, 1H), 2.56 (dd, J=17.4, 4.5 Hz, 1H), 2.17 (s, 3H); ^13C NMR (100 MHz, CDCl$_3$) δ ppm 206.4, 160.5, 160.3, 145.7, 145.2, 142.6, 138.3, 131.0, 129.2, 128.5, 125.0, 124.9, 124.1, 123.2, 121.0, 115.7, 115.4, 114.1, 108.6, 61.0, 55.3, 46.7, 31.6; HRMS (ESI) m/z calculated for C_{25}H_{21}NO_{18} (M+H)^+, calcd 432.1485, found 432.1861.

4-(7-Fluoro-3-(4-methoxyphenyl)-1-(2-oxopropyl)isoquinolin-2(1H)-yl)benzonitrile (14f)

300 mg, 68% yield, light yellow solid, mp 79.7-82.8°C; ^1H NMR (400 MHz, CDCl$_3$) δ ppm 7.36-7.32 (m, 4H), 7.26-7.21 (m, 1H), 7.13 (d, J=8.8 Hz, 2H), 6.97-6.92 (m, 1H), 6.82-6.79 (m, 3H), 6.75 (s, 1H), 5.59-5.55 (m, 1H), 3.78 (s, 3H), 3.29 (dd, J=17.6, 9.5 Hz, 1H), 2.55 (dd, J=17.5, 4.4 Hz, 1H), 2.14 (s, 3H); ^13C NMR (100 MHz, CDCl$_3$) δ ppm 206.6, 163.0, 160.6, 159.9, 150.4, 138.0, 134.6, 132.8, 128.6, 127.8, 127.6, 126.4, 121.4, 119.4, 119.4, 114.3, 112.4, 112.3, 112.1, 104.0, 59.1, 55.3, 46.3, 31.4; HRMS (ESI) m/z calculated for C_{26}H_{21}FNO_{2} (M+H)^+, calcd 413.1665, found 413.1628.

1-(7-Bromo-2-(4-iodophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)propan-2-one (14g)

509 mg, 82% yield, brown solid, mp 113.3-115.5°C; ^1H NMR (400 MHz, CDCl$_3$) δ ppm 7.39-7.33 (m, 5H), 7.18 (bs, 1H), 7.11 (d, J=8.04 Hz, 1H), 6.86-6.78 (m, 4H), 6.64 (s, 1H), 5.45-5.41 (m, 1H), 3.77 (s, 3H), 3.25 (dd, J=16.8, 9.52 Hz, 1H), 2.47 (dd, J=16.8, 4.4 Hz, 1H), 2.13 (s, 3H); ^13C NMR (100 MHz, CDCl$_3$) δ ppm 206.8, 159.8, 146.6, 146.0, 140.1, 137.8, 137.5, 133.6, 130.6, 130.5, 128.9, 128.3, 128.0, 125.9, 124.5, 119.7, 117.2, 114.1, 110.6, 85.7, 79.3, 60.1, 55.2, 46.6, 31.6; HRMS (ESI) m/z calculated for C_{25}H_{21}BrINO_{2} (M+H)^+, calcd 573.9878, found 574.0090.

1-(7-Bromo-2-(4-fluorophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)propan-2-one (14h)

325 mg, 81% yield, brown solid, mp 107.5-109.4°C; ^1H NMR (400 MHz, CDCl$_3$) δ ppm 7.40 (d, J=8.8 Hz, 2H), 7.34 (d, J=8.8 Hz, 1H), 7.17-7.16 (m, 1H), 7.11 (d, J=8.8 Hz, 1H), 7.04-7.01 (m, 2H), 6.80-6.75 (m, 4H), 6.63 (s, 1H), 5.40-5.36 (m, 1H), 3.76 (s, 3H), 3.26 (dd, J=17.6, 9.52 Hz, 1H), 2.46 (dd, J=16.8, 4.4 Hz, 1H), 2.15 (s, 1H); ^13C NMR (100 MHz, CDCl$_3$) δ ppm 207.0, 159.8, 141.0, 133.2, 130.8, 130.5, 129.2, 128.5, 128.0, 125.7, 124.3, 124.2, 119.6, 115.4, 115.2, 114.0, 109.8, 60.8, 55.2, 46.9, 31.7; HRMS (ESI) m/z calculated for C_{25}H_{21}BrINO_{2} (M+H)^+, calcd 466.0818, found 466.0823.
1-(2-(2-Chloro-4-fluorophenyl)-7-fluoro-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-3-yl)propan-2-one (14i)

365 mg, 77% yield, white solid, mp 176.4-178.9°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.51 (d, \(J=8.24\) Hz, 2H), 7.23-7.20 (m, 1H), 7.11 (d, \(J=7.32\) Hz, 1H), 6.97-6.89 (m, 2H), 6.81 (d, \(J=7.8\) Hz, 2H), 6.67 (s, 1H), 6.56-6.54 (m, 2H), 5.07-5.04 (m, 1H), 3.77 (s, 3H), 3.26-3.10 (m, 2H), 2.04 (s, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 206.2, 162.9, 160.6, 160.5, 160.1, 158.1, 141.3, 140.3, 133.5, 129.0, 128.4, 128.0, 125.8, 117.8, 117.5, 114.7, 114.5, 114.3, 114.0, 110.1, 58.6, 15.3, 49.3, 31.0; HRMS (ESI) \(m/z\) calculated for C\(_{25}\)H\(_{20}\)ClF\(_2\)NO\(_2\) (M+H\(^+\)), calcd 440.1229, found 440.1348.

1-(7-Fluoro-3-(4-methoxyphenyl)-2-(p-tolyl)-1,2-dihydroisoquinolin-3-yl)-3-hydroxybutan-2-one (14j)

Diastereomeric mixture, 238 mg, 51% yield, brown solid, in 1:1 isomeric form; mp 138.2-139.6°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.42 (d, \(J=8.8\) Hz, 1H, first isomer), 7.38 (d, \(J=8.8\) Hz, 1H, second isomer), 7.23-7.18 [m, 1H; (0.5H, first isomer) (0.5H, second isomer)], 6.96-6.89 [m, 5H; (2.5H, first isomer) (2.5H, second isomer)], 6.80-6.71 [m, 3H; (1.5H, first isomer) (1.5H, second isomer)], 6.67 (s, 0.5H, first isomer), 6.64 (s, 0.5H, second isomer), 5.53-5.49 [m, 1H; (0.5H, first isomer) (0.5H, second isomer)], 4.19-4.15 (m, 0.5H, first isomer), 4.08-4.05 (m, 0.5H, second isomer), 3.75 [s, 3H, (1.5H, first isomer) (1.5H, second isomer)], 3.63-3.61 [m, 1H; (0.5H, first isomer) (0.5H, second isomer)], 3.49-3.48 [m, 1H; (0.5H, first isomer) (0.5H, second isomer)], 3.34-3.21 [m, 1H; (0.5H, first isomer) (0.5H, second isomer)], 2.63-2.52 [m, 1H; (0.5H, first isomer) (0.5H, second isomer)], 2.18 [s, 3H; (1.5H, first isomer) (1.5H, second isomer)], 1.29 (d, \(J=7.3\) Hz, 1.5H, first isomer), 1.19 (d, \(J=7.3\) Hz, 1.5H, second isomer); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 211.1, 210.6, 162.8, 162.7, 160.4, 160.3, 159.6, 159.5, 144.5, 144.4, 140.0, 139.8, 132.9, 132.2, 132.1, 129.3, 128.4, 128.2, 125.7, 125.6, 122.6, 122.4, 114.64, 114.6, 114.4, 114, 113.7, 112.4, 112.3, 112.2, 112.1, 109.7, 109.3, 73.9, 73.0, 61.3, 60.4, 55.1, 41.7, 41.3, 20.6, 19.8, 18.7; HRMS (ESI) \(m/z\) calculated for C\(_{25}\)H\(_{20}\)F\(_2\)NO\(_3\) (M+H\(^+\)), calcd 432.1975, found 432.1967.

1-(7-Fluoro-3-(4-methoxyphenyl)-2-(p-tolyl)-1,2-dihydroisoquinolin-3-yl)-3-hydroxypropan-2-one (14k)

252 mg, 56% yield, brown solid, mp 88.7-90.9°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.39 (d, \(J=8.8\) Hz, 2H), 7.23-7.19 (m, 1H), 6.96-6.86 (m, 5H), 6.80 (d, \(J=8.8\) Hz, 3H), 6.76-6.73 (m, 1H), 6.66 (s, 1H), 5.51-5.48 (m, 1H), 4.21-4.05 (m, 2H), 3.75 (s, 3H), 3.18 (dd, \(J=11.7, 9.5\) Hz, 2H), 2.50 (dd, \(J=16.1, 5.1\) Hz, 1H), 2.17 (s, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 208.3, 162.8, 160.4, 159.6, 144.4, 144.3, 139.7, 132.7, 132.3, 129.4, 128.2, 125.84, 125.77, 122.5, 114.5, 114.0, 112.1, 109.6, 69.7, 61.3, 55.2, 42.1, 20.6; HRMS (ESI) \(m/z\) calculated for C\(_{26}\)H\(_{24}\)F\(_2\)NO\(_3\) (M+H\(^+\)), calcd 418.1838, found 418.1821.
$^{1}$H and $^{13}$C Spectra

$^{1}$H NMR

1,2-Dihydro-1-(nitromethyl)-2,3-diphenylisoquinoline (2a)
$^{13}$C NMR

1,2-Dihydro-1-(nitromethyl)-2,3-diphenylisoquinoline (2a)
2-(4-Fluorophenyl)-1,2-dihydro-1-(nitromethyl)-3-phenylisoquinoline (2b)
$^{13}$C NMR

2-(4-Fluorophenyl)-1,2-dihydro-1-(nitromethyl)-3-phenylisoquinoline (2b)
$^1$H NMR

1,2-Dihydro-1-(nitromethyl)-3-phenyl-2-p-tolylisoquinoline (2c)
\[ ^{13}C \text{ NMR} \]

1,2-Dihydro-1-(nitromethyl)-3-phenyl-2-p-tolylisoquinoline (2c)
$^1$H NMR

1,2-Dihydro-1-(nitromethyl)-2-phenyl-3-p-tolylisoquinoline (2d)

![NMR spectrum of 1,2-Dihydro-1-(nitromethyl)-2-phenyl-3-p-tolylisoquinoline (2d)]
$^{13}$C NMR

1,2-Dihydro-1-(nitromethyl)-2-phenyl-3-p-tolyisoquinoline (2d)
\[ ^1 \text{H NMR} \]

2-(4-Fluorophenyl)-1,2-dihydro-1-(nitromethyl)-3-p-tolylisoquinoline (2e)
2-(4-Fluorophenyl)-1,2-dihydro-1-(nitromethyl)-3-p-tolylisoquinoline (2e)
7-Fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)-2-phenylisoquinoline (2f)

\( ^1H \text{NMR} \)

S22
$^{13}$C NMR

7-Fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)-2-phenyisoquinoline (2f)
$^1$H NMR

2-(4-Chlorophenyl)-7-fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)isoquinoline (2g)
$^{13}$C NMR

2-(4-Chlorophenyl)-7-fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)isoquinoline (2g)
$^1$H NMR

7-Fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)-2-p-tolylisoquinoline (2h)
$^{13}$C NMR

7-Fluoro-1,2-dihydro-3-(4-methoxyphenyl)-1-(nitromethyl)-2-$p$-tolylisoquinoline (2h)
$\text{H NMR}$

$2-(4\text{-Chlorophenyl})-1,2\text{-dihydro-7-nitro-1-(nitromethyl)-3-phenylisoquinoline (2i)}$
$^{13}$C NMR

2-(4-Chlorophenyl)-1,2-dihydro-7-nitro-1-(nitromethyl)-3-phenylisoquinoline (2i)

$^1$H NMR
1,2-Dihydro-3-(4-methoxyphenyl)-7-nitro-1-(nitromethyl)-2-phenylisoquinoline (2j)

$\text{^{13}C NMR}$
1,2-Dihydro-3-(4-methoxyphenyl)-7-nitro-1-(nitromethyl)-2-phenylisoquinoline (2j)
1,2-Dihydro-3-(4-methoxyphenyl)-7-nitro-1-(nitromethyl)-2-p-tolyisoquinoline (2k)

\[ \text{\textsuperscript{1}H NMR} \]

\begin{align*}
\text{O}_2\text{N} & \quad \text{O}_2\text{N} \\
\text{N} & \quad \text{O} \\
\text{Me} & \quad \text{1,2-Dihydro-3-(4-methoxyphenyl)-7-nitro-1-(nitromethyl)-2-p-tolyisoquinoline (2k)}
\end{align*}
$^{13}$C NMR

1,2-Dihydro-3-(4-methoxyphenyl)-7-nitro-1-(nitromethyl)-2-p-tolylisoquinoline (2k)
1,2-Dihydro-7-methoxy-3-(4-methoxyphenyl)-1-(nitromethyl)-2-phenylisoquinoline (2l)
1,2-Dihydro-7-methoxy-3-(4-methoxyphenyl)-1-(nitromethyl)-2-phenylisoquinoline (2l)
$^{1}$H NMR

1-(1-Nitroethyl)-2,3-diphenyl-1,2-dihydrisoquinoline (3)
$^{13}$C NMR

1-(1-Nitroethyl)-2,3-diphenyl-1,2-dihydroisoquinoline (3)
$^1$H NMR

2,3-Diphenylisoquinolin-2-ium (10)
$^{13}\text{C NMR}$

2,3-Diphenylisoquinolin-2-ium (10)
$^1$H NMR

1-(7-Fluoro-3-(4-methoxyphenyl)-2-p-tolyl-1,2-dihydroisoquinolin-1-yl)propan-2-one (14a)
$^{13}$C NMR

1-(7-Fluoro-3-(4-methoxyphenyl)-2-p-tolyl-1,2-dihydroisoquinolin-1-yl)propan-2-one (14a)
$^1$H NMR

1-(7-Methoxy-3-(4-methoxyphenyl)-2-p-tolyl-1,2-dihydroisoquinolin-1-yl)propan-2-one (14b)
13C NMR

1-(7-Methoxy-3-(4-methoxyphenyl)-2-p-tolyl-1,2-dihydroisoquinolin-1-yl)propan-2-one (14b)
1-(2-(4-Hydroxyphenyl)-3-(4-methoxyphenyl)-7-nitro-1,2-dihydroisoquinolin-1-yl)propan-2-one (14c)
$^{13}$C NMR

1-(2-(4-Hydroxyphenyl)-3-(4-methoxyphenyl)-7-nitro-1,2-dihydroisoquinolin-1-yl)propan-2-one (14c)
$\text{H NMR}$

1-(2-(4-Chlorophenyl)-7-methoxy-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl) propan-2-one (14d)
$^{13}$C NMR

1-(2-(4-Chlorophenyl)-7-methoxy-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl) propan-2-one (14d)
$^1$H NMR

1-(2-(4-Fluorophenyl)-3-(4-methoxyphenyl)-7-nitro-1,2-dihydroisoquinolin-1-yl)propan-2-one (14e)
$^{13}$C NMR

1-(2-(4-Fluorophenyl)-3-(4-methoxyphenyl)-7-nitro-1,2-dihydroisoquinolin-1-yl)propan-2-one (14e)
$^1$H NMR

4-(7-Fluoro-3-(4-methoxyphenyl)-1-(2-oxopropyl)isoquinolin-2(1H)-yl)benzonitrile (14f)
4-(7-Fluoro-3-(4-methoxyphenyl)-1-(2-oxopropyl)isoquinolin-2(1H)-yl)benzonitrile (14f)
1-(7-Bromo-2-(4-iodophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)propan-2-one (14g)
1-(7-Bromo-2-(4-iodophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)propan-2-one (14g)
1-(7-Bromo-2-(4-fluorophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)propan-2-one (14h)
$^{13}$C NMR

1-(7-Bromo-2-(4-fluorophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)propan-2-one (14h)
$^1$H NMR

1-(2-(2-Chloro-4-fluorophenyl)-7-fluoro-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl) propan-2-one (14i)
$^{13}$C NMR

1-(2-(2-Chloro-4-fluorophenyl)-7-fluoro-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl) propan-2-one (14i)
$^1$H NMR

1-(7-Fluoro-2-(4-fluorophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)-3-hydroxybutan-2-one (14j)
$^{13}$C NMR

1-(7-Fluoro-2-(4-fluorophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)-3-hydroxybutan-2-one (14j)
\(^1\)H NMR

1-(7-Fluoro-3-(4-methoxyphenyl)-2-p-tolyl-1,2-dihydroisoquinolin-1-yl)-3-hydroxypropan-2-one (14k)
$^{13}$C NMR

1-(7-Fluoro-3-(4-methoxyphenyl)-2-p-tolyl-1,2-dihydroisoquinolin-1-yl)-3-hydroxypropan-2-one (14k)