Electrochemical-Induced Benzyl C-H Amination towards the

Synthesis of Isoindolinones via Aroyloxy Radical-Mediated

C-H Activation

Mingming Yu,^{†[1]} Yuhan Gao,^{†[1]} Lin Zhang^[2] Yingjie Zhang,^[1] Yiyan Zhang,^[1] Hong Yi,^[1] Zhiliang Huang, *^[1] Aiwen Lei, *^{[1], [2]}

1. College of Chemistry and Molecular Sciences, The Institute for Advanced Studies (IAS), Wuhan University, Wuhan 430072 Hubei, People's Republic of China.

2. National Research Center for Carbohydrate Synthesis Jiangxi Normal University, Nanchang 330022, Jiangxi, P. R. China.

[†]. These authors contributed equally to this work.

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General information

All reactions were run under a nitrogen atmosphere on an undivided cell. All glassware was oven dried at 110 °C for hours and cooled down under vacuum. The instrument for electrolysis was dual display potentiostat (DJS-292B) (made in China). Cyclic voltammograms were obtained on a CHI 605E potentiostat. The anodic electrode was graphite rod (ϕ 6 mm, hard) and cathodic electrode was platinum sheet (1.5 cm×1.5 cm×0.3 mm). These electrodes were commercially available from Gaoss Union China. Unless otherwise noted, all the substrates were obtained from commercial suppliers and used without further purification. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60-90 °C). All new compounds were characterized by ¹H NMR, ¹³C NMR data were recorded with ADVANCE III 400 MHz with tetramethylsilane as an internal standard. High resolution mass spectra (HRMS) were measured with Thermo Fisher Orbitrap Elite mass spectrometer. All chemical shifts (δ) were reported in ppm and coupling constants (*J*) in Hz. All chemical shifts were reported relative to tetramethylsilane (0 ppm for ¹H), CDCl₃ (77.16 ppm for ¹³C).

Experimental Section

1) Experimental procedures

General Procedure for electrochemical oxidative amination of benzylic C-H bonds towards isoindolinones



The synthesis of **3a** is representative: *o*-methyl benzoic acid (**1a**, 0.5 mmol, 1 equiv.), "Bu4NBF₄ (0.5 mmol, 1.0 equiv.), were placed in an oven-dried undivided three-necked bottle. The bottle was equipped with a stir bar, a carbon rods (Φ 6 mm) anode and a platinum plate (15 mm×15 mm×0.3 mm) cathode. The bottle was flushed with nitrogen. Degased dry acetonitrile (MeCN, 10 mL) and commercially 2,2,2-trifluoroethanol (TFE, 1 mL) were added. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA at 40 °C. After completion of the reaction, the product was identified by TLC. The solvent was removed under reduced pressure by an aspirator, then the pure product was obtained by flash column chromatography on silica gel (eluent: petroleum ether/ ethyl acetate = 10:1). It's worth noting that some products yield is moderate or even low, because some side reactions occurred, eg: intramolecular lactonization of *o*-alkyl benzoic acid, *o*-alkyl benzoic acid.

2) Procedure and analytical data of compounds



2-Acetylisoindolin-1-one (3a) following the general procedure, the title compound was obtained as white solid (71mg, 81% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.92 – 7.86 (m, 1H), 7.70 – 7.64 (m, 1H), 7.51 (t, *J* = 7.2 Hz, 2H), 4.79 (s, 2H), 2.67 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 171.16, 167.67, 141.08, 134.09, 131.16, 128.60, 125.12, 123.44, 48.05, 24.84. HRMS (ESI) m/z calcd for C₁₀H₁₀NO₂ (M+H)⁺ : 176.0706, found : 176.0701.



2-Acetyl-4-methylisoindolin-1-one (3b) following the general procedure, the title compound was obtained as white solid (39mg, 41% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 7.1 Hz, 1H), 7.48 – 7.40 (m, 2H), 4.72 (s, 2H), 2.69 (s, 3H), 2.38 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.45, 168.21, 140.14, 134.95, 133.43, 131.02, 129.01, 122.69, 47.45, 24.98, 17.56. **HRMS (ESI)** m/z calcd for C₁₁H₁₂NO₂ (M+H)⁺: 190.0862, found : 190.0862.



2-Acetyl-5-methylisoindolin-1-one (3c) following the general procedure, the title compound was obtained as white solid (49mg, 52% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.78 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 6.8 Hz, 2H), 4.75 (s, 2H), 2.67 (s, 3H), 2.49 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.33, 167.76, 145.38, 141.53, 129.82, 128.63, 125.00, 123.82, 47.93, 24.84, 22.16. **HRMS (ESI)** m/z calcd for C₁₁H₁₂NO₂ (M+H)⁺: 190.0862, found : 190.0857.



2-Acetyl-7-methylisoindolin-1-one (3d) following the general procedure, the title compound was obtained as white solid (62 mg, 65% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.51 (t, *J* = 7.6 Hz, 1H), 7.32 – 7.28 (m, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), 4.74 (s, 2H), 2.70 (s, 3H), 2.67 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 171.33, 167.76, 145.38, 141.53, 129.82, 128.63, 125.00, 123.82, 47.93, 24.84, 22.16. HRMS (ESI) m/z calcd for C₁₁H₁₂NO₂ (M+H)⁺: 190.0862, found : 190.0859.



2-Acetyl-4-chloroisoindolin-1-one (3e) following the general procedure, 15 mA, 4h, the title compound was obtained as white solid (66 mg, 63% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 7.6 Hz, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 1H), 4.80 (s, 2H), 2.69 (s, 3H). ¹³**C NMR** (101

MHz, Chloroform-*d*) δ 171.10, 166.86, 139.46, 134.05, 133.29, 130.42, 130.11, 123.65, 47.42, 25.01. **HRMS (ESI)** m/z calcd for C₁₀H₉Cl₁NO₂ (M+H)⁺: 210.0316, found : 210.0313.



2-Acetyl-5-chloroisoindolin-1-one (3f) following the general procedure, without BF₃•OEt₂, 15 mA, 4 h, the title compound was obtained as white solid (60 mg, 57% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 8.1 Hz, 1H), 7.54 – 7.48 (m, 2H), 4.80 (s, 2H), 2.68 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 171.19, 166.75, 142.67, 140.80, 129.82, 129.59, 126.58, 124.00, 47.79, 24.93. HRMS (ESI) m/z calcd for C₁₀H₉Cl₁NO₂ (M+H)⁺: 210.0316, found : 210.0316.

2-Acetyl-6-chloroisoindolin-1-one (3g) following the general procedure, the title compound was obtained as white solid (61 mg, 58% yield).¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.86 (d, *J* = 1.8 Hz, 1H), 7.63 (dd, *J* = 8.1, 1.9 Hz, 1H), 7.47 (d, *J* = 8.1 Hz, 1H), 4.79 (s, 2H), 2.68 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.12, 166.48, 139.27, 135.05, 134.40, 133.04, 125.18, 124.92, 47.88, 24.95. **HRMS (ESI)** m/z calcd for C₁₀H₉Cl₁NO₂ (M+H)⁺: 210.0316, found : 210.0310.



2-Acetyl-7-chloroisoindolin-1-one (3h) following the general procedure, 15 mA, 4h, benzoic acid (1 equiv.), the title compound was obtained as white solid (52 mg, 50% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.59 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 11.8, 7.9 Hz, 2H), 4.78 (s, 2H), 2.68 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 171.17, 165.37, 143.59, 134.80, 133.19, 130.41, 127.21, 122.05, 47.14, 25.05. HRMS (ESI) m/z calcd for C₁₀H₉Cl₁NO₂ (M+H)⁺ : 210.0316, found : 210.0313.



2-Acetyl-4-bromoisoindolin-1-one (3i) following the general procedure, 15 mA, 4h, the title compound was obtained as white solid (81 mg, 64% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.83 – 7.77 (m, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 4.75 (s, 2H), 2.69 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.15, 166.97, 141.72, 137.08, 133.30, 130.58, 124.23, 118.49, 49.03, 25.07. **HRMS** (**ESI**) m/z calcd for C₁₀H₉Br₁NO₂ (M+H)⁺: 253.9811, found : 253.9810.



2-Acetyl-6-bromoisoindolin-1-one (3j) following the general procedure, the title compound was obtained as white solid (84 mg, 66% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 8.05 – 8.00 (m, 1H), 7.78 (dt, *J* = 8.1, 1.4 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 1H), 4.77 (s, 2H), 2.67 (d, *J* = 1.1 Hz, 3H). ¹³**C NMR**

 $(101 \text{ MHz}, \text{Chloroform-}d) \delta 171.11, 166.33, 139.76, 137.18, 133.32, 128.25, 125.21, 122.73, 47.94, 24.96.$ **HRMS (ESI)** m/z calcd for C₁₀H₉Br₁NO₂ (M+H)⁺: 253.9811, found : 253.9813.



2-Acetyl-7-bromoisoindolin-1-one (3k) following the general procedure, 15 mA, 4h, the title compound was obtained as white solid (59 mg, 46% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 7.4 Hz, 1H), 7.53 – 7.46 (m, 2H), 4.77 (s, 2H), 2.70 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.32, 165.79, 143.91, 134.92, 133.82, 128.67, 122.70, 120.76, 46.96, 25.18. **HRMS (ESI)** m/z calcd for C₁₀H₉Br₁NO₂ (M+H)⁺: 253.9811, found : 253.9810.



2-Acetyl-5-methoxyisoindolin-1-one(3l) following the general procedure, the title compound was obtained as colorless oil (32 mg, 31% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 8.03 – 7.96 (m, 1H), 6.81 – 6.74 (m, 2H), 4.64 (q, *J* = 8.5 Hz, 2H), 3.85 (s, 3H), 2.61 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 165.11, 163.22, 144.25, 133.71, 123.47 (d, *J* = 76.3 Hz), 119.82, 117.31, 111.35, 60.44 (q, *J* = 36.6 Hz), 55.50, 22.57.



2-Acetyl-4-fluoroisoindolin-1-one (3m) following the general procedure, 15 mA, 4h, the title compound was obtained as white solid (74 mg, 77% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 7.6 Hz, 1H), 7.53 (td, *J* = 7.9, 4.6 Hz, 1H), 7.36 (t, *J* = 8.4 Hz, 1H), 4.86 (s, 2H), 2.69 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.13, 166.67, 159.05, 156.55, 134.19 (d, *J* = 4.8 Hz), 130.94 (d, *J* = 6.4 Hz), 127.50 (d, *J* = 18.8 Hz), 121.21 (d, *J* = 4.0 Hz), 120.70 (d, *J* = 19.3 Hz), 45.18, 24.90. ¹⁹**F NMR** (377 MHz, Chloroform-*d*) δ -119.07. **HRMS (ESI)** m/z calcd for C₁₀H₉F₁NO₂ (M+H)⁺: 194.0611, found : 194.0611.



2-Acetyl-5-fluoroisoindolin-1-one (3n) following the general procedure, without BF₃•OEt₂, 15 mA, 4 h, the title compound was obtained as white solid (57 mg, 59% yield). ¹H NMR (400 MHz, Chloroformd) δ 7.92 (s, 1H), 7.21 (d, J = 7.7 Hz, 2H), 4.81 (s, 2H), 2.67 (s, 3H). ¹³C NMR (101 MHz, Chloroformd) δ 171.09, 167.98, 166.63, 165.45, 143.76 (d, J = 10.7 Hz), 127.71 (d, J = 10.2 Hz), 127.40 (d, J = 18.2 Hz), 117.00 (d, J = 23.94 Hz), 110.84 (d, J = 24.2 Hz), 47.86, 24.84. ¹⁹F NMR (377 MHz, Chloroformd) δ -102.69. HRMS (ESI) m/z calcd for C₁₀H₉FNO₂ (M+H)⁺ : 194.0611, found : 194.0610.



2-Acetyl-6-fluoroisoindolin-1-one (30) following the general procedure, the title compound was obtained as white solid (63 mg, 65% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.58 – 7.49 (m, 2H), 7.39 (td, J = 8.6, 2.5 Hz, 1H), 4.79 (s, 2H), 2.68 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.10, 166.76 (d, J = 3.8 Hz), 164.18, 161.70, 136.63 (d, J = 2.4 Hz), 133.22 (d, J = 8.7 Hz), 125.26 (d, J = 8.3 Hz), 122.00 (d, J = 23.9 Hz), 111.57 (d, J = 23.5 Hz), 47.75, 24.89. ¹⁹**F NMR** (377 MHz, Chloroform-*d*) δ -111.89. **HRMS (ESI)** m/z calcd for C₁₀H₉FNO₂ (M+H)⁺: 194.0611, found : 194.0610.



2-Acetyl-7-fluoroisoindolin-1-one (3p) following the general procedure, 15 mA, 4h, the title compound was obtained as white solid (69 mg, 71% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.67 (td, *J* = 7.9, 4.8 Hz, 1H), 7.33 – 7.30 (m, 1H), 7.18 – 7.12 (m, 1H), 4.82 (s, 2H), 2.67 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 171.07, 164.52 (d, *J* = 2.5 Hz), 161.31, 158.69, 143.44 (d, *J* = 2.2 Hz), 136.29 (d, *J* = 8.2 Hz), 119.50 (d, *J* = 4.3 Hz), 118.84 (d, *J* = 12.5 Hz), 115.79 (d, *J* = 18.8 Hz), 47.93, 24.92. ¹⁹F NMR (377 MHz, Chloroform-*d*) δ -115.85. **HRMS (ESI)** m/z calcd for C₁₀H₉FNO₂ (M+H)⁺: 194.0611, found : 194.0612.



2-Acetyl-6-(4-(trifluoromethoxy) phenyl) isoindolin-1-one (3q) following the general procedure, the title compound was obtained as white solid (91 mg, 54% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 8.07 (d, J = 1.3 Hz, 1H), 7.86 (dd, J = 7.9, 1.7 Hz, 1H), 7.66 – 7.58 (m, 3H), 7.33 (d, J = 8.1 Hz, 2H), 4.85 (s, 2H), 2.70 (s, 3H). ¹³**C NMR** (101 MHz, CDCl₃) δ 171.28, 167.58, 149.25, 140.77, 140.34, 138.34, 133.11, 132.14, 128.68, 124.10, 123.41, 121.56, 120.536 (q, J = 258.46 Hz), 48.03, 24.95. ¹⁹**F NMR** (377 MHz, CDCl₃) δ -57.80. **HRMS (ESI)** m/z calcd for C₁₇H₁₃F₃NO₃ (M+H)⁺: 336.0842, found : 336.0846.



2-Acetyl-6-(4-(trifluoromethyl) phenyl) isoindolin-1-one (3r) following the general procedure, the title compound was obtained as white solid (86 mg, 54% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 8.11 (s, 1H), 7.91 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.73 (s, 4H), 7.63 (d, *J* = 7.9 Hz, 1H), 4.87 (s, 2H), 2.70 (s, 3H). ¹³**C NMR** (101 MHz, CDCl₃) δ 171.24, 167.47, 143.11, 140.84, 140.69, 133.23, 132.25, 130.19 (q, *J* = 32.7 Hz), 127.61, 126.09 (q, *J* = 37.4 Hz), 124.24 (q, *J* = 273.0 Hz), 124.17, 123.67, 48.06, 24.96. ¹⁹**F NMR** (377 MHz, CDCl₃) δ -62.49. **HRMS (ESI)** m/z calcd for C₁₇H₁₃F₃NO₂ (M+H)⁺: 320.0892, found : 321.3142.

F₃C

5-Acetyl-5,6-dihydro-4H-thieno[2,3-c] pyrrol-4-one (3s) following the general procedure, the title compound was obtained as white solid (32 mg, 35% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.42 (d, *J* = 4.2 Hz, 1H), 7.28 (d, *J* = 5.7 Hz, 2H), 4.88 (s, 2H), 2.64 (s, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 170.91, 163.38, 152.80, 138.99, 131.25, 121.01, 47.11, 24.63. **HRMS (ESI)** m/z calcd for C₈H₈NO₂S (M+H)⁺: 182.0270, found : 182.0267.



2-Acetyl-3-methylisoindolin-1-one (3t) following the general procedure, without BF₃•OEt₂, 15 mA, 4 h, the title compound was obtained as white solid (50 mg, 53% yield). ¹H NMR (400 MHz, Chloroformd) δ 7.90 (d, J = 7.7 Hz, 1H), 7.68 (td, J = 7.5, 1.1 Hz, 1H), 7.53 – 7.46 (m, 2H), 5.21 (q, J = 6.5 Hz, 1H), 2.68 (s, 3H), 1.62 (d, J = 6.5 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 171.28, 167.94, 147.72, 134.35, 129.94, 128.75, 125.26, 122.67, 55.79, 25.75, 20.20. HRMS (ESI) m/z calcd for C₁₁H₁₂NO₂ (M+H)⁺ : 190.0862, found : 190.0860.



2-Acetyl-3-ethylisoindolin-1-one (3u) following the general procedure, the title compound was obtained as colorless oil (62 mg, 61% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.90 (dt, *J* = 7.7, 1.0 Hz, 1H), 7.67 (td, *J* = 7.5, 1.2 Hz, 1H), 7.54 – 7.44 (m, 2H), 5.29 (dd, *J* = 5.8, 2.8 Hz, 1H), 2.69 (s, 3H), 2.37 (dtd, *J* = 14.8, 7.4, 5.8 Hz, 1H), 2.10 – 1.99 (m, 1H), 0.55 (t, *J* = 7.5 Hz, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.29, 168.54, 145.88, 134.28, 130.99, 128.76, 125.18, 122.82, 60.04, 25.67, 24.80, 6.75. **HRMS (ESI)** m/z calcd for C₁₂H₁₄NO₂ (M+H)⁺ : 204.1019, found : 204.1019.



2-Acetyl-3-propylisoindolin-1-one (**3v**) following the general procedure, the title compound was obtained as colorless oil (60 mg, 55% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 7.6 Hz, 1H), 7.67 (td, *J* = 7.5, 1.2 Hz, 1H), 7.53 – 7.45 (m, 2H), 5.28 (dd, *J* = 6.1, 2.9 Hz, 1H), 2.68 (s, 3H), 2.29 – 2.20 (m, 1H), 1.99 (tdd, *J* = 11.6, 4.8, 2.4 Hz, 1H), 1.16 – 1.07 (m, 1H), 0.94 – 0.76 (m, 5H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.29, 168.43, 146.36, 134.24, 130.79, 128.72, 125.23, 122.89, 59.50, 34.19, 25.72, 16.21, 14.02. **HRMS (ESI)** m/z calcd for C₁₃H₁₆NO₂ (M+H)⁺ : 218.1175, found : 218.1175.



2-Acetyl-3-butylisoindolin-1-one (**3w**) following the general procedure, the title compound was obtained as colorless oil (67 mg, 58% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.90 (dt, *J* = 7.7, 1.1

Hz, 1H), 7.67 (td, J = 7.5, 1.2 Hz, 1H), 7.54 – 7.44 (m, 2H), 5.28 (dd, J = 6.2, 2.8 Hz, 1H), 2.68 (s, 3H), 2.33 – 2.19 (m, 1H), 2.02 (dddd, J = 14.1, 11.7, 4.8, 2.9 Hz, 1H), 1.25 – 1.15 (m, 2H), 1.10 – 0.98 (m, 1H), 0.79 (t, J = 7.3 Hz, 4H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.29, 168.47, 146.35, 134.25, 130.82, 128.72, 125.22, 122.87, 59.52, 31.74, 25.72, 24.89, 22.61, 14.01. **HRMS (ESI)** m/z calcd for C₁₄H₁₈NO₂ (M+H)⁺ : 232.1332, found : 232.1332.



2-Acetyl-3-isobutylisoindolin-1-one (3x) following the general procedure, the title compound was obtained as colorless oil (59 mg, 51% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.90 (dt, *J* = 7.6, 1.0 Hz, 1H), 7.66 (td, *J* = 7.5, 1.2 Hz, 1H), 7.55 – 7.46 (m, 2H), 5.27 (dd, *J* = 7.7, 3.4 Hz, 1H), 2.67 (d, *J* = 0.8 Hz, 3H), 2.03 (ddd, *J* = 13.8, 8.4, 3.4 Hz, 1H), 1.83 (ddd, *J* = 13.6, 7.7, 5.5 Hz, 1H), 0.88 (dd, *J* = 11.3, 6.6 Hz, 6H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 171.22, 168.27, 146.85, 134.08, 130.59, 128.71, 125.40, 123.26, 58.58, 41.69, 25.81, 24.49, 23.92, 22.66. **HRMS (ESI)** m/z calcd for C₁₄H₁₈NO₂ (M+H)⁺ : 232.1332, found : 232.1333.



1-Acetyl-6,7,8,8a-tetrahydrobenzo[cd]indol-2(1H)-one (3y) following the general procedure, without BF₃•OEt₂, the title compound was obtained as white solid (38 mg, 35% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 6.8 Hz, 1H), 7.42 (q, *J* = 7.7 Hz, 2H), 4.80 (dd, *J* = 11.4, 4.7 Hz, 1H), 3.04 (ddd, *J* = 17.6, 8.1, 3.1 Hz, 1H), 2.94 – 2.87 (m, 1H), 2.77 (dt, *J* = 17.4, 8.4 Hz, 1H), 2.66 (s, 3H), 2.16 – 1.99 (m, 2H), 1.13 (qd, *J* = 11.7, 5.4 Hz, 1H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 172.48, 168.60, 144.61, 134.98, 132.90, 129.39, 128.57, 122.19, 57.58, 26.75, 25.58, 25.16, 20.54. **HRMS (ESI)** m/z calcd for C₁₃H₁₄NO₂ (M+H)⁺: 216.1019, found : 216.1018.



2-Butyrylisoindolin-1-one (3z) following the general procedure, 80 min, the title compound was obtained as white solid (7.1 mg, 35% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.94 – 7.88 (m, 1H), 7.67 (td, *J* = 7.5, 1.2 Hz, 1H), 7.54 – 7.47 (m, 2H), 4.82 (s, 2H), 3.08 (t, *J* = 7.4 Hz, 2H), 1.78 (h, *J* = 7.4 Hz, 2H), 1.04 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 174.44, 167.76, 141.30, 134.13, 131.60, 128.72, 125.28, 123.55, 48.33, 38.92, 17.91, 13.96. HRMS (ESI) m/z calcd for C₁₂H₁₄NO₂ (M+H)⁺ : 204.1019, found : 204.1019.



2-Isobutyrylisoindolin-1-one (3aa) following the general procedure, the title compound was obtained as white solid (45 mg, 44% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.92 (d, *J* = 8.2 Hz, 1H), 7.67

(td, J = 7.5, 1.1 Hz, 1H), 7.55 - 7.49 (m, 2H), 4.83 (s, 2H), 3.96 (hept, J = 6.8 Hz, 1H), 1.27 (s, 3H), 1.25 (s, 3H).¹³C NMR (101 MHz, Chloroform-*d*) δ 178.83, 167.39, 141.31, 134.12, 131.67, 128.69, 125.30, 123.50, 48.69, 34.09, 18.97. **HRMS (ESI)** m/z calcd for C₁₂H₁₄NO₂ (M+H)⁺ : 204.1019, found : 204.1017.



2-Pentanoylisoindolin-1-one (3ab) following the general procedure, 80 min, the title compound was obtained as white solid (8.5 mg, 39% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.93 – 7.88 (m, 1H), 7.67 (td, *J* = 7.5, 1.2 Hz, 1H), 7.54 – 7.48 (m, 2H), 4.82 (s, 2H), 3.13 – 3.06 (m, 2H), 1.73 (p, *J* = 7.6 Hz, 2H), 1.45 (h, *J* = 7.3 Hz, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 174.64, 167.76, 141.31, 134.13, 131.61, 128.72, 125.28, 123.55, 48.37, 36.79, 26.58, 22.54, 14.03. **HRMS (ESI)** m/z calcd for C₁₃H₁₆NO₂ (M+H)⁺ : 218.1175, found : 218.1176.



2-(Cyclopropanecarbonyl) isoindolin-1-one (3ac) following the general procedure, 80 min, the title compound was obtained as white solid (12.0 mg, 60% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.96 – 7.91 (m, 1H), 7.67 (td, *J* = 7.4, 1.2 Hz, 1H), 7.52 (t, *J* = 7.1 Hz, 2H), 4.83 (s, 2H), 3.49 (tt, *J* = 7.9, 4.7 Hz, 1H), 1.22 (dt, *J* = 4.6, 3.3 Hz, 2H), 1.05 (dq, *J* = 7.3, 3.7 Hz, 2H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 175.57, 168.23, 141.09, 134.16, 131.79, 128.69, 125.36, 123.46, 48.81, 13.74, 10.79. **HRMS (ESI)** m/z calcd for C₁₂H₁₂NO₂ (M+H)⁺ : 202.0862, found : 202.0867.



2-(Acetyl-d3) isoindolin-1-one (3ad) following the general procedure, 80min, the title compound was obtained as white solid (8.2 mg, 46% yield). ¹**H NMR** (400 MHz, Chloroform-*d*) δ 7.94 – 7.90 (m, 1H), 7.68 (td, *J* = 7.5, 1.2 Hz, 1H), 7.52 (dt, *J* = 7.6, 3.5 Hz, 2H), 4.82 (s, 2H). ¹³**C NMR** (101 MHz, CDCl₃) δ 171.44, 167.89, 141.22, 134.25, 131.33, 128.77, 125.33, 123.58, 48.17, 29.82. **HRMS (ESI)** m/z calcd for C₁₀H₇NO₂D₃(M+H)⁺: 179.0894, found : 179.0892.

3) Substrates synthesis

General Procedure A for substrate Syntheses:

To a solution of 13 (10 mmol) in THF (30 mL) was added *n*-BuLi (1.54 M in hexane, 15 mmol) at -78 °C. After stirring for 0.5 h at the same temperature, anhydrous CO_2 was bubbled through the mixture for 1 h. Then the mixture was stirred for an additional 20 min at room temperature, quenched with aqueous solution of saturated NaHCO₃, and washed with Et₂O. Aqueous layer was acidified with aqueous solution of 10% HCl and extracted with Et₂O. The extract was dried and concentrated to dryness to afford the

product as a white solid¹; ¹**H NMR** (400 MHz, Chloroform-d) δ 8.05 (dd, J = 7.8, 1.5 Hz, 1H), 7.48 (td, J = 7.5, 1.5 Hz, 1H), 7.29 (ddd, J = 15.0, 7.5, 1.3 Hz, 2H), 3.07 (q, J = 7.5 Hz, 2H), 1.27 (t, J = 7.5 Hz, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 173.81, 147.43, 133.20, 131.78, 130.62, 128.12, 125.96, 27.89, 16.05.

General Procedure B for substrate Syntheses:



A solution of potassium tert-butoxide (20 mmol) in THF (10 mL) was added dropwise to a solution of R_1PPh_3Br bromide (14 mmol) in THF (20 mL) at room temperature. The reaction mixture was stirred for 90 min, before dropwise addition of 2-carboxybenzaldehyde (8.7 mmol). When the addition was complete, the reaction temperature was raised to 60 °C and stirring was continued overnight. After addition of acetic acid (0.5 mL), the reaction mixture was filtered through a pad of celite and concentrated by rotary evaporation. The residue was taken up in ethyl acetate and washed thoroughly with sat. NaHCO₃ (aq). The combined aqueous washings were acidified with 1.0 M HCl (aq), then back-extracted with ethyl acetate. The organic layer was washed with water and brine, then dried over MgSO₄, filtered, and rotary evaporated . ²

A flame dried Schlenk tube equipped with a stirrer bar was charged with the purified compound from the previous step, Pd/C (20 w%) and ethanol (5 mL/mmol) were added and the reaction was stirred under atmospheric pressure of hydrogen gas overnight. The mixture was filtered through Celite to afford the product.³



2-Propylbenzoic acid This compound was following general procedure B. ¹H NMR (400 MHz, Chloroform-d) δ 8.04 (dt, J = 8.0, 1.3 Hz, 1H), 7.47 (td, J = 7.5, 1.5 Hz, 1H), 7.28 (ddd, J = 7.2, 4.0, 2.6 Hz, 2H), 3.06 – 2.98 (m, 2H), 1.73 – 1.61 (m, 2H), 0.99 (t, J = 7.3 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 173.39, 145.82, 132.89, 131.75, 131.39, 128.33, 125.99, 36.70, 25.03, 14.32.



2-Butylbenzoic acid This compound was following general procedure B. ¹**H** NMR (400 MHz, Chloroform-*d*) δ 8.08 – 8.01 (m, 1H), 7.46 (td, *J* = 7.5, 1.5 Hz, 1H), 7.27 (dd, *J* = 7.8, 6.5 Hz, 2H), 3.08 – 2.98 (m, 2H), 1.62 (tt, *J* = 7.9, 6.4 Hz, 2H), 1.41 (h, *J* = 7.3 Hz, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). ¹³**C** NMR (101 MHz, Chloroform-*d*) δ 173.85, 146.13, 132.94, 131.78, 131.36, 128.30, 125.93, 34.46, 34.10, 22.91, 14.05.



2-Pentylbenzoic acid This compound was following general procedure B. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 (dd, J = 8.3, 1.5 Hz, 1H), 7.47 (td, J = 7.5, 1.5 Hz, 1H), 7.29 (dd, J = 7.6, 1.4 Hz,

2H), 3.08 – 2.97 (m, 2H), 1.68 – 1.58 (m, 2H), 1.37 (tt, *J* = 6.6, 2.6 Hz, 4H), 0.94 – 0.86 (m, 3H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 173.46, 146.16, 132.94, 131.75, 131.36, 128.25, 125.94, 34.72, 32.06, 31.61, 22.61, 14.19.



2-Isopentylbenzoic acid This compound was following general procedure B. ¹**H NMR** (400 MHz, Chloroform-*d*) δ 10.90 (s, 1H), 8.07 – 8.02 (m, 1H), 7.49 – 7.42 (m, 1H), 7.27 (dd, *J* = 8.1, 6.8 Hz, 2H), 3.06 – 2.99 (m, 2H), 1.74 – 1.59 (m, *J* = 6.6 Hz, 1H), 1.55 – 1.47 (m, 2H), 0.97 (d, *J* = 6.6 Hz, 6H). ¹³**C NMR** (101 MHz, Chloroform-*d*) δ 173.80, 146.48, 133.01, 131.81, 131.34, 128.24, 125.90, 41.34, 32.85, 28.51, 22.63.

4) Application of the products

Synthesis of 3ae



Compound **3a** (1 mmol) was diluted in 4 M NaOH (5 mL) and MeOH (5 ml). The mixture was heated to 50 °C with stirring for 4 h. The solution was cooled and washed with dichloromethane (3 × 10 mL). The organic phase was concentrated to give **3ae** as a white solid (93%).⁴ ¹**H** NMR (400 MHz, Chloroform-*d*) δ 7.91 – 7.86 (m, 1H), 7.77 (d, *J* = 11.1 Hz, 1H), 7.58 (td, *J* = 7.4, 1.2 Hz, 1H), 7.52 – 7.46 (m, 2H), 4.48 (s, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 172.26, 143.80, 132.27, 131.86, 128.13, 123.85, 123.30, 45.86. **HRMS (ESI)** m/z calcd for C₈H₈NO (M+H)⁺ : 134.0600, found : 134.0600. **Synthesis of 3af**



To a stirred solution of **3ae** (1.0 mmol) in anhydrous THF (4 mL) at -78 °C was slowly added a solution of *n*-BuLi (2.4 M solution in hexanes, 1.1 mmol). The resulting reaction mixture was stirred for 15 minutes at the same temperature, time after which a solution of TsCl (1.1 mmol) was added and stirring was continued at room temperature overnight. H₂O was eventually added, and the aqueous phase was extracted with EtOAc (3×10 mL). The combined organic phases were then washed with a saturated aqueous solution of brine (10 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel to afford the desired product **3af** as a white solid in 44% yield.⁵ **¹H** NMR (400 MHz, Chloroform-d) δ 8.06 – 7.99 (m, 2H), 7.80 (dt, J = 7.5, 1.1 Hz, 1H), 7.63 (td, J = 7.5, 1.2 Hz, 1H), 7.47 (td, J = 7.8, 1.0 Hz, 2H), 7.33 (d, J = 8.2 Hz, 2H), 4.91 (s, 2H), 2.41 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 166.21, 145.34, 141.12, 135.49, 133.97, 130.29, 129.86, 128.91, 128.22, 125.11, 123.46, 49.95, 21.76. HRMS (ESI) m/z calcd for C₁₅H₁₄NO₃S (M+H)⁺ : 288.0688, found : 288.0692.

Synthesis of 3ag



Following a previously reported procedure, A 40-mL oven-dried scintillation vial equipped with a magnetic stir bar was charged with **3ae** (1.5 mmol), CuI (5 mol%) and K₂CO₃ (3 mmol). The vial was transferred to a glovebox filled with N₂, wherein DMEDA (10 mol%), vinyl bromide (2.25 mmol) and anhydrous toluene (2 mL) were added. The vial was tightly capped with a PTFE-lined green cap, removed from the glovebox, and stirred at 110 °C for 12 h. The crude reaction mixture was concentrated in vacuo. The crude residue was finally purified by flash column chromatography over silica gel to afford **3ag** in 85% yield.⁶ ¹**H** NMR (400 MHz, Chloroform-*d*) δ 7.77 (dd, *J* = 7.5, 1.4 Hz, 1H), 7.51 (td, *J* = 7.5, 1.2 Hz, 1H), 7.44 – 7.37 (m, 2H), 4.80 (s, 1H), 4.52 (s, 2H), 4.43 (d, *J* = 1.4 Hz, 1H), 2.34 (d, *J* = 1.4 Hz, 3H). ¹³**C** NMR (101 MHz, Chloroform-*d*) δ 167.10, 141.57, 139.90, 132.96, 131.85, 127.96, 123.62, 122.34, 97.89, 50.46, 21.07. **HRMS (ESI)** m/z calcd for C₁₁H₁₂NO (M+H)⁺ : 174.0913, found : 174.0909. **Synthesis of 3ah**



In an oven-dried undivided three-necked flask (25 mL) equipped with a stir bar, **3ae** (0.50 mmol), aniline (1.0 mmol), Cp₂Fe (20 mol%), "Bu₄NBF₄(1.0 mmol) and CH₃CN/AcOH (8/1 mL) were combined and added. The bottle was equipped with graphite electrode as the anode and platinum electrode ($1.5 \times 1.5 \times 0.3$ cm) as the cathode and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA under 60 °C for 3 h. When the reaction was finished, the solution was removed with a rotary evaporator. The pure product **3ah** was obtained by flash chromatography on silica gel using petroleum ether and ethyl acetate as the eluent as a white solid in 60% yield.⁷ **H NMR** (400 MHz, Chloroform-*d*) δ 7.84 (dt, *J* = 7.4, 1.1 Hz, 1H), 7.48 (td, *J* = 7.4, 1.3 Hz, 1H), 7.41 (td, *J* = 7.5, 1.2 Hz, 1H), 7.33 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.07 (dd, *J* = 7.5, 1.4 Hz, 2H), 6.85 – 6.80 (m, 2H), 5.18 (s, 2H), 4.22 (s, 2H), 2.99 (s, 3H), 2.25 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 168.81, 145.91, 141.29, 132.14, 131.56, 129.92, 127.97, 127.43, 123.79, 122.83, 113.39, 60.02, 49.41, 37.89, 20.28. HRMS (ESI) m/z calcd for C₁₇H₁₉N₂O₁(M+H)+ : 267.1491, found : 267.1488. **Synthesis of 3ai**



Isoindolin-1-one **3ae** (0.50 mmol) was dissolved in super-dry DMSO (2 mL), and Cs₂CO₃, (1.25 mmol), CuI (0.10 mmol) and N', N-dimethylethane-1,2-diamine (0.10 mmol) were added to the solution. The resulting mixture was stirred at room temperature for 10 min, then 4-chloroiodobenzene (0.75 mmol) was added. Finally, the mixture was heated to 120 °C for 3 hours $_{\circ}$ The reaction mixture was extracted with ethyl acetate (20 mL) and H₂O (10 mL). The organic layer was combined and washed with brine (10 mL). Then the solution was dried over anhydrous NaSO₄, filtered and concentrated, and the crude

residue was purified by flash chromatography over silica gel to afford the title compound **3ai** as a yellow solid in 80% yield.⁸ ¹**H NMR** (400 MHz, Chloroform-d) δ 7.88 (d, J = 7.7 Hz, 1H), 7.83 – 7.78 (m, 2H), 7.59 (t, J = 7.4 Hz, 1H), 7.49 (d, J = 7.1 Hz, 2H), 7.34 (dd, J = 8.8, 1.4 Hz, 2H), 4.78 (s, 2H). ¹³**C NMR** (101 MHz, CDCl₃) δ 167.60, 139.94, 138.13, 132.91, 132.40, 129.52, 129.20, 128.57, 124.21, 122.74, 120.39, 50.68. **HRMS (ESI)** m/z calcd for C₁₄H₁₁ClNO (M+H)⁺ : 244.0523, found : 244.0528. **Synthesis of 3aj**



Compound **3ae** (1 mmol) was diluted in 6 M HCl (10 mL) and heated to reflux with stirring overnight. The solution was cooled and washed with dichloromethane (3×10 mL). The aqueous phase was concentrated to give **3aj** as a white solid in 80% yield.⁴

5) Cyclic voltammetry (CV) experiments



Figure. S1a) Cyclic voltammetry of 1a-Na.



Figure. S1b) Cyclic voltammetry of 1a.

General procedure for cyclic voltammetry (CV): Cyclic voltammetry was performed in a three electrodes cell connected to a Schlenk line under N₂ at room temperature. The working electrode was a glassy carbon electrode, the counter electrode a platinum wire. The reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution, and separated from reaction by a salt bridge. **Figure. S1a** :10 mL of CH₃CN and 1 mL MeOH containing 1 mmol "Bu₄NBF₄ were poured into the electrochemical cell in all experiments. The scan rate is 0.1 V/s, ranging from 0 V to 2.0 V for **1a-Na**. **Figure. S1b** :10 mL of CH₃CN and 1 mL TFE containing 1mmol "Bu₄NBF₄ were poured into the electro chemical cell in all experiments. The scan rate is 0.1 V/s, ranging from 0 V to 3.0 V for **1a**.

6) General procedure for constant voltage reaction:

In an oven-dried undivided three-necked flask equipped with a stir bar, **1a** (0.50 mmol), PhCOOH (0.5 equiv.), "Bu₄NBF₄ (0.3 equiv.), BF₃•OEt₂ and CH₃CN/TFE (10/1 mL) were combined and added. The bottle was equipped with graphite electrode as the anode, platinum electrode ($1.5 \times 1.5 \times 0.03 \text{ cm}^3$) as the cathode and Ag/AgCl as the reference electrode. The reaction mixture was stirred and electrolyzed at a constant voltage 1.6 V under 40 °C. When TLC showed that **1a** had been fully converted, the reaction was stopped, and the solvent was removed with a rotary evaporator. The crude residue was purified by flash chromatography on silica gel using petroleum ether and ethyl acetate as the eluent (10:1) to afford the title compound **3a** in 52% yield.

7) Gram-scale synthesis

Typical procedure for gram-scale synthesis for 1a:



A mixture of **1a** (10 mmol), PhCOOH (0.5 equiv.), "Bu₄NBF₄ (0.6 equiv.), CH₃CN (50 mL)/TFE (5 mL), in an undivided cell at nitrogen atmosphere with carbon electrode anode, plate cathode, constant current= 10 mA, r.t., 80 h. Then, the system was concentrated under reduced pressure. The resulting crude product was separated on a silica gel column with petroleum ether and ethyl acetate as eluent to afford the desired product.

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NMR Spectra of Products



¹H NMR CDCl₃ Yuhan Gao 202104/gyh-1-212-3







 $^{1}\mathrm{H}\ \mathrm{NMR}\ \mathrm{CDCl}_{3}$ Yuhan Gao 202105/gyh-1-217-4



¹H NMR CDCl₃ Yuhan Gao 202105/gyh-1-217-2



$$-4.739$$

 $^{1}\mathrm{H}$ NMR CDCl_{3} Yuhan Gao 202105/gyh-1-217-3



 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202105/gyh-1-217-3



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¹H NMR CDCl₃ Yuhan Gao 202105/gyh-1-235-3



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 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202105/gyh-1-235-3





¹H NMR CDCl₃ Yuhan Gao 202105/gyh-1-240-1



 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202105/gyh-1-240-1



¹H NMR CDCl₃ Yuhan Gao 202105/gyh-1-232-1



 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202105/gyh-1-232-1





¹H NMR CDCl₃ Yuhan Gao 202105/gyh-1-239-3



¹H NMR CDCl₃ Yuhan Gao 202105/gyh-1-233-1



 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202105/gyh-1-233-1



 $^{1}\mathrm{H}$ NMR CDCl_{3} Yuhan Gao 202105/gyh-1-238-3



 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202105/gyh-1-238-3



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 $^{1}\mathrm{H}$ NMR CDCl_{3} Yuhan Gao 202108/gyh-2-48-2



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 $^1\mathrm{H}$ NMR CDCl_3 Yuhan Gao 202105/gyh-1-236-3



¹³C NMR CDCl₃ Yuhan Gao 202105/gyh-1-236-3



¹⁹F NMR CDCl₃ Yuhan Gao 202105/gyh-1-236-3





10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm)



171.095 166.779 166.741 164.176 161.703	136.645 136.621 133.266 133.180 125.304 122.118 122.118 122.118 111.450 111.450	47.754	24.891
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$^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202107/gyh-2-27-4





S34

171.068 164.536 164.511 161.310 158.690	143.447 143.425 136.327 136.246	119.522 119.479 118.904 118.780 115.880 115.694 115.694	47.929	24.924
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 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202105/gyh-1-231-4



10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm)

8.070 8.070 7.875 7.871 7.851 7.855 7.647 7.642 7.630 7.630 7.630 7.638 7.638 7.638 7.638 7.589 7.589 7.539 7.539 7.539	4.849	2.696
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¹H NMR CDCl₃ Yuhan Gao 202106/gyh-2-8-1



 ^{19}F NMR CDCl₃ Yuhan Gao 202106/gyh-2-8-1





 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202106/gyh-2-5-1



10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 f1 (ppm)

$$\mathcal{L}_{7.271}^{7.426}$$

$$\mathcal{L}_{7.271}^{7.426}$$

¹H NMR CDCl₃ Yuhan Gao 202106/gyh-1-252-1



7.905 7.005 7.708 7.698 7.681 7.663 7.663 7.663 7.663 7.663 7.663 7.683 7.683 7.487 7.487 7.487 7.487 7.487 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.485 7.565 7.663 7.664 7.764 7.664 7.7647 7.7647 7.7647 7.7647 7.7647 7.7647 7.7647 7.7647 7.7647 7.7	5.238 5.222 5.189 5.189	2.679	1.626 1.610
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¹H NMR CDCl₃ Yuhan Gao 202106/gyh-2-3-1



¹H NMR CDCl₃ Yuhan Gao 202106/gyh-2-3-1



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7,902 7,7647 7,7687 7,7687 7,7687 7,7648 7,7648 7,7549 7,7549 7,7549 7,7549 7,7552 2,5274 2,



¹H NMR CDCl₃ Yuhan Gao 202108/gyh-2-74-1





7,915 7,7915 7,7575 7,7575 7,7575 7,7575 7,7575 7,7557 7,7557 7,7557 7,75527 7,75527 7,75527 7,75527 7,75527 7,75527 7,75527 7,75527 7,75527 7

¹H NMR CDCl₃ Yuhan Gao 202108/gyh-2-80-1



7.675 7.675 7.658 7.658 7.7482 7.7482 7.7482 7.7482 3.3029 3.3029 3.3025 3.3029 3.3009 3.300





7.927 7.907 7.691 7.692 7.653 7.653 7.653 7.653 7.563 7.563 7.563 7.553 7.553 7.553 7.553 7.553 7.553 7.7515 7.7515	-4.828 -4.828 -4.010 -4.010 -4.010 -4.010 -4.010 -4.028 -4.010 -4.028 $-$	$<^{1.271}_{1.254}$
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¹H NMR CDCl₃ Yuhan Gao 202106/gyh-1-257-1





¹H NMR CDCl₃ Yuhan Gao 202108/gyh-2-69-1





-3.123-3.123-3.104-3.104-1.765-1.741-1.741-1.741-1.741-1.741-1.732-1.732-1.732-1.732-1.732-1.732-1.732-1.741-1.732-1.741-1.742-1.445-1.4555-1.4555-1.4555-1.4555-1.4555-1.4555-1.4

¹H NMR CDCl₃ Yuhan Gao 202108/gyh-2-68-1





¹H NMR CDCl₃ Yuhan Gao 202108/gyh-2-69-3



7.532 7.911 7.701 7.701 7.678 7.678 7.678 7.658 7.550 7.550 7.550 7.553 7.553 7.553 7.553 7.553 7.553	4.823
	1

¹H NMR CDCl₃ Yuhan Gao 202107/gyh-2-33-2



¹H NMR CDCl₃ Yuhan Gao 202106/gyh-1-264



173.813	147.432	133.199 131.778 130.617 128.120 125.956	27.888	16.051
I	I.	\sim	I	I

¹³C NMR CDCl₃ Yuhan Gao 202106/gyh-1-264



$\begin{array}{c} 8.054\\ 8.037\\ 8.037\\ 8.037\\ 8.038\\ 8.038\\ 8.036\\ 8.035\\ 8.036\\ 8.036\\ 8.036\\ 8.036\\ 8.036\\ 8.036\\ 8.036\\ 8.036\\ 7.730\\ 7.730\\ 7.730\\ 7.730\\ 7.723\\ 3.030\\ 7.720\\ 7.723\\ 3.010\\ 7.7233\\ 3.010\\ 7.7233\\ 7.7233\\ 7.7233\\ 7.7233\\ 7.7233\\ 7.7233\\ 7.7233\\ 7.723\\ 7.7233\\$

¹H NMR CDCl₃ Yuhan Gao 202108/gyh-2-70-1



8.056 8.051 8.051 8.033 8.034 8.034 7.479 7.446 7.446 7.446 7.446 7.445 7.445 7.445 7.445 7.745 7.748 7.7266 7.7266 7.7266

3.057 3.037 3.037 3.037 3.037 3.037 3.037 3.037 1.642 1.648 1.6333 1.6333 1.6333 1.6333 1.6333 1.6333 1.6333 1.6333 1.6333 1.6333 1.6333 1.6333 1.6333 1.633

¹H NMR CDCl₃ Yuhan Gao 202108/gyh-2-72-3



 $^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202108/gyh-2-72-3



¹H NMR CDCl₃ Yuhan Gao 202108/gyh-2-72-4



¹H NMR CDCl₃ Yuhan Gao 202109/gyh-2-77-1

- 10.900





¹H NMR CDCl₃ Yuhan Gao 202106/gyh-2-9-1



¹H NMR CDCl₃ Yuhan Gao 202107/gyh-2-43-1



7.783 7.761 7.751 7.751 7.522 7.520 7.520 7.512	∠ 4.795 ∠ 4.520 ∠ 4.436 4.432	2.346

¹H NMR CDCl₃ Yuhan Gao 202107/gyh-2-37-2



¹³C NMR CDCl₃ Yuhan Gao 202107/gyh-2-37-2



7,852 7,894 7,814 7,828 7,833 7,838 7,445 7,4467 7,4467 7,4467 7,4467 7,4467777777777	- 2.988	- 2.245

¹H NMR CDCl₃ Yuhan Gao 202107/gyh-2-18-2



168.810	145.914 141.291 132.141 131.560 127.429 127.429 127.429 122.826 113.390	60.025	49.406	37.894	20.278
1		1		1	1

¹³C NMR CDCl₃ Yuhan Gao 202107/gyh-2-18-2



 $^{1}\mathrm{H}$ NMR CDCl_{3} Yuhan Gao 202108/gyh-2-60-2



167.602	139,943 138,131 132,912 132,401 132,523 129,523 129,524 129,524 122,740 122,740 122,740 122,740 122,740 120,392	50.678
1		1

$^{13}\mathrm{C}\ \mathrm{NMR}\ \mathrm{CDCl}_3$ Yuhan Gao 202108/gyh-2-60-2

