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Supporting Information

Ortho-Nitro-Substituted Diaryliodonium Salts Enabled Regioselective Cyclization of Arylcarboxylic Acids toward 3,4-Naphthocoumarins

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Part 1. General Information

a. Methods:

NMR spectrum: ¹H, ¹³C, and ¹⁹F NMR spectra were recorded in CDCl₃ or DMSO- d_6 (with tetramethylsilane as an internal standard) on a Bruker AVANCE 400 spectrometer at ambient temperature, operating at 400 MHz, 101 MHz, and 376 MHz respectively. Data were reported as follows: Chemical shifts (δ) are reported in ppm, and coupling constants (J) are in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad.

Mass spectroscopy: Mass spectra were in general recorded on a Waters LCT Premier XE spectrometer or EI Mass spectra were measured on HP HP5989A, Aglient HP5873 or Waters Micromass GCT mass spectrometer. ESI-MS analyses were performed in positive ionization mode on an Agilent 1100-MSD or Bruker Daltonics FTMS-7 mass spectrometer.

Chromatography: Column chromatography was performed with silica gel (200-300 mesh ASTM).

b. Materials:

All solvents were purchased from Adamas-beta and dried and/or distilled by standard methods. All reagents were purchased from commercial sources (Adamas-beta; Sinopharma reagents; TCI; Acros) and used without further purification. Reactions were monitored by TLC (detection with UV light).The preparation of all other materials is described in detail below.

Part 2. Synthesis and Characterization of *ortho*-Nitro-Substituted Diaryliodonium Salts



General procedure 1:^[1] To a solution of substituted 1-nitro-2-iodobenzene (5.0 mmol) in CH₂Cl₂ (20 mL) was slowly added *m*CPBA (0.95 g, 5.5 mmol) at room temperature. After stirring for 20 minutes, trifluoromethanesulfonic acid (1.18 ml, 15mmol)was added dropwise over 2 minute at 0 °C, and the solution was stirred at room temperature for 1 hour. Mesitylene (0.74 ml, 5.5mmol) was added at 0 °C and then the solution was stirred at room temperature for 1-2 hours until TLC indicated that the 1-nitro-2-iodobenzene was completely consumed. The solvent was removed in vacuo and Et₂O was added in the mixture. The mixture was stirred vigorously until the solid precipitated. The obtained solids were washed with Et₂O and collected with filtration under reduced pressure to give the diaryliodonium salts (**2a-2l**) in a pure form.

Mesityl(2-nitrophenyl)- λ^3 -iodaneyl trifluoromethanesulfonate (2a)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2a) was obtained as white solid (2.14 g, 83%). M.P.: 168-171 °C.

¹**H NMR (400 MHz, DMSO-***d*₆) δ 8.63 (d, *J* = 7.9 Hz, 1H), 7.88 (dt, *J* = 21.3, 7.5 Hz, 2H), 7.45 (s, 2H), 7.07 (d, *J* = 7.8 Hz, 1H), 2.58 (s, 6H), 2.44 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 145.69, 144.89, 143.08, 138.06, 132.20, 130.19, 127.95, 127.78, 120.51 (q, *J* = 322.3 Hz), 119.68, 107.03, 25.96, 20.73.

HRMS m/z (ESI-TOF): calculated for C₁₅H₁₅NO₂I⁺ [M-OTf]⁺ 368.0142, found 368.0143.

Mesityl(4-methyl-2-nitrophenyl)- λ^3 -iodaneyl trifluoromethanesulfonate (2b)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2b) was obtained as white solid (2.31 g, 87%). M.P.: 198-201 °C.

¹**H NMR (400 MHz, DMSO-***d*_{*b*}) δ 8.47 (d, *J* = 2.0 Hz, 1H), 7.67 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.43 (s, 2H), 6.96 (d, *J* = 8.3 Hz, 1H), 2.58 (s, 6H), 2.47 (s, 3H), 2.43 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 145.58, 144.79, 143.25, 142.98, 138.58, 130.17, 130.03, 127.99, 120.51 (q, *J* = 322.5 Hz), 119.74, 103.21, 26.03, 20.60, 19.92.

HRMS m/z (ESI-TOF): calculated for C₁₆H₁₇NO₂I⁺ [M-OTf]⁺ 382.0298, found 382.0297.

(4-Fluoro-2-nitrophenyl)(mesityl)- λ^3 -iodaneyl trifluoromethanesulfonate (2c)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2c) was obtained as white solid (1.84 g, 69%). M.P.: 180-183 °C.

¹H NMR (400 MHz, DMSO- d_6) δ 8.59 (dd, J = 8.6, 2.9 Hz, 1H), 7.78 (td, J = 8.3, 2.9 Hz, 1H), 7.44 (s,

2H), 7.11 (dd, *J* = 9.0, 4.9 Hz, 1H), 2.59 (s, 6H), 2.44 (s, 3H).

¹³C NMR (101 MHz, DMSO- d_6) δ 164.26, 161.74, 147.14 (d, J = 9.2 Hz), 144.91, 143.00, 132.32 (d, J = 8.8 Hz), 130.24, 124.98 (d, J = 22.6 Hz), 120.53 (q, J = 322.5 Hz), 115.70 (d, J = 28.1 Hz), 101.83, 26.05, 20.60.

¹⁹F NMR (376 MHz, DMSO-d₆) δ -77.80, -107.06.

HRMS m/z (EI-TOF): calculated for C₁₅H₁₄FNO₂I⁺ [M-OTf]⁺ 386.0048, found 386.0045.

(4-Chloro-2-nitrophenyl)(mesityl)- λ^3 -iodaneyl trifluoromethanesulfonate (2d)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2d) was obtained as white solid (2.41 g, 87%). M.P.: 184-187 °C.

¹**H NMR (400 MHz, DMSO-***d*_{*b*}) δ 8.68 (d, *J* = 2.4 Hz, 1H), 7.92 (dd, *J* = 8.7, 2.4 Hz, 1H), 7.44 (s, 2H), 7.04 (d, *J* = 8.6 Hz, 1H), 2.59 (s, 6H), 2.44 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 147.14, 145.44, 143.50, 137.81, 137.27, 132.13, 130.71, 127.81, 120.93 (q, *J* = 322.4 Hz), 120.39, 106.08, 26.50, 21.05.

HRMS m/z (ESI-TOF): calculated for $C_{15}H_{14}CINO_2I^+$ [M-OTf]⁺ 401.9752, found 401.9757.

(4-Bromo-2-nitrophenyl)(mesityl)- λ^3 -iodaneyl trifluoromethanesulfonate (2e)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2e) was obtained as white solid (2.51 g, 84%). M.P.: 197-200 °C.

¹**H NMR (400 MHz, DMSO-***d*₆) δ 8.75 (d, *J* = 2.2 Hz, 1H), 8.04 (dd, *J* = 8.6, 2.2 Hz, 1H), 7.44 (s, 2H), 6.96 (d, *J* = 8.5 Hz, 1H), 2.59 (s, 6H), 2.44 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 146.64, 145.08, 143.13, 140.38, 131.82, 130.34, 130.13, 124.97, 120.54 (q, *J* = 322.4 Hz), 119.90, 106.33, 26.11, 20.67. HRMS m/z (ESI-TOF): calculated for C₁₅H₁₄BrNO₂I⁺ [M-OTf]⁺ 445.9247, found 445.9250.

Mesityl(4-methoxy-2-nitrophenyl)- λ^3 -iodaneyl trifluoromethanesulfonate (2f)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2f) was obtained as yellow solid (2.25 g, 82%). M.P.: 185-188 °C.

¹**H NMR (400 MHz, DMSO-***d*_{*b*}) δ 8.07 (d, *J* = 2.9 Hz, 1H), 7.47 (dd, *J* = 9.1, 3.0 Hz, 1H), 7.42 (s, 2H), 7.02 (d, *J* = 9.0 Hz, 1H), 3.91 (s, 3H), 2.59 (s, 6H), 2.42 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 161.89, 147.19, 144.92, 143.09, 131.94, 130.38, 123.88, 120.75 (q, *J* = 322.3 Hz), 120.32, 113.28, 95.75, 56.76, 26.27, 20.79.

HRMS m/z (ESI-TOF): calculated for C₁₆H₁₇NO₃I⁺ [M-OTf]⁺ 398.0248, found 398.0246.

Methyl 4-(mesityl(((trifluoromethyl)sulfonyl)oxy)- λ^3 -iodaneyl)-3-nitrobenzoate (2g)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2g) was obtained as white solid (2.43 g, 85%). M.P.: 162-165 °C.

¹**H NMR (400 MHz, DMSO-***d*₆) δ 8.84 (d, *J* = 1.9 Hz, 1H), 8.29 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.45 (s, 2H), 7.25 (d, *J* = 8.4 Hz, 1H), 3.92 (s, 3H), 2.59 (s, 6H), 2.44 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 163.73, 146.45, 145.40, 143.41, 141.14, 137.60, 133.43, 131.56, 130.63, 128.13, 127.38, 120.82 (q, J = 322.4 Hz), 120.29, 112.58, 53.31, 29.02, 26.35, 20.91. HRMS m/z (ESI-TOF): calculated for C₁₇H₁₇NO₄I⁺ [M-OTf]⁺ 426.0197, found 426.0196.

Mesityl(5-methyl-2-nitrophenyl)- λ^3 -iodaneyl trifluoromethanesulfonate (2h)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2h) was obtained as white solid (2.2 g, 83%). M.P.: 186-189 °C.

¹**H NMR (400 MHz, DMSO-***d*_{*b*}) δ 8.51 (d, *J* = 8.4 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.45 (s, 2H), 6.80 (s, 1H), 2.61 (s, 6H), 2.45 (s, 3H), 2.32 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 150.22, 145.32, 144.15, 143.58, 133.17, 130.63, 130.53, 128.11, 120.97 (q, *J* = 322.4 Hz), 119.95, 107.49, 26.49, 21.62, 21.05.

HRMS m/z (ESI-TOF): calculated for C₁₆H₁₇NO₂I⁺ [M-OTf]⁺ 382.0298, found 382.0300.

(5-Fluoro-2-nitrophenyl)(mesityl)- λ^3 -iodaneyl trifluoromethanesulfonate (2i)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2i) was obtained as white solid (2.14 g, 80%). M.P.: 197-200 °C.

¹**H NMR (400 MHz, DMSO-***d*_{*b*}) δ 8.70 (dd, *J* = 9.1, 4.6 Hz, 1H), 7.79 (ddd, *J* = 9.5, 7.4, 2.5 Hz, 1H), 7.46 (s, 2H), 6.65 (dd, *J* = 7.5, 2.6 Hz, 1H), 2.61 (s, 6H), 2.45 (s, 3H).

¹³C NMR (101 MHz, DMSO- d_6) δ 167.79, 165.17, 145.65, 143.60, 143.12 (d, J = 2.8 Hz), 130.84 (d, J = 15.7 Hz), 120.69, 120.54 (q, J = 322.4 Hz), 119.70 (d, J = 23.5 Hz), 117.92 (d, J = 28.2 Hz), 110.46 (d, J = 8.0 Hz), 26.47, 21.06

¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -77.79, -97.31.

HRMS m/z (EI-TOF): calculated for C₁₅H₁₄FNO₂I⁺ [M-OTf]⁺ 386.0048, found 386.0045.

(5-Chloro-2-nitrophenyl)(mesityl)- λ^3 -iodaneyl trifluoromethanesulfonate (2j)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2j) was obtained as white solid (2.49 g, 90%). M.P.: 181-184 °C.

¹**H NMR (400 MHz, DMSO-***d*₆) δ 8.60 (d, *J* = 8.8 Hz, 1H), 7.99 (dd, *J* = 8.8, 2.1 Hz, 1H), 7.47 (s, 2H), 6.82 (d, *J* = 2.1 Hz, 1H), 2.61 (s, 6H), 2.45 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 145.21, 144.94, 143.13, 142.39, 132.21, 130.30, 129.31, 129.03, 120.50 (q, *J* = 322.4 Hz), 120.17, 109.24, 26.02, 20.63.

HRMS m/z (ESI-TOF): calculated for C₁₅H₁₄ClNO₂I⁺ [M-OTf]⁺ 401.9752, found 401.9754.

(5-Bromo-2-nitrophenyl)(mesityl)- λ^3 -iodaneyl trifluoromethanesulfonate (2k)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (2k) was obtained as white solid (2.35 g, 79%). M.P.: 195-198 °C.

¹**H NMR (400 MHz, DMSO-***d*₆) δ 8.51 (d, *J* = 8.7 Hz, 1H), 8.11 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.48 (s, 2H), 6.97 (d, *J* = 1.9 Hz, 1H), 2.63 (s, 6H), 2.45 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 145.74, 143.65, 135.67, 132.48, 132.13, 130.79, 129.57, 120.56, 120.54 (q, *J* = 322.4 Hz), 119.37, 109.67, 26.49, 21.11.

HRMS m/z (ESI-TOF): calculated for $C_{15}H_{14}BrNO_2I^+$ [M-OTf]⁺ 445.9247, found 445.9252.

Mesityl(2-methyl-6-nitrophenyl)- λ^3 -iodaneyl trifluoromethanesulfonate (21)



Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (21) was obtained as white solid (2.29 g, 86%). M.P.: 169-172 °C.

¹**H NMR (400 MHz, DMSO-***d*₆) δ 8.00 (d, *J* = 7.9 Hz, 1H), 7.91 (d, *J* = 7.6 Hz, 1H), 7.80 (t, *J* = 7.8 Hz, 1H), 7.19 (s, 2H), 2.71 (s, 3H), 2.43 (s, 6H), 2.28 (s, 3H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 150.44, 144.94, 143.29, 142.08, 136.53, 133.43, 130.19, 124.81, 121.11, 120.58 (q, *J* = 322.5 Hz), 109.60, 28.79, 25.32, 20.22.

HRMS m/z (ESI-TOF): calculated for $C_{16}H_{17}NO_2I^+$ [M-OTf]⁺ 382.0298, found 382.0301.

(2-Nitrophenyl)(2,4,6-trimethoxyphenyl)- λ^3 -iodaneyl trifluoromethanesulfonate (2m)

NO₂ OTf

Prepared according to the *General procedure 1* on 5 mmol scale. The desired product of *ortho*-Nitro-Substituted Diaryliodonium Salt (**2m**) was obtained as white solid (2.03 g, 72%).

M.P.: 176-179 °C.

¹**H NMR (400 MHz, DMSO-***d*₆) δ 8.65 (d, *J* = 9.7 Hz, 1H), 8.16 – 7.69 (m, 2H), 7.54 (s, 2H), 7.22 – 6.57 (m, 1H), 3.15 (dq, *J* = 18.9, 6.8 Hz, 3H), 1.31 (d, *J* = 6.9 Hz, 18H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 156.38, 152.93, 145.96, 138.44, 132.63, 130.43, 128.13, 125.50, δ 120.90 (q, *J* = 322.3 Hz), 120.03, 108.50, 33.91, 24.28.

HRMS m/z (ESI-TOF): calculated for C₂₁H₂₇NO₂I⁺ [M-OTf]⁺452.1086, found 452.1079.

Part 3. Table of Reaction Condition Optimization



Table S1: Base optimization

| 1 | | |
|-------|---------------------------------|-------|
| Entry | Base | Yield |
| 1 | K ₂ CO ₃ | 48% |
| 2 | Cs_2CO_3 | 11% |
| 3 | ^t BuOK | 0% |
| 4 | AcONa | 0% |
| 5 | Na ₂ CO ₃ | 35% |
| 6 | KH ₂ PO ₄ | 0% |
| 7 | ^t BuONa | 0% |
| 8 | CH ₃ ONa | 0% |
| 9 | КОН | 10% |
| 10 | NaH | 0% |
| 11 | ^t BuOLi | 0% |
| 12 | K ₃ PO ₄ | 51% |
| 13 | DMAP | 0% |
| 14 | AcOH | 0% |
| 15 | / | 0% |
| 16 | $K_2CO_3(2eq)$ | 55% |
| 17 | $K_3PO_4(2eq)$ | 54% |

Reaction conditions unless specifically noted: 1a (68.8mg, 0.4 mmol, 1eq), 2a (310.2mg, 0.6 mmol, 1.5eq), base (0.4mmol,1eq), and catalyst $Pd(OAc)_2$ (9.1mg, 0.04mmol, 10 mol %) in solvent DMF(2 mL) at 110 °C for 24 h, Isolated yields were obtained after purification with column chromatography.

Table S2: Solvent optimization

| Entry | Solvent | Yield |
|-------|-------------------|-------|
| 1 | DMF | 55% |
| 2 | DMSO | 9% |
| 3 | DCE | 0% |
| 4 | Tol | 0% |
| 5 | Dioxane | 0% |
| 6 | NMP | 26% |
| 7 | H ₂ O | 0% |
| 8 | ^t BuOH | 6% |
| 9 | MeCN | 17% |
| 10 | PivOH | 0% |
| 11 | AcOH | 0% |

Reaction conditions unless specifically noted: 1a (68.8mg, 0.4 mmol, 1eq), 2a (310.2mg, 0.6 mmol, 1.5eq), K_2CO_3 (110.6mg, 0.8mmol, 2eq), and catalyst Pd(OAc)₂ (9.1mg, 0.04mmol, 10 mol %) in solvent (2 mL) at 110 °C for 24 h, Isolated yields were obtained after purification with column chromatography.

| Entry | Catalyst | Yield |
|-------|------------------------------------|-------|
| 1 | $Pd(OAc)_2$ | 55% |
| 2 | $Pd(NO_3)_2$ | 0% |
| 3 | $Pd(TFA)_2$ | 42% |
| 4 | $[Pd(PPh_3)_2]Cl_2$ | 11% |
| 5 | Pd(PPh ₃) ₄ | 0% |
| 6 | Cu(OTf) ₂ | 0% |
| 7 | CuCl | 0% |
| 8 | / | 0% |

Table S3: Catalyst optimization

Reaction conditions unless specifically noted: 1a (68.8mg, 0.4 mmol, 1eq), 2a (310.2mg, 0.6 mmol, 1.5eq), K_2CO_3 (110.6mg, 0.8mmol, 2eq), and catalyst (0.04mmol,10 mol %) in solvent DMF (2 mL) at 110 °C for 24 h, Isolated yields were obtained after purification with column chromatography.

Table S4: Temperature optimization

| Entry | Temperature(°C) | Yield |
|-------|-----------------|-------|
| 1 | 50 | 0% |
| 2 | 80 | 12% |
| 3 | 110 | 55% |
| 4 | 130 | 62% |
| 5 | 150 | 56% |

Reaction conditions unless specifically noted: 1a (68.8mg, 0.4 mmol, 1eq), 2a (310.2mg, 0.6 mmol, 1.5eq), K_2CO_3 (110.6mg, 0.8mmol, 2eq), and catalyst Pd(OAc)₂ (9.1mg, 0.04mmol, 10 mol %) in solvent DMF (2 mL) at different temperatures for 24 h, Isolated yields were obtained after purification with column chromatography.



Part 4. Synthesis and Characterization of Products from Arylation Reactions

General procedure 2: To an oven-dried Schlenk tube was added 1-naphthoic acid or 2-naphthoic acid 1 (0.4 mmol, 1.0 equiv.), *ortho*-Nitro-substituted diaryliodonium salts 2 (0.6 mmol, 1.5 equiv.), $Pd(OAc)_2$ (10 mol %) and K_2CO_3 (0.8 mmol, 2.0 equiv.), and then DMF (2 mL) was added via a syringe. The mixture was stirred at 130 °C in an oil bath until TLC indicated that the 1-naphthoic acid or 2-naphthoic acid was completely consumed. The solvent was washed with water (30 mL) and extracted with DCM (15 mL) for three times, and then the solvent was evaporated under vacuum. The crude products were purified using flash column chromatography (eluent: Petroleum ether: EtOAc) on silica gel to afford the desired product **3**.

5H-Naphtho[1,2-c]chromen-5-one (3aa)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 62% (61.0 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 190-193 °C.

¹**H NMR (400 MHz, CDCl₃)** δ 9.80 (d, *J* = 8.8 Hz, 1H), 8.23 (d, *J* = 8.8 Hz, 1H), 8.21 – 8.13 (m, 2H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.77 (t, *J* = 7.2 Hz, 1H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.54 (t, *J* = 7.0 Hz, 1H), 7.42 (d, *J* = 7.4 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 160.84, 152.10, 137.05, 136.76, 133.64, 132.42, 131.27, 130.06, 129.17, 127.79, 127.64, 124.79, 123.79, 119.18, 118.55, 117.58, 115.56.

HRMS m/z (EI-TOF): calculated for C₁₇H₁₀O₂ [M]⁺ 246.0681, found 246.0678.

12-Methyl-5*H*-naphtho[1,2-*c*]chromen-5-one (3ab)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1b** (74.4 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 68% (70.7 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.85 (d, *J* = 8.7 Hz, 1H), 8.15 (d, *J* = 9.2 Hz, 1H), 8.07 (d, *J* = 8.3 Hz, 1H), 8.00 (s, 1H), 7.80 – 7.70 (m, 1H), 7.65 (t, *J* = 8.2 Hz, 1H), 7.56 – 7.47 (m, 1H), 7.44 – 7.31 (m, 2H), 2.84 (s, 3H).

LRMS m/z (EI-TOF): calculated for $C_{18}H_{12}O_2$ [M]⁺ 260.1, found 260.1. Analytical data are consistent with the reported ones.^[2]

12-Ethyl-5*H*-naphtho[1,2-*c*]chromen-5-one (3ac)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1c** (80.0 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 55% (60.3 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.86 (d, *J* = 8.6 Hz, 1H), 8.23 – 8.07 (m, 2H), 8.00 (s, 1H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.37 (dd, *J* = 16.7, 8.2 Hz, 2H), 3.23 (q, *J* = 7.4 Hz, 2H), 1.47 (t, *J* = 7.4 Hz, 3H).

LRMS m/z (EI-TOF): calculated for $C_{19}H_{14}O_2$ [M]⁺ 274.1, found 274.1. Analytical data are consistent with the reported ones.^[2]

12-Propyl-5*H*-naphtho[1,2-*c*]chromen-5-one (3ad)



Prepared according to the general procedure 2 on 0.4 mmol scale, the reaction of 1d (85.6 mg, 0.4

mmol), **2a** (310.2 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), Pd(OAc)₂ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 50% (57.6 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel. **M.P.**: 152-155 °C.

¹**H NMR (400 MHz, CDCl₃)** δ 9.83 (d, *J* = 8.4 Hz, 1H), 8.09 (dd, *J* = 11.4, 7.9 Hz, 2H), 7.92 (s, 1H), 7.70 (t, *J* = 8.4 Hz, 1H), 7.60 (t, *J* = 8.1 Hz, 1H), 7.48 (t, *J* = 7.0 Hz, 1H), 7.33 (dd, *J* = 15.0, 7.8 Hz, 2H), 3.18 – 3.03 (m, 2H), 1.84 (h, *J* = 7.4 Hz, 2H), 1.08 (t, *J* = 7.3 Hz, 3H).

 $\label{eq:stars} \begin{array}{l} {}^{13}\text{C NMR} \ (101 \ \text{MHz}, \text{CDCl}_3) \ \delta \ 160.40 \ , \ 151.83 \ , \ 147.91 \ , \ 136.11 \ , \ 132.46 \ , \ 132.00 \ , \ 130.70 \ , \ 128.96 \ , \\ 127.96 \ , \ 127.83 \ , \ 127.01 \ , \ 124.20 \ , \ 123.47 \ , \ 118.70 \ , \ 118.14 \ , \ 117.11 \ , \ 113.65 \ , \ 36.29 \ , \ 23.75 \ , \ 14.35. \\ \textbf{HRMS m/z} \ (\textbf{EI-TOF}): \ calculated \ for \ C_{20}H_{16}O_2 \ [\text{M}]^+ \ 288.1150, \ found \ 288.1153. \end{array}$

12-Chloro-5*H*-naphtho[1,2-*c*]chromen-5-one (3ae)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of 1e (82.4 mg, 0.4 mmol), 2a (310.2 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 41% (45.9 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 165-168 °C.

¹**H NMR (400 MHz, CDCl₃)** δ 9.83 (d, *J* = 8.2 Hz, 1H), 8.36 (d, *J* = 8.2 Hz, 1H), 8.22 (s, 1H), 8.05 (d, *J* = 8.0 Hz, 1H), 7.80 (t, *J* = 7.6 Hz, 1H), 7.70 (t, *J* = 7.4 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.43 – 7.33 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 159.52, 151.46, 140.82, 136.25, 132.93, 131.16, 130.24, 130.08, 127.91, 127.49, 127.35, 124.90, 124.33, 123.04, 119.32, 116.91, 113.86. HRMS m/z (EI-TOF): calculated for $C_{17}H_9ClO_2$ [M]⁺ 280.0291, found 280.0293.

12-Bromo-5*H*-naphtho[1,2-*c*]chromen-5-one (3af)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1f** (100.0 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 47% (60.9 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.87 (d, J = 8.7 Hz, 1H), 8.54 (s, 1H), 8.40 (d, J = 8.0 Hz, 1H), 8.13 (d,

J = 7.9 Hz, 1H), 7.83 (t, J = 7.5 Hz, 1H), 7.74 (t, J = 7.3 Hz, 1H), 7.58 (t, J = 7.5 Hz, 1H), 7.42 (dd, J = 17.2, 8.1 Hz, 2H). **LRMS m/z (EI-TOF)**: calculated for C₁₇H₉BrO₂ [M]⁺ 324.0, found 324.0. Analytical data are consistent with the reported ones.^[2]

3-Bromo-5*H***-naphtho**[1,2-*c*]**chromen-5-one** (**3ag**)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1g** (100.0 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 35% (45.4 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 205-208 °C.

¹H NMR (400 MHz, CDCl₃) δ 9.97 (s, 1H), 8.16 (s, 2H), 8.12 (d, J = 8.0 Hz, 1H), 7.73 (d, J = 8.6 Hz, 1H), 7.67 (d, J = 8.6 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 7.38 (dd, J = 12.1, 7.7 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 160.43 , 152.11 , 137.70 , 136.53 , 133.26 , 132.00 , 131.69 , 131.14 , 130.36 , 129.98 , 125.14 , 124.85 , 124.03 , 119.64 , 118.14 , 117.73 , 114.55. HRMS m/z (ESI-TOF): calculated for C₁₇H₁₀BrO₂ [M+H]⁺ 324.9859, found 324.9856.

12-Methoxy-5H-naphtho[1,2-c]chromen-5-one (3ah)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1h** (80.8 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 45% (49.7 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.77 (d, *J* = 8.8 Hz, 1H), 8.33 (d, *J* = 8.3 Hz, 1H), 8.06 (d, *J* = 7.9 Hz, 1H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.59 (t, *J* = 7.5 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.42 – 7.29 (m, 3H), 4.18 (s, 3H).

LRMS m/z (EI-TOF): calculated for $C_{18}H_{12}O_3$ [M]⁺ 276.1, found 276.1. Analytical data are consistent with the reported ones.^[2]

12-Ethoxy-5*H*-naphtho[1,2-*c*]chromen-5-one (3ai)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1i** (86.4 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 31% (36.0 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 193-196°C

¹**H NMR (400 MHz, CDCl₃)** δ 9.78 (d, *J* = 8.7 Hz, 1H), 8.38 (d, *J* = 8.6 Hz, 1H), 8.06 (d, *J* = 9.1 Hz, 1H), 7.80 – 7.72 (m, 1H), 7.65 – 7.56 (m, 1H), 7.54 – 7.47 (m, 1H), 7.41 – 7.31 (m, 3H), 4.40 (q, *J* = 7.0 Hz, 2H), 1.65 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 160.86 , 152.22 , 138.96 , 133.91 , 131.20 , 130.29 , 127.68 , 127.53 , 126.97 , 126.47 , 124.49 , 123.49 , 122.86 , 118.69 , 117.75 , 109.00 , 97.10 , 64.87 , 15.00. HRMS m/z (EI-TOF): calculated for $C_{19}H_{14}O_3$ [M]⁺ 290.0943, found 290.0945.

2-Methoxy-5H-naphtho[1,2-c]chromen-5-one (3aj)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1j** (80.8 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 51% (56.3 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.64 (d, *J* = 9.5 Hz, 1H), 8.04 (d, *J* = 1.5 Hz, 3H), 7.46 (ddd, *J* = 8.5, 7.2, 1.5 Hz, 1H), 7.40 – 7.22 (m, 3H), 7.13 (d, *J* = 2.8 Hz, 1H), 3.92 (s, 3H).

LRMS m/z (EI-TOF): calculated for $C_{18}H_{12}O_3$ [M]⁺ 276.1, found 276.1.

Analytical data are consistent with the reported ones.^[2]

1,2,3,4-Tetrahydro-5*H*-naphtho[1,2-*c*]chromen-5-one (3ak)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of 1k (70.4 mg, 0.4 mmol), 2a (310.2 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 18% (18 mg) as a white solid after column

chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 7.98 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.84 (d, *J* = 8.3 Hz, 1H), 7.46 (d, *J* = 8.3 Hz, 1H), 7.41 (ddd, *J* = 8.4, 7.0, 1.5 Hz, 1H), 7.30 – 7.24 (m, 2H), 3.39 (t, *J* = 6.2 Hz, 2H), 2.87 (t, *J* = 6.2 Hz, 2H), 2.12 – 1.68 (m, 4H).

LRMS m/z (EI-TOF): calculated for $C_{17}H_{14}O_2$ [M]⁺ 250.1, found 250.1.

Analytical data are consistent with the reported ones.^[3]

6H-Naphtho[2,3-c]chromen-6-one (3am)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1m** (68.8 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 70% (68.8 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

М.Р.: 176-179 °С

¹**H NMR (400 MHz, CDCl₃)** δ 8.97 (s, 1H), 8.48 (s, 1H), 8.18 (dd, J = 8.3, 1.5 Hz, 1H), 7.99 (dd, J = 9.9, 8.4 Hz, 2H), 7.66 (ddd, J = 8.2, 6.8, 1.3 Hz, 1H), 7.57 (ddd, J = 8.1, 6.8, 1.2 Hz, 1H), 7.51 – 7.41 (m, 1H), 7.35 (td, J = 7.7, 1.2 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 161.30, 150.60, 135.95, 132.66, 132.56, 132.20, 129.95, 129.41, 129.39, 127.97, 126.99, 124.40, 122.75, 120.56, 118.95, 118.09, 117.78.

HRMS m/z (EI-TOF): calculated for $C_{17}H_{10}O_2$ [M]⁺ 246.0681, found 246.0678. Analytical data are consistent with the reported ones.^[4]

10-Methoxy-6*H*-naphtho[2,3-*c*]chromen-6-one (3an)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1n** (80.8 mg, 0.4 mmol), **2a** (310.2 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 60% (66.3 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

М.Р.:. 195-198 °С

¹**H NMR (400 MHz, CDCl₃)** δ 8.67 (s, 1H), 8.08 (s, 1H), 7.96 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.71 (d, *J* = 9.0 Hz, 1H), 7.37 – 7.34 (m, 1H), 7.26 – 7.20 (m, 2H), 7.08 (dd, *J* = 9.0, 2.5 Hz, 1H), 7.04 (d, *J* = 2.4 Hz, 1H), 3.91 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 161.91, 160.72, 151.15, 138.23, 132.54, 131.27, 130.35, 130.32, 128.39, 124.79, 123.06, 121.10, 119.17, 118.57, 118.10, 117.10, 105.44, 55.83. HRMS m/z (ESI-TOF): calculated for $C_{18}H_{13}O_3$ [M+H]⁺ 277.0859, found 277.0855.

8-Methyl-5*H*-naphtho[1,2-*c*]chromen-5-one (3ba)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2b** (318.6 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 61% (63.5 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.78 (d, *J* = 8.8 Hz, 1H), 8.19 (d, *J* = 8.8 Hz, 1H), 8.12 (d, *J* = 8.9 Hz, 1H), 8.00 (d, *J* = 8.2 Hz, 1H), 7.89 (d, *J* = 7.9 Hz, 1H), 7.75 (ddd, *J* = 8.6, 6.9, 1.4 Hz, 1H), 7.69 – 7.53 (m, 1H), 7.23 – 7.08 (m, 2H), 2.46 (s, 3H).

HRMS m/z (ESI-TOF): calculated for $C_{18}H_{13}O_2$ [M+H]⁺ 261.0910, found 261.0907. Analytical data are consistent with the reported ones.^[2]

8-Fluoro-5*H*-naphtho[1,2-*c*]chromen-5-one (3ca)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2c** (321.0 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 49% (51.8 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.72 (d, *J* = 8.8 Hz, 1H), 8.20 (d, *J* = 8.8 Hz, 1H), 8.08 (dd, *J* = 12.5, 8.0 Hz, 2H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.75 (t, *J* = 7.8 Hz, 1H), 7.62 (t, *J* = 7.3 Hz, 1H), 7.10 (d, *J* = 8.7 Hz, 2H).

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}FO_2$ [M+H]⁺ 265.0659 found 265.0658. Analytical data are consistent with the reported ones.^[2]

8-Chloro-5*H*-naphtho[1,2-*c*]chromen-5-one (3da)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2d** (330.6 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 54% (60.5 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹H NMR (400 MHz, CDCl₃) δ 9.60 (d, J = 8.7 Hz, 1H), 8.09 (d, J = 8.7 Hz, 1H), 7.93 (dd, J = 12.4, 8.9 Hz, 2H), 7.79 (d, J = 7.9 Hz, 1H), 7.70 – 7.61 (m, 1H), 7.53 (t, J = 7.4 Hz, 1H), 7.32 – 7.14 (m, 2H). HRMS m/z (ESI-TOF): calculated for C₁₇H₁₀ClO₂ [M+H]⁺ 281.0364, found 281.0361. Analytical data are consistent with the reported ones.^[2]

8-Bromo-5*H*-naphtho[1,2-*c*]chromen-5-one (3ea)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2e** (356.9 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 55% (71.3 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.70 (d, *J* = 8.7 Hz, 1H), 8.19 (d, *J* = 8.8 Hz, 1H), 8.05 (d, *J* = 8.8 Hz, 1H), 7.94 (d, *J* = 8.6 Hz, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.74 (t, *J* = 7.8 Hz, 1H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.52 (s, 1H), 7.44 (d, *J* = 8.6 Hz, 1H).

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}BrO_2$ [M+H]⁺ 324.9859, found 324.9858. Analytical data are consistent with the reported ones.^[2]

8-Methoxy-5H-naphtho[1,2-c]chromen-5-one (3fa)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2f** (328.2 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 71% (78.4 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 165-168 °C.

¹**H NMR (400 MHz, CDCl**₃δ 9.76 (d, *J* = 8.8 Hz, 1H), 8.18 (d, *J* = 8.8 Hz, 1H), 8.06 (dd, *J* = 15.3, 8.9 Hz, 2H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.75 (t, *J* = 7.8 Hz, 1H), 7.60 (t, *J* = 7.3 Hz, 1H), 6.94 (d, *J* = 9.0 Hz, 1H), 6.90 (s, 1H), 3.90 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 160.62, 158.37, 151.36, 135.27, 135.02, 134.40, 130.30, 128.97, 126.98, 124.28, 123.09, 121.17, 119.40, 118.32, 117.18, 115.27, 107.25, 55.57.

HRMS m/z (ESI-TOF): calculated for C₁₈H₁₃O₃ [M+H]⁺ 277.0859, found 277.0854.

Methyl 5-oxo-5*H*-naphtho[1,2-*c*]chromene-8-carboxylate (3ga)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2g** (345.0 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 35% (42.6 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.78 (d, *J* = 8.7 Hz, 1H), 8.27 (d, *J* = 8.7 Hz, 1H), 8.21 (t, *J* = 7.8 Hz, 2H), 8.01 (d, *J* = 12.6 Hz, 2H), 7.93 (d, *J* = 7.9 Hz, 1H), 7.78 (t, *J* = 7.7 Hz, 1H), 7.66 (t, *J* = 7.4 Hz, 1H), 3.98 (s, 3H).

HRMS m/z (ESI-TOF): calculated for $C_{19}H_{13}O_4$ [M+H]⁺ 305.0808, found 305.0806. Analytical data are consistent with the reported ones.^[2]

9-Methyl-5*H*-naphtho[1,2-*c*]chromen-5-one (3ha)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2h** (318.6 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 65% (67.6 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.82 (d, J = 8.8 Hz, 1H), 8.20 (q, J = 8.9 Hz, 2H), 8.00 – 7.87 (m, 2H), 7.77 (ddd, J = 8.6, 6.9, 1.4 Hz, 1H), 7.70 – 7.55 (m, 1H), 7.40 – 7.28 (m, 2H), 2.49 (s, 3H).

HRMS m/z (ESI-TOF): calculated for $C_{18}H_{13}O_2$ [M+H]⁺ 261.0906, found 261.0910. Analytical data are consistent with the reported ones.^[2]

9-Fluoro-5*H*-naphtho[1,2-*c*]chromen-5-one (3ia)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2i** (321.0 mg, 0.6 mmol), K₂CO₃ (110.6 mg, 0.8 mmol), Pd(OAc)₂ (9.1 mg, 10 mol%), and DMF

(2 mL) at 130 °C for 24 h, and obtained an isolated yield of 49% (51.8 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.81 (d, *J* = 8.8 Hz, 1H), 8.27 (d, *J* = 8.8 Hz, 1H), 8.07 (d, *J* = 8.8 Hz, 1H), 7.94 (d, *J* = 7.9 Hz, 1H), 7.80 (ddd, *J* = 17.0, 9.0, 2.1 Hz, 2H), 7.73 – 7.59 (m, 1H), 7.41 (dd, *J* = 9.0, 4.7 Hz, 1H), 7.25 (s, 1H).

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}FO_2$ [M+H]⁺ 265.0659, found 265.0659. Analytical data are consistent with the reported ones.^[2]

9-Chloro-5*H*-naphtho[1,2-*c*]chromen-5-one (3ja)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2j** (330.6 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 48% (53.8 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.80 (d, *J* = 8.8 Hz, 1H), 8.28 (d, *J* = 8.8 Hz, 1H), 8.16 – 8.09 (m, 2H), 7.96 (d, *J* = 7.9 Hz, 1H), 7.80 (ddd, *J* = 8.6, 6.9, 1.4 Hz, 1H), 7.71 – 7.64 (m, 1H), 7.49 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.38 (d, *J* = 8.8 Hz, 1H).

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}ClO_2$ [M+H]⁺ 281.0364, found 281.0362. Analytical data are consistent with the reported ones.^[2]

9-Bromo-5*H*-naphtho[1,2-*c*]chromen-5-one (3ka)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **1a** (68.8 mg, 0.4 mmol), **2k** (356.9 mg, 0.6 mmol), $K_2CO_3(110.6 \text{ mg}, 0.8 \text{ mmol})$, $Pd(OAc)_2(9.1 \text{ mg}, 10 \text{ mol}\%)$, and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 50% (64.8 mg) as a yellow solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.74 (d, *J* = 8.8 Hz, 1H), 8.22 (dd, *J* = 9.5, 2.0 Hz, 2H), 8.06 (d, *J* = 8.9 Hz, 1H), 7.90 (d, *J* = 7.6 Hz, 1H), 7.75 (ddd, *J* = 8.6, 6.9, 1.4 Hz, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.62 – 7.54 (m, 1H), 7.26 (d, *J* = 17.8 Hz, 1H).

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}BrO_2$ [M+H]⁺ 324.9859, found 324.9856. Analytical data are consistent with the reported ones.^[2]

3-Methyl-6*H*-naphtho[2,3-*c*]chromen-6-one (3la)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **11** (68.8 mg, 0.4 mmol), **2b** (318.6 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 58% (60.3 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

М.Р.: 227-230 °С.

¹H NMR (400 MHz, CDCl₃) δ 8.94 (s, 1H), 8.42 (s, 1H), 8.03 (d, J = 8.1 Hz, 1H), 7.97 (dd, J = 12.2, 8.2 Hz, 2H), 7.72 – 7.59 (m, 1H), 7.60 – 7.51 (m, 1H), 7.20 – 7.06 (m, 2H), 2.44 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.87, 150.87, 141.05, 136.36, 132.93, 132.31, 129.99, 129.73, 129.61,

128.18, 127.04, 125.77, 122.68, 120.39, 119.16, 118.23, 115.74, 21.46.

HRMS m/z (ESI-TOF): calculated for C₁₈H₁₃O₂ [M+H]⁺ 261.0910, found 261.0907.

3-Fluoro-6*H*-naphtho[2,3-*c*]chromen-6-one (3ma)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **11** (68.8 mg, 0.4 mmol), **2c** (321.0 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), Pd(OAc)₂ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 56% (59.1 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.:192-195 °C.

¹**H** NMR (400 MHz, CDCl₃) δ 8.86 (s, 1H), 8.31 (s, 1H), 8.08 (dd, J = 8.8, 5.9 Hz, 1H), 7.93 (dd, J = 8.0, 4.6 Hz, 2H), 7.65 (t, J = 7.5 Hz, 1H), 7.54 (t, J = 7.5 Hz, 1H), 7.05 (td, J = 8.5, 2.5 Hz, 1H), 6.99 (dd, J = 8.9, 2.4 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 164.87, 161.90 (d, J = 95.8 Hz), 151.96 (d, J = 12.2 Hz), 136.60, 133.37, 132.63, 130.19, 129.93, 129.35, 128.40, 127.63, 124.78 (d, J = 9.7 Hz), 120.76, 118.75, 115.19 (d, J = 3.5 Hz), 112.83 (d, J = 22.3 Hz), 105.60 (d, J = 25.3 Hz).

¹⁹F NMR (376 MHz, CDCl₃) δ -110.24.

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}FO_2$ [M+H]⁺ 265.0659, found 265.0655.

3-Chloro-6*H*-naphtho[2,3-*c*]chromen-6-one (3na)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **11** (68.8 mg, 0.4 mmol), **2d** (330.6 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 41% (45.9 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 196-199 °C.

¹**H NMR (400 MHz, CDCl₃)** δ 8.87 (s, 1H), 8.36 (s, 1H), 8.04 (d, *J* = 8.5 Hz, 1H), 7.96 (d, *J* = 8.5 Hz, 2H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.31 – 7.26 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 161.22, 151.35, 136.51, 135.87, 133.39, 132.83, 130.25, 129.95, 129.09, 128.47, 127.83, 125.44, 124.23, 121.08, 118.99, 118.42, 117.33.

HRMS m/z (ESI-TOF): calculated for C₁₇H₁₀ClO₂ [M+H]⁺ 281.0364, found 281.0359.

3-Bromo-6*H*-naphtho[2,3-*c*]chromen-6-one (3oa)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **11** (68.8 mg, 0.4 mmol), **2e** (356.9 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 53% (68.7 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 188-191°C.

¹**H NMR (400 MHz, CDCl₃)** δ 8.86 (s, 1H), 8.35 (s, 1H), 7.97 – 7.89 (m, 3H), 7.66 (t, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.41 (d, *J* = 9.0 Hz, 2H).

¹³**C NMR (101 MHz, CDCl₃)** δ 161.14, 151.37, 136.51, 133.40, 132.89, 130.26, 129.97, 129.14, 128.49, 128.27, 127.87, 124.40, 123.64, 121.36, 121.10, 119.07, 117.76.

HRMS m/z (ESI-TOF): calculated for C₁₇H₁₀BrO₂ [M+H]⁺ 324.9859, found 324.9852.

2-Methyl-6*H*-naphtho[2,3-*c*]chromen-6-one (3pa)

Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **11** (68.8 mg, 0.4 mmol), **2h** (318.6 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 73% (75.9 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel. **M.P.**: 230-233 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.77 (s, 1H), 8.23 (s, 1H), 7.85 (t, *J* = 7.9 Hz, 2H), 7.75 (s, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.14 (d, *J* = 9.0 Hz, 1H), 7.09 (d, *J* = 8.3 Hz, 1H), 2.41 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.87, 149.07, 136.34, 134.45, 132.88, 132.53, 131.28, 129.87, 129.78, 129.74, 128.32, 127.35, 123.11, 120.70, 119.38, 118.03, 117.80, 21.47.

HRMS m/z (ESI-TOF): calculated for C₁₈H₁₃O₂ [M+H]⁺ 261.0910, found 261.0906.

2-Fluoro-6*H*-naphtho[2,3-*c*]chromen-6-one (3qa)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **11** (68.8 mg, 0.4 mmol), **2i** (321.0 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 65% (68.7 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 204-207 °C.

¹**H NMR (400 MHz, CDCl₃)** δ 8.93 (s, 1H), 8.35 (s, 1H), 8.03 – 7.92 (m, 2H), 7.79 (dd, *J* = 9.2, 2.7 Hz, 1H), 7.68 (t, *J* = 7.5 Hz, 1H), 7.59 (t, *J* = 7.5 Hz, 1H), 7.30 (dd, *J* = 9.0, 4.7 Hz, 1H), 7.15 (td, *J* = 9.0, 8.5, 2.8 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 161.26 (d, J = 45.1 Hz), 158.62, 147.33, 136.47, 133.27 (d, J = 29.3 Hz), 130.24, 129.99, 129.20, 128.57, 128.01, 123.87 (d, J = 25.5 Hz), 121.55, 119.91 (dd, J = 16.4, 8.5 Hz), 119.21, 117.72 (d, J = 24.2 Hz), 112.08 (d, J = 24.1 Hz), 109.33 (d, J = 24.9 Hz). ¹⁹F NMR (376 MHz, CDCl₃) δ -113.14.

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}FO_2 [M+H]^+ 265.0659$, found 265.0655.

2-Chloro-6*H*-naphtho[2,3-*c*]chromen-6-one (3ra)



Prepared according to the general procedure 2 on 0.4 mmol scale, the reaction of 11 (68.8 mg, 0.4 mmol),

2j (330.6 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 45% (50.4 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

¹**H NMR (400 MHz, CDCl₃)** δ 9.01 (s, 1H), 8.48 (s, 1H), 8.16 (s, 1H), 8.11 – 7.97 (m, 2H), 7.78 – 7.67 (m, 1H), 7.67 – 7.59 (m, 1H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 1H).

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}CIO_2$ [M+H]⁺ 281.0364, found 281.0359.

Analytical data are consistent with the reported ones.^[5]

2-Bromo-6*H*-naphtho[2,3-*c*]chromen-6-one (3sa)



Prepared according to the *general procedure 2* on 0.4 mmol scale, the reaction of **11** (68.8 mg, 0.4 mmol), **2k** (356.9 mg, 0.6 mmol), K_2CO_3 (110.6 mg, 0.8 mmol), $Pd(OAc)_2$ (9.1 mg, 10 mol%), and DMF (2 mL) at 130 °C for 24 h, and obtained an isolated yield of 53% (68.6 mg) as a white solid after column chromatography (petroleum ether: EtOAc = 10:1) on silica gel.

M.P.: 182-185 °C.

¹**H NMR (400 MHz, CDCl₃)** δ 8.97 (s, 1H), 8.42 (s, 1H), 8.27 (s, 1H), 8.01 (dd, *J* = 12.5, 8.1 Hz, 2H), 7.74 – 7.63 (m, 1H), 7.65 – 7.54 (m, 1H), 7.56 – 7.48 (m, 1H), 7.22 (d, *J* = 8.7 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 161.31, 150.19, 136.56, 133.54, 133.33, 133.18, 130.34, 130.04, 128.63, 128.09, 126.16, 121.52, 120.66, 120.11, 119.28, 118.68, 118.03.

HRMS m/z (ESI-TOF): calculated for $C_{17}H_{10}BrO_2$ [M+H]⁺ 324.9859, found 324.9853.

Part 5. Derivatization of 3aa and 3ka

Methyl 2-(2-methoxyphenyl)-1-naphthoate (4)



To a stirred solution of **3aa**(73.8 mg, 0.3 mmol) in MeCN (3 mL) was added KOH(84.2mg, 1.5 mmol) in one portion, followed by methyl iodide(0.2 ml, 3 mmol). The mixture was stirred at rt for 12 h, and the course of the reaction was followed by TLC until completion. The solution was extracted with EtOAc three times. The combined organic extract was washed with brine, dried over Na_2SO_4 , and concentrated in vacuo. The crude product was purified by flash column chromatography (eluent: petroleum ether/EtOAc = 10/1) on a silicagel to afford the desired product **4** (83.3 mg, 95%) as a yellow oil.^[6]

¹**H NMR (600 MHz, CDCl₃)** δ 8.12 (d, *J* = 8.4 Hz, 1H), 7.96 (d, *J* = 8.5 Hz, 1H), 7.90 (d, *J* = 8.5 Hz, 1H), 7.62 – 7.48 (m, 3H), 7.43 – 7.36 (m, 1H), 7.33 (dd, *J* = 7.5, 1.6 Hz, 1H), 7.12 – 7.04 (m, 1H), 7.01 (d, *J* = 8.2 Hz, 1H), 3.79 (s, 3H), 3.69 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 169.79, 156.73, 135.98, 132.78, 130.97, 130.60, 130.47, 130.21, 129.90, 129.52, 128.88, 128.43, 127.49, 126.52, 125.60, 120.90, 111.15, 55.84, 52.16.
HRMS m/z (EI-TOF): calculated for C₁₉H₁₆O₃ [M]⁺ 292.1099, found 292.1103.

5*H*-naphtho[1,2-*c*]chromene (5)



A cooled solution of **3aa** (73.8 mg, 0.3 mmol) in a mixture of $BF_3 \cdot Et_2O$ (1 mL) and THF (2 mL) was added dropwisely to a suspension of NaBH₄ (34.1 mg, 0.9 mmol) in THF (2 mL) under argon while maintaining the reaction temperature below 10 °C. The reaction mixture was then raised to the reflux temperature, kept under reflux for 6 h. Afterwards, the mixture was concentrated under vacuum, which was purified by flash column chromatography (eluent: petroleum ether/EtOAc = 100/1), thereby affording a white solid **5** (59.8 mg ,86%).^[7,8]

M.P.: 102-105 °C.

¹**H NMR (600 MHz, CDCl₃)** δ 7.73 (d, J = 9.9 Hz, 3H), 7.70 (d, J = 8.4 Hz, 1H), 7.67 (dd, J = 7.7, 1.4 Hz, 1H), 7.44 – 7.39 (m, 1H), 7.39 – 7.32 (m, 1H), 7.17 – 7.12 (m, 1H), 6.96 (td, J = 7.6, 1.1 Hz, 1H), 6.93 (dd, J = 8.0, 0.9 Hz, 1H), 5.53 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 154.68, 133.25, 129.76, 129.36, 129.12, 128.76, 127.45, 127.12, 126.29, 126.09, 123.91, 123.34, 122.65, 122.43, 120.61, 117.33, 65.45.

HRMS m/z (EI-TOF): calculated for $C_{17}H_{12}O [M]^+ 232.0888$, found 232.0886.

9-Phenyl-5*H*-naphtho[1,2-*c*]chromen-5-one (6)



An oven-dried Schlenk tube equipped with a teflon cap and a magnetic stir bar was charged with 3ka

(97.2mg, 0.3mmol), Phenylboric acid (54.8mg, 0.45mmol), Cs_2CO_3 (195.5mg, 0.6mmol) and Pd(PPh_3)₄ (17.3mg, 0.015mmol). The contents were placed under argon with three vacuum/refill cycles, and then DMF (2 mL) was added. The mixture was allowed to be stirred at 120°C for 24 h. After cooling to room temperature, the mixture was quenched with water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated. The crude product was purified by flash chromatography on silica gel to afford the desired product **6** (62.8mg, 65%) as a white solid.

M.P.: 207-210 °C.

¹**H NMR (600 MHz, CDCl₃)** δ 9.79 (d, *J* = 8.7 Hz, 1H), 8.28 (s, 1H), 8.25 – 8.13 (m, 2H), 7.89 (d, *J* = 7.8 Hz, 1H), 7.75 (ddd, *J* = 8.5, 6.9, 1.3 Hz, 1H), 7.71 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.66 (d, *J* = 7.1 Hz, 2H), 7.61 (t, *J* = 7.9 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 2H), 7.44 (dd, *J* = 10.6, 7.9 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 160.18, 150.92, 140.04, 137.49, 136.35, 133.10, 131.87, 129.68, 129.52, 128.90, 128.56, 127.56, 127.16, 127.10, 121.78, 118.56, 118.08, 117.39, 115.08.

HRMS m/z (EI-TOF): calculated for C₂₃H₁₄O₂ [M]⁺ 322.0994, found 322.0991.

Part 6. References

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Part 7. 1H, 13C, and 19F NMR spectra of Products







































































































