

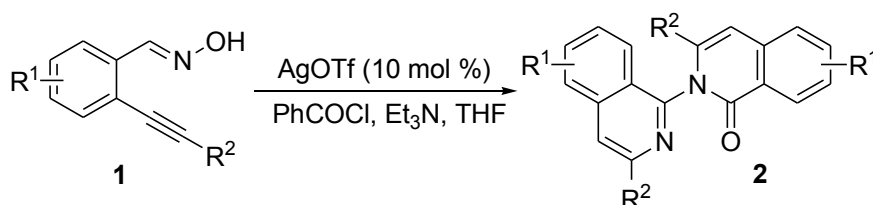
Supporting Information

1. General experimental methods (S2)
2. General experimental procedure and characterization data (S2-S9)
3. ^1H and ^{13}C NMR spectra of compound **2** (S10-S37)

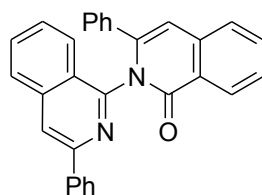
General experimental methods:

Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 μm, standard grade). Analytical thin-layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 25–35 °C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^1H and ^{13}C NMR spectra were recorded in CDCl_3 on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

General experimental procedure for the silver triflate-catalyzed reaction of 2-alkynylbenzaldoxime 1 in the presence of benzoyl chloride

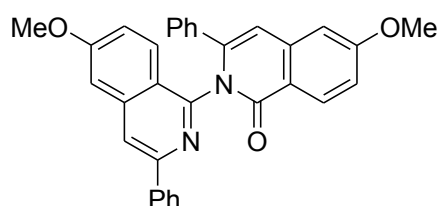


Silver triflate (0.04 mmol, 10.3 mg) was added to a solution of 2-alkynylbenzaldoxime **1** (0.4 mmol) in THF (2.0 mL). The solution was stirred at 60 °C under N_2 for 1 hour. Subsequently, Et_3N (0.6 mmol, 61 mg) and benzoyl chloride (0.6 mmol, 85 mg) in THF (1.0 mL) were added. The reaction was stirred at room temperature under air. After completion of the reaction as indicated by TLC (10 min), the solvent was evaporated and the residue was purified by flash column chromatograph on silica gel (eluting with $\text{PE/EA} = 4/1$) to afford the product **2**.



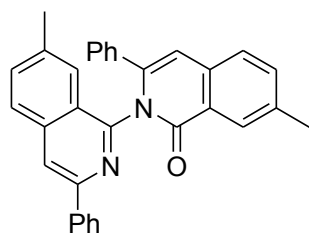
3-Phenyl-2-(3-phenylisoquinolin-1-yl)isoquinolin-1(2*H*)-one **2a**

^1H NMR (400 MHz, CDCl_3): δ 8.48 (d, $J = 8.0$ Hz, 1H), 7.87-7.80 (m, 4H), 7.75 (d, $J = 8.0$ Hz, 1H), 7.69 (t, $J = 7.2$ Hz, 1H), 7.61-7.54 (m, 2H), 7.52-7.47 (m, 2H), 7.39 (t, $J = 7.2$ Hz, 2H), 7.32 (d, $J = 7.2$ Hz, 1H), 7.20-7.18 (m, 2H), 6.97-6.95 (m, 3H), 6.69 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 163.2, 151.1, 149.8, 143.7, 138.6, 138.3, 137.1, 135.6, 133.0, 130.5, 129.9, 128.7, 128.5, 128.4, 128.2, 128.1, 127.5, 127.3, 127.0, 126.8, 126.2, 125.5, 125.2, 124.6, 117.4, 108.0; HRMS calcd. for $\text{C}_{30}\text{H}_{21}\text{N}_2\text{O}^+ [\text{M}+\text{H}]^+$: 425.1648, found 425.1625.



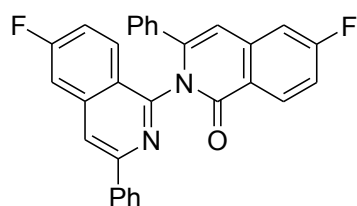
6-Methoxy-2-(6-methoxy-3-phenylisoquinolin-1-yl)-3-phenylisoquinolin-1(2*H*)-one **2b**

^1H NMR (400 MHz, CDCl_3): δ 8.47 (s, 1H), 7.88 (t, $J = 7.6$ Hz, 3H), 7.79-7.71 (m, 3H), 7.63-7.57 (m, 3H), 7.52 (t, $J = 7.6$ Hz, 1H), 7.13 (d, $J = 8.4$ Hz, 2H), 6.95 (d, $J = 8.8$ Hz, 2H), 6.67 (s, 1H), 6.49 (d, $J = 8.8$ Hz, 2H), 3.84 (s, 3H), 3.58 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 163.4, 160.1, 159.2, 151.5, 149.7, 143.6, 138.6, 137.3, 133.6, 133.0, 131.4, 130.5, 130.2, 130.1, 128.4, 128.2, 128.0, 127.7, 127.2, 126.8, 126.1, 124.6, 116.4, 114.0, 112.9, 107.7, 55.3, 55.0; HRMS calcd. for $\text{C}_{32}\text{H}_{25}\text{N}_2\text{O}_3^+ [\text{M}+\text{H}]^+$: 485.1860, found 485.1846.



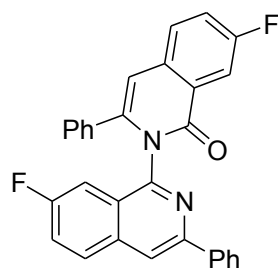
7-Methyl-2-(7-methyl-3-phenylisoquinolin-1-yl)-3-phenylisoquinolin-1(2*H*)-one **2c**

^1H NMR (400 MHz, CDCl_3): δ 8.31 (s, 1H), 7.81 (d, $J = 5.6$ Hz, 3H), 7.64 (d, $J = 8.0$ Hz, 1H), 7.57 (s, 1H), 7.53 (s, 1H), 7.37 (t, $J = 8.8$ Hz, 4H), 7.31 (d, $J = 7.2$ Hz, 1H), 7.20-7.17 (m, 2H), 6.97-6.96 (m, 3H), 6.68 (s, 1H), 2.49 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 163.2, 150.9, 149.0, 142.8, 138.7, 137.0, 136.7, 135.8, 134.8, 134.5, 132.9, 129.9, 128.7, 128.4, 128.2, 128.0, 127.8, 127.4, 127.1, 126.8, 126.1, 125.7, 125.1, 123.1, 117.3, 107.9, 22.0, 21.4; HRMS calcd. for $\text{C}_{32}\text{H}_{25}\text{N}_2\text{O}^+$ $[\text{M}+\text{H}]^+$: 453.1961, found 453.1926.



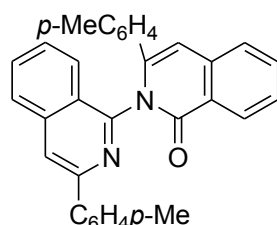
6-Fluoro-2-(6-fluoro-3-phenylisoquinolin-1-yl)-3-phenylisoquinolin-1(2*H*)-one **2d**

^1H NMR (400 MHz, CDCl_3): δ 8.49-8.46 (m, 1H), 7.85-7.80 (m, 4H), 7.39-7.32 (m, 5H), 7.28-7.16 (m, 4H), 6.98 (d, $J = 6.4$ Hz, 3H), 6.63 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 165.7 (d, $^1J_{\text{CF}} = 251.4$ Hz), 163.2 (d, $^1J_{\text{CF}} = 252.9$ Hz), 162.5, 151.2, 150.9, 144.9, 138.0, 135.1, 133.3, 131.5 (d, $^3J_{\text{CF}} = 10.0$ Hz), 129.9, 128.8, 128.5, 128.4, 128.2, 127.8 (d, $^3J_{\text{CF}} = 9.7$ Hz), 127.6, 126.9, 122.6, 121.7, 118.6 (d, $^2J_{\text{CF}} = 25.8$ Hz), 117.0, 115.7 (d, $^2J_{\text{CF}} = 23.4$ Hz), 111.1 (d, $^2J_{\text{CF}} = 21.9$ Hz), 110.6 (d, $^2J_{\text{CF}} = 21.2$ Hz), 107.4; HRMS calcd. for $\text{C}_{30}\text{H}_{19}\text{F}_2\text{N}_2\text{O}^+$ $[\text{M}+\text{H}]^+$: 461.1460, found 461.1440.



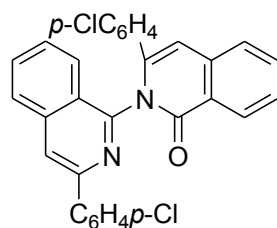
7-Fluoro-2-(7-fluoro-3-phenylisoquinolin-1-yl)-3-phenylisoquinolin-1(2*H*)-one **2e**

^1H NMR (400 MHz, CDCl_3): δ 8.14-8.11 (m, 1H), 7.87-7.77 (m, 4H), 7.65-7.61 (m, 1H), 7.45-7.32 (m, 6H), 7.20-7.18 (m, 2H), 7.01-6.99 (m, 3H), 6.70 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 162.4, 161.6 (d, $^1J_{\text{CF}} = 248.0$ Hz), 161.4 (d, $^1J_{\text{CF}} = 250.9$ Hz), 150.9, 150.8, 149.7, 142.8, 138.2, 135.3 (d, $^2J_{\text{CF}} = 28.5$ Hz), 133.6 (d, $^2J_{\text{CF}} = 23.0$ Hz), 130.2, 130.1, 130.0, 128.8, 128.6, 128.3, 127.6, 126.8, 122.1, 121.9, 121.7, 121.4, 117.2, 113.4 (d, $^2J_{\text{CF}} = 22.9$ Hz), 108.1 (d, $^2J_{\text{CF}} = 22.2$ Hz), 107.5; HRMS calcd. for $\text{C}_{30}\text{H}_{19}\text{F}_2\text{N}_2\text{O}^+ [\text{M}+\text{H}]^+$: 461.1460, found 461.1425.



3-*p*-Tolyl-2-(3-*p*-tolylisoquinolin-1-yl)isoquinolin-1(2*H*)-one **2f**

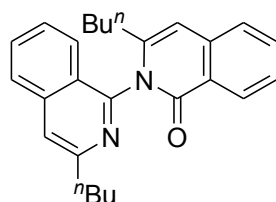
^1H NMR (400 MHz, CDCl_3): δ 8.47 (d, $J = 8.0$ Hz, 1H), 7.91 (s, 1H), 7.82-7.78 (m, 4H), 7.75-7.71 (m, 1H), 7.64-7.59 (m, 2H), 7.55-7.50 (m, 2H), 7.23 (d, $J = 8.0$ Hz, 2H), 7.10 (d, $J = 7.6$ Hz, 2H), 6.78 (d, $J = 8.0$ Hz, 2H), 6.68 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 163.4, 151.6, 145.0, 143.9, 138.5, 138.4, 138.0, 137.3, 136.0, 133.0, 132.8, 130.5, 130.1, 129.3, 128.7, 128.4, 128.2, 127.9, 127.3, 126.8, 126.1, 125.4, 125.3, 124.7, 117.0, 107.8, 21.2, 21.0; HRMS calcd. for $\text{C}_{32}\text{H}_{25}\text{N}_2\text{O}^+ [\text{M}+\text{H}]^+$: 453.1961, found 453.1964.



3-(4-Chlorophenyl)-2-(3-(4-chlorophenyl)isoquinolin-1-yl)isoquinolin-1(2*H*)-one **2g**

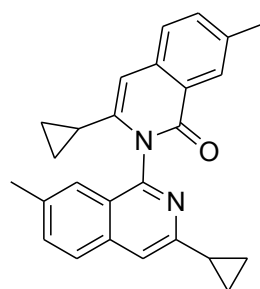
^1H NMR (400 MHz, CDCl_3): δ 8.48 (d, $J = 8.0$ Hz, 1H), 7.96 (s, 1H), 7.88-7.84 (m, 3H), 7.81-7.75 (m, 2H), 7.68-7.60 (m, 4H), 7.40 (d, $J = 8.4$ Hz, 2H), 7.13 (d, $J = 8.0$ Hz, 2H), 6.96 (d, $J = 8.0$ Hz, 2H), 6.69 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): 71.7,

163.3, 151.5, 148.7, 142.5, 138.5, 137.0, 136.9, 134.8, 133.7, 133.3, 131.0, 130.2, 130.1, 128.9, 128.5, 128.1, 127.9, 127.6, 127.4, 126.3, 125.6, 125.4, 124.6, 117.6, 108.4; HRMS calcd. for $C_{30}H_{19}Cl_2N_2O^+ [M+H]^+$: 493.0869, found 493.0837.



3-Butyl-2-(3-butylisoquinolin-1-yl)isoquinolin-1(2*H*)-one **2h**

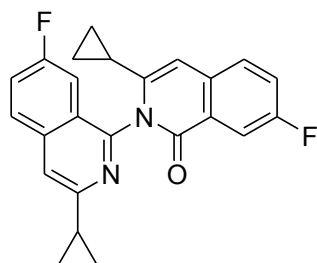
1H NMR (400 MHz, $CDCl_3$): δ 8.40 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 8.4 Hz, 1H), 7.68-7.61 (m, 3H), 7.59-7.53 (m, 2H), 7.47-7.41 (m, 2H), 6.53 (s, 1H), 3.07-2.91 (m, 2H), 2.56-2.18 (m, 1H), 2.12-2.04 (m, 1H), 1.84-1.77 (m, 2H), 1.51-1.34 (m, 4H), 1.15-1.06 (m, 2H), 0.95 (t, J = 7.6 Hz, 3H), 0.68 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.6, 154.9, 150.8, 143.3, 138.6, 137.5, 133.4, 132.8, 130.7, 130.0, 128.3, 128.0, 126.7, 126.1, 125.5, 124.1, 119.7, 104.8, 37.4, 32.2, 32.0, 29.8, 22.4, 22.0, 13.9, 13.5; HRMS calcd. for $C_{26}H_{29}N_2O^+ [M+H]^+$: 385.2274, found 385.2262.



3-Cyclopropyl-2-(3-cyclopropyl-7-methylisoquinolin-1-yl)-7-methylisoquinolin-1(2*H*)-one **2i**

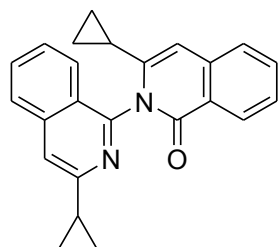
1H NMR (400 MHz, $CDCl_3$): δ 8.22 (s, 1H), 7.70 (d, J = 8.4 Hz, 1H), 7.60 (t, J = 7.6 Hz, 1H), 7.54 (s, 1H), 7.49-7.48 (m, 2H), 7.27 (s, 1H), 6.40 (s, 1H), 2.48 (s, 3H), 2.37 (s, 3H), 2.22-2.16 (m, 1H), 1.28-1.20 (m, 2H), 1.13-1.10 (m, 1H), 1.04-0.97 (m, 2H), 0.79-0.72 (m, 1H), 0.57-0.50 (m, 1H), 0.38-0.32 (m, 1H), 0.20-0.13 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.4, 154.6, 150.8, 143.6, 136.3, 135.2, 134.2, 133.5,

132.9, 130.1, 129.6, 128.4, 127.8, 126.2, 125.6, 122.8, 117.5, 103.5, 21.9, 21.4, 16.8, 13.7, 9.3, 9.3, 7.1, 6.8; HRMS calcd. for $C_{26}H_{25}N_2O^+$ $[M+H]^+$: 381.1961, found 381.1942.



3-Cyclopropyl-2-(3-cyclopropyl-7-fluoroisoquinolin-1-yl)-7-fluoroisoquinolin-1(2H)-one **2j**

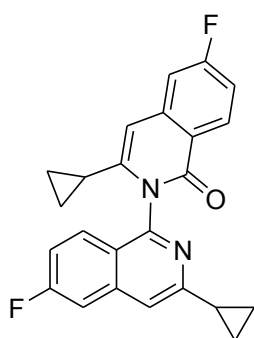
1H NMR (400 MHz, $CDCl_3$): δ 8.07-8.04 (s, 1H), 7.82-7.78 (m, 1H), 7.58 (t, $J = 7.2$ Hz, 2H), 7.53-7.50 (m, 1H), 7.39 (d, $J = 8.0$ Hz, 1H), 7.13 (d, $J = 7.6$ Hz, 1H), 6.42 (s, 1H), 2.22-2.16 (m, 1H), 1.29-1.22 (m, 2H), 1.15-1.11 (m, 1H), 1.04-1.00 (m, 2H), 0.81-0.75 (m, 1H), 0.61-0.54 (m, 1H), 0.37-0.31 (m, 1H), 0.21-0.14 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.6, 161.1 (d, $^1J_{CF} = 247.0$ Hz), 160.8 (d, $^1J_{CF} = 249.3$ Hz), 155.2, 143.5, 135.5, 134.0, 133.5, 130.0, 129.1 (d, $^3J_{CF} = 8.3$ Hz), 128.3, 128.1 (d, $^3J_{CF} = 7.3$ Hz), 126.2 (d, $^3J_{CF} = 8.3$ Hz), 124.8 (d, $^3J_{CF} = 7.8$ Hz), 117.7, 113.0 (d, $^2J_{CF} = 22.8$ Hz), 107.6 (d, $^2J_{CF} = 22.0$ Hz), 103.3, 16.9, 13.8, 9.8, 9.7, 7.2, 7.1; HRMS calcd. for $C_{24}H_{19}F_2N_2O^+$ $[M+H]^+$: 389.1460, found 389.1443.



3-Cyclopropyl-2-(3-cyclopropylisoquinolin-1-yl)isoquinolin-1(2H)-one **2k**

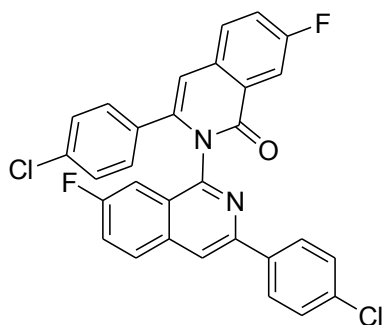
1H NMR (400 MHz, $CDCl_3$): δ 8.39 (d, $J = 8.0$ Hz, 1H), 7.80 (d, $J = 8.4$ Hz, 1H), 7.69-7.60 (m, 3H), 7.57-7.52 (m, 2H), 7.44-7.38 (m, 2H), 6.42 (s, 1H), 2.24-2.18 (m, 1H), 1.31-1.25 (m, 2H), 1.17-1.13 (m, 1H), 1.07-0.99 (m, 2H), 0.83-0.76 (m, 1H),

0.62-0.54 (m, 1H), 0.37-0.31 (m, 1H), 0.20-0.13 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 163.4, 155.5, 151.4, 144.6, 138.5, 137.5, 133.1, 132.7, 130.5, 130.0, 128.2, 127.0, 126.3, 125.7, 125.0, 124.4, 117.7, 103.5, 16.9, 13.8, 9.5, 9.5, 7.0; HRMS calcd. for $\text{C}_{24}\text{H}_{21}\text{N}_2\text{O}^+ [\text{M}+\text{H}]^+$: 353.1648, found 353.1627.



3-Cyclopropyl-2-(3-cyclopropyl-6-fluoroisoquinolin-1-yl)-6-fluoroisoquinolin-1(2H)-one **2l**

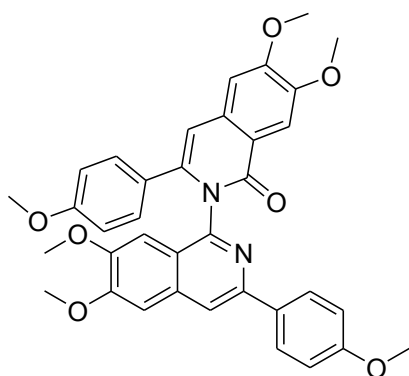
^1H NMR (400 MHz, CDCl_3): δ 8.17 (d, $J = 7.6$ Hz, 1H), 7.61-7.53 (m, 3H), 7.41 (d, $J = 7.2$ Hz, 1H), 7.15 (d, $J = 7.2$ Hz, 2H), 6.35 (s, 1H), 2.22-2.15 (m, 1H), 1.96-1.89 (m, 1H), 1.16-1.13 (m, 1H), 1.05-0.98 (m, 2H), 0.89-0.77 (m, 2H), 0.66-0.59 (m, 1H), 0.36-0.30 (m, 1H), 0.24-0.17 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.9, 163.7 (d, $^1J_{\text{CF}} = 206.3$ Hz), 163.3 (d, $^1J_{\text{CF}} = 217.5$ Hz), 157.0, 151.2, 145.9, 144.6, 133.3, 131.4 (d, $^3J_{\text{CF}} = 10.0$ Hz), 130.1, 128.8, 128.3, 127.5 (d, $^3J_{\text{CF}} = 10.0$ Hz), 117.6 (d, $^2J_{\text{CF}} = 26.3$ Hz), 115.0 (d, $^2J_{\text{CF}} = 23.7$ Hz), 110.5 (d, $^2J_{\text{CF}} = 21.9$ Hz), 109.5 (d, $^2J_{\text{CF}} = 21.2$ Hz), 103.0, 16.9, 13.8, 9.8, 9.7, 7.2, 7.1; HRMS calcd. For $\text{C}_{24}\text{H}_{19}\text{F}_2\text{N}_2\text{O}^+ [\text{M}+\text{H}]^+$: 389.1460, found 389.1470.



3-(4-Chlorophenyl)-2-(3-(4-chlorophenyl)-7-fluoroisoquinolin-1-yl)-7-fluoroisoquinolin-1(2H)-one

lin-1(2*H*)-one **2m**

¹H NMR (400 MHz, CDCl₃): δ 8.13-8.10 (s, 1H), 7.96 (s, 1H), 7.90-7.83 (m, 3H), 7.69-7.65 (m, 1H), 7.54-7.35 (m, 5H), 7.12 (d, *J* = 8.4 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 2H), 6.69 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 162.3, 161.9 (d, ¹*J*_{CF} = 248.7 Hz), 161.7 (d, ¹*J*_{CF} = 251.8 Hz), 150.8, 148.5, 141.5, 136.5, 135.6, 135.0, 134.6, 133.7, 133.5, 130.4 (d, *J* = 8.8 Hz), 130.1, 128.9, 128.7 (d, ³*J*_{CF} = 7.5 Hz), 128.0, 126.9 (d, ³*J*_{CF} = 8.3 Hz), 126.4 (d, ³*J*_{CF} = 8.4 Hz), 122.3, 122.1, 121.8, 117.2, 113.6 (d, ²*J*_{CF} = 22.8 Hz), 108.1 (d, ²*J*_{CF} = 22.2 Hz), 107.8; HRMS calcd. for C₃₀H₁₇Cl₂F₂N₂O⁺ [M+H]⁺: 529.0681, found 529.0648.



6,7-Dimethoxy-2-(6,7-dimethoxy-3-(4-methoxyphenyl)isoquinolin-1-yl)-3-(4-methoxyphenyl)isoquinolin-1(2*H*)-one **2n**

¹H NMR (400 MHz, CDCl₃): δ 7.87 (d, *J* = 7.2 Hz, 3H), 7.75 (s, 1H), 7.13 (d, *J* = 7.6 Hz, 2H), 7.04 (s, 1H), 6.99 (s, 1H), 6.95 (d, *J* = 7.2 Hz, 2H), 6.84 (s, 1H), 6.60 (s, 1H), 6.51 (d, *J* = 7.6 Hz, 2H), 4.04 (s, 3H), 4.01 (s, 3H), 3.99 (s, 3H), 3.86 (s, 3H), 3.84 (s, 3H), 3.61 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 162.5, 159.8, 159.0, 153.9, 153.0, 150.6, 149.7, 149.1, 148.8, 142.3, 135.5, 132.8, 131.8, 130.0, 128.2, 128.0, 121.1, 119.0, 115.4, 113.8, 112.9, 108.2, 107.1, 106.1, 105.1, 102.2, 56.1, 56.0, 55.3, 55.0; HRMS calcd. for C₃₆H₃₃N₂O₇⁺ [M+H]⁺: 605.2282, found 605.2278.

