

## Supporting Information

### **Mn-Mediated Electrochemical Trifluoromethylation/C(sp<sup>2</sup>)-H**

### **Functionalization Cascade for the Synthesis of Azaheterocycles**

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## 1. General information

Unless otherwise noted, chemicals and solvents were purchased with the highest purity grade available and were used without further purification. Purification of products was conducted by column chromatography on silica gel (200-300 mesh, from Qingdao, China). NMR spectra were measured on a Bruker ARX400 ( $^1\text{H}$  at 400 MHz,  $^{13}\text{C}$  at 101 MHz,  $^{19}\text{F}$  at 471 MHz) magnetic resonance spectrometer. Chemical shifts ( $\delta$ ) are reported in ppm using tetramethylsilane as internal standard (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, dt = doublet of triplets, ddd = doublet of doublet of doublets, m = multiplet), and coupling constants ( $J$ ) were reported in Hertz (Hz). Infrared spectra were recorded on a Thermal Fisher Nicolet iS50 Fourier transform spectrometer (FT-IR) and were reported in wave numbers ( $\text{cm}^{-1}$ ). HRMS data were obtained on a VG ZAB-HS mass spectrometer, Bruker Apex IV FTMS spectrometer.

Materials: Pt electrodes were purchased from Tianjin Aida Corp. The potentiostat was purchased from JD.com, ITECH IT6720. Gram-scale reaction was carried out by using IKA Electrasyn 2.0.

The starting materials **1** were synthesized as reported by Liu<sup>[1]</sup> The starting materials **2** were synthesized as reported also by Liu<sup>[2]</sup>. The starting materials **3** were synthesized as reported by Nevado<sup>[3]</sup>.

## 2. Equipments and experiments setup pictures

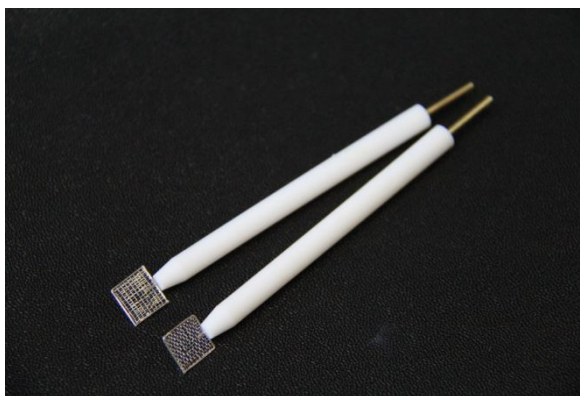


Figure S1. Pt net electrodes



Figure S2. The potentiostat

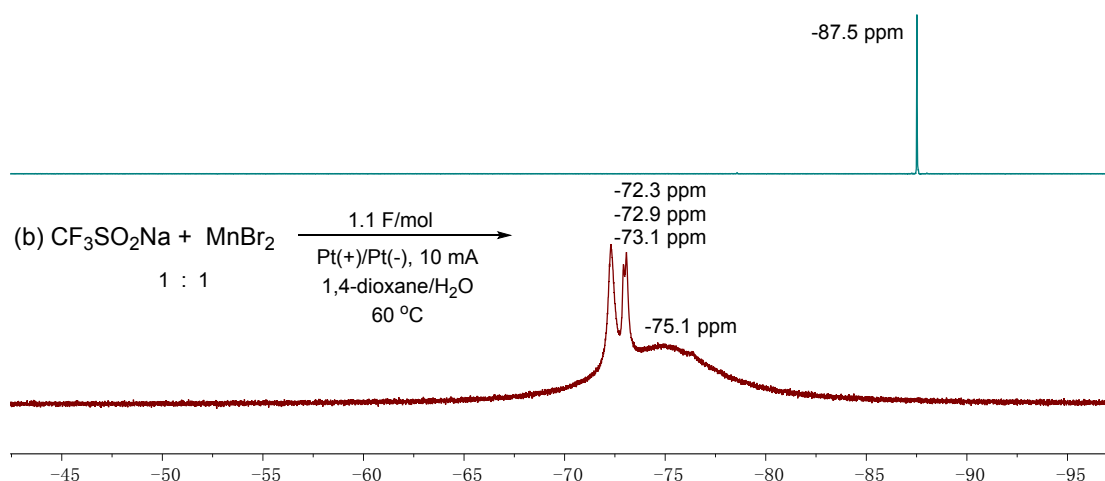


Figure S3. 0.3 mmol scale electrolysis experiment

### 3. Cyclic voltammetry and $^{19}\text{F}$ NMR studies

Cyclic voltammetry (CV) experiments were conducted in a 50 mL glass vial fitted with an L-type glassy carbon working electrode, a saturated calomel electrode (SCE) reference electrode, and a platinum wire counter electrode. The solution of interest was sparged with nitrogen for 3-5 minutes before data collection.

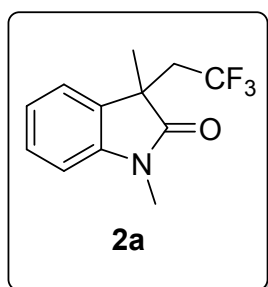
(a)  $\text{CF}_3\text{SO}_2\text{Na}$  in 1,4-dioxane/ $\text{H}_2\text{O}$



**Figure S4.**  $^{19}\text{F}$  NMR (471 MHz) spectra of (a)  $\text{CF}_3\text{SO}_2\text{Na}$  in 1,4-dioxane/ $\text{H}_2\text{O}$  solution and (b) electrochemical reaction mixture of  $\text{CF}_3\text{SO}_2\text{Na}$  and  $\text{MnBr}_2 \cdot 4\text{H}_2\text{O}$  in 1,4-dioxane/ $\text{H}_2\text{O}$  at  $60\text{ }^\circ\text{C}$  after passing 1.1 F/mol electricity.

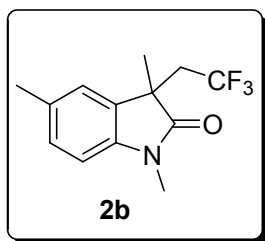
### 4. Compound data

*General Procedure for the 0.3 mmol scale electrochemical experiments:* To a 15 mL test tube with a stir bar was charged with 0.3 mmol **1**, 0.45 mmol Langlois reagent and 0.06 mmol  $\text{MnBr}_2$ , followed by 3 mL dioxane and 0.5 mL aqueous  $\text{H}_3\text{PO}_4$  solution (1.2 M, 0.6 mmol). Two platinum net electrodes (1.0 cm  $\times$  1.0 cm) were set up in the tube. Make sure that the electrodes be totally immersed. The resulting mixture was electrolyzed at constant current mode with a current of 10 mA under ambient temperature. The reaction was monitored by TLC or GC. If the reaction was not completed after 3 hours, add 0.45 mmol Langlois reagent. Upon completion, the reaction mixture was concentrated under reduced pressure. The residue was chromatographed through silica gel eluting with PE/EA to give the desired product **2**, **4**, **6**.



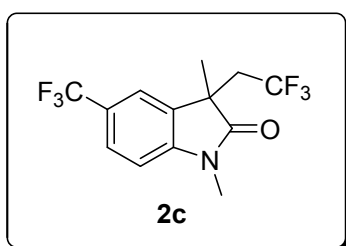
1,3-Dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2a**). White solid, 49mg, 67%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.31 (td,  $J = 7.7, 1.3$  Hz, 1H), 7.27 (d,  $J = 7.3$  Hz, 1H), 7.09 (td,  $J = 7.5, 1.0$  Hz, 1H), 6.88 (d,  $J = 7.8$  Hz, 1H), 3.23 (s, 3H), 2.82 (dq,  $J = 15.1, 10.7$  Hz, 1H), 2.65 (dq,  $J =$

15.2, 10.5 Hz, 1H), 1.41 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.50, 142.88, 131.01, 128.54, 125.27 (q,  $J = 278.1$  Hz), 123.56, 122.66, 108.47, 44.38 (q,  $J = 2.1$  Hz), 40.64 (q,  $J = 28.5$  Hz), 26.42, 25.01.<sup>[4]</sup>



1,3,5-Trimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2b**). White solid, 58mg, 75%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.11 (ddd,  $J = 7.9, 1.7, 0.8$  Hz, 1H), 7.08 (d,  $J = 1.6$  Hz, 1H), 6.77 (d,  $J = 7.9$  Hz, 1H), 3.21 (s, 3H), 2.81 (dq,  $J = 15.1, 10.8$  Hz, 1H), 2.63 (dq,  $J = 15.1, 10.5$  Hz, 1H), 2.35 (s, 3H), 1.39 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.43, 140.48, 132.18, 131.08, 128.77, 126.67, 125.29 (q,  $J = 278.2$  Hz), 124.34, 108.17, 44.43 (q,  $J = 2.2$  Hz), 40.62 (q,  $J = 28.0$  Hz), 26.44, 25.04,

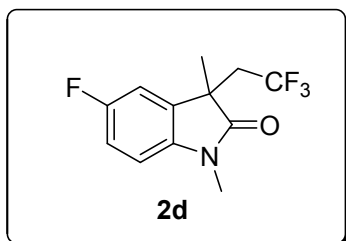
21.12.<sup>[4]</sup>



1,3-Dimethyl-3-(2,2,2-trifluoroethyl)-5-

(trifluoromethyl)indolin-2-one (**2c**). White solid, 72mg, 77%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.66 – 7.57 (m, 1H), 7.50 (d,  $J = 1.7$  Hz, 1H), 6.97 (d,  $J = 8.2$  Hz, 1H), 3.28 (s, 3H), 2.87 (dq,  $J = 15.2, 10.6$  Hz, 1H), 2.69 (dq,  $J = 15.2, 10.3$  Hz, 1H), 1.44 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.36, 145.86, 131.54, 126.38 (q,  $J = 4.3$  Hz), 125.01 (q,  $J = 32.5$  Hz), 125.00 (q,  $J = 276.6$  Hz),

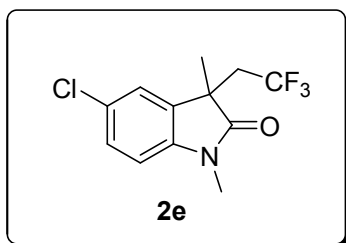
124.28 (q,  $J = 269.9$  Hz), 120.66 (q,  $J = 1.7$  Hz), 108.30, 44.32 (q,  $J = 2.7$  Hz), 40.57 (q,  $J = 28.5$  Hz), 26.63, 24.89.<sup>[5]</sup>



5-Fluoro-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one

(**2d**). White solid, 58mg, 74%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.02 (m, 2H), 6.81 (dd,  $J = 9.2, 4.2$  Hz, 1H), 3.23 (s, 3H), 2.93 – 2.75 (m, 1H), 2.72 – 2.55 (m, 1H), 1.41 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.1, 159.2 (d,  $J = 239.5$ ), 138.8 (d,  $J = 2.0$  Hz), 132.6 (d,  $J = 7.7$  Hz), 125.1 (q,  $J = 277.9$  Hz), 114.8 (d,  $J = 23.6$  Hz), 111.7 (dd,  $J = 1.5$  Hz, 24.7 Hz), 108.9 (d,  $J = 8.1$ ), 44.8, 40.5

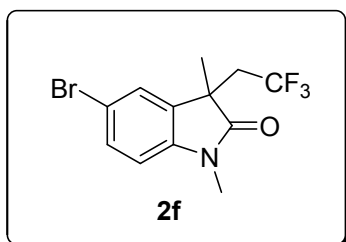
(q,  $J = 28.4$  Hz), 26.5, 24.9 (d,  $J = 10.0$  Hz).<sup>[4]</sup>



5-Chloro-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one

(**2e**). White solid, 69mg, 83%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.29 (dd,  $J = 8.3, 2.1$  Hz, 1H), 7.24 (d,  $J = 2.0$  Hz, 1H), 6.81 (d,  $J = 8.3$  Hz, 1H), 3.22 (s, 3H), 2.83 (dd,  $J = 15.2, 10.7$  Hz, 1H), 2.63 (dd,  $J = 15.2, 10.4$  Hz, 1H), 1.41 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 177.9, 141.4, 132.6, 128.5, 128.1, 125.0 (q,  $J = 276.3$  Hz), 124.0 (d,  $J = 1.6$  Hz), 109.4, 44.5 (d,  $J = 1.9$  Hz), 40.5

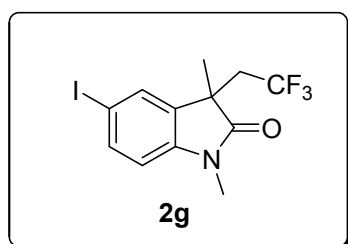
(q,  $J = 28.3$  Hz), 26.6, 24.9.<sup>[4]</sup>



5-Bromo-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2f**).

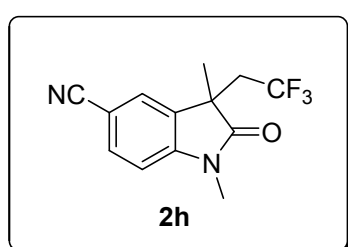
White solid, 52mg, 54%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.44 (dd,  $J = 8.3, 2.0$  Hz, 1H), 7.38 (d,  $J = 1.9$  Hz, 1H), 6.77 (d,  $J =$

8.3 Hz, 1H), 3.22 (s, 3H), 2.83 (dq,  $J = 15.2, 10.6$  Hz, 1H), 2.63 (dq,  $J = 15.2, 10.4$  Hz, 1H), 1.41 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 177.83, 141.95, 133.05, 131.45, 126.84, 125.05 (q,  $J = 276.3$ ), 115.34, 109.93, 44.54 (q,  $J = 2.3$ ), 40.60 (q,  $J = 28.2$ ), 26.55, 24.95.<sup>[4]</sup>



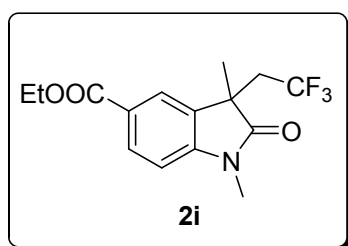
5-Iodo-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2g**). Pale yellow solid, 33mg, 30%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.63 (dd,  $J = 8.2, 1.7$  Hz, 1H), 7.54 (d,  $J = 1.8$  Hz, 1H), 6.67 (d,  $J = 8.2$  Hz, 1H), 3.21 (s, 3H), 2.82 (dq,  $J = 15.2, 10.6$  Hz, 1H), 2.62 (dq,  $J = 15.2, 10.4$  Hz, 1H), 1.40 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 177.67, 142.63, 137.41, 133.39, 132.38, 125.05 (q,  $J = 278.1$  Hz), 110.53, 85.09, 77.25, 44.36 (q,  $J = 2.1$  Hz),

40.59 (q,  $J = 28.4$  Hz), 26.51, 24.97.<sup>[4]</sup>



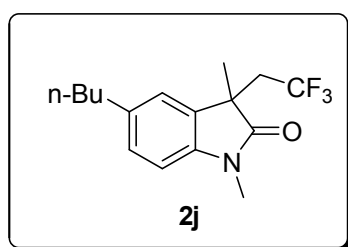
1,3-Dimethyl-2-oxo-3-(2,2,2-trifluoroethyl)indoline-5-carbonitrile (**2h**). White solid, 49mg, 61%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.66 (dd,  $J = 8.2, 1.7$  Hz, 1H), 7.54 (d,  $J = 1.6$  Hz, 1H), 6.98 (d,  $J = 8.2$  Hz, 1H), 3.28 (s, 3H), 2.88 (dq,  $J = 15.3, 10.6$  Hz, 1H), 2.70 (dq,  $J = 15.2, 10.3$  Hz, 1H), 1.44 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.09, 146.74, 133.88, 131.99, 127.02 (q,  $J = 1.6$  Hz), 124.89 (q,  $J = 278.0$  Hz), 118.99, 109.02,

105.93, 44.15 (q,  $J = 2.2$  Hz), 40.51 (q,  $J = 28.6$  Hz), 26.70, 24.85.<sup>[6]</sup>



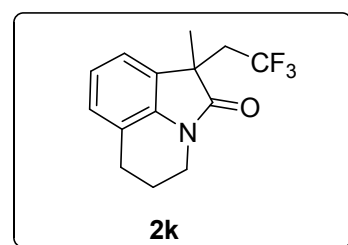
Ethyl 1,3-dimethyl-2-oxo-3-(2,2,2-trifluoroethyl)indoline-5-carboxylate (**2i**). White solid, 58mg, 61%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.08 (dd,  $J = 8.2, 1.7$  Hz, 1H), 7.94 (d,  $J = 1.7$  Hz, 1H), 6.93 (d,  $J = 8.3$  Hz, 1H), 4.38 (q,  $J = 7.3$  Hz, 2H), 3.28 (s, 3H), 2.88 (dq,  $J = 15.2, 10.6$  Hz, 1H), 2.71 (dq,  $J = 15.2, 10.3$  Hz, 1H), 1.53 – 1.34 (m, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.70, 166.25, 146.88, 131.16, 130.92, 125.06, 125.02 (q,  $J = 278.2$  Hz),

124.74, 108.00, 61.00, 44.18 (q,  $J = 2.2$  Hz), 40.65 (q,  $J = 28.2$  Hz), 26.64, 25.03, 14.38.<sup>[6]</sup>



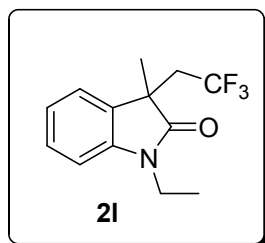
5-Butyl-1,3-dimethyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2j**). White solid, 64mg, 71%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.11 (dd,  $J = 7.9, 1.7$  Hz, 1H), 7.08 (s, 1H), 6.79 (d,  $J = 7.9$  Hz, 1H), 3.22 (s, 3H), 2.81 (dq,  $J = 15.2, 10.8$  Hz, 1H), 2.72 – 2.55 (m, 3H), 1.65 – 1.52 (m, 2H), 1.40 (s, 3H), 1.33 (dt,  $J = 14.6, 7.4$  Hz, 2H), 0.92 (t,  $J = 7.3$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.53, 140.64, 137.46, 130.95, 128.21, 125.30 (q,  $J = 278.5$  Hz),

123.77, 108.14, 44.46 (q,  $J = 2.4$  Hz), 40.65 (q,  $J = 28.3$  Hz), 35.36, 34.02, 26.45, 25.02, 22.18, 13.94.<sup>[5]</sup>



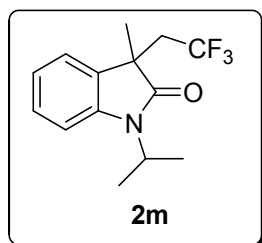
1-Methyl-1-(2,2,2-trifluoroethyl)-5,6-dihydro-1H-pyrrolo[3,2,1-i]quinolin-2(4H)-one (**2k**). White solid, 54mg, 67%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.11 (d,  $J = 7.4$  Hz, 1H),

7.06 (dd,  $J = 7.7, 1.1$  Hz, 1H), 6.97 (t,  $J = 7.5$  Hz, 1H), 3.76 – 3.69 (m, 2H), 2.85 – 2.71 (m, 3H), 2.65 (dq,  $J = 15.2, 10.5$  Hz, 1H), 2.06 – 1.97 (m, 2H), 1.42 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 177.37, 138.64, 129.69, 127.29, 125.43 (q,  $J = 276$  Hz), 122.09, 121.48, 120.52, 45.65 (q,  $J = 1.9$  Hz), 40.46 (q,  $J = 1.9$  Hz), 39.06, 24.61, 24.57, 21.13.<sup>[4]</sup>

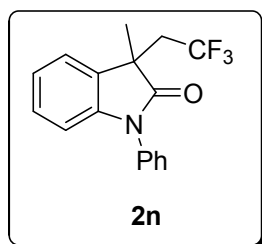


1-Ethyl-3-methyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2l**). White solid, 53mg, 69%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.30 (td,  $J = 7.7, 1.3$  Hz, 1H), 7.26 (t,  $J = 3.7$  Hz, 1H), 7.08 (td,  $J = 7.6, 1.0$  Hz, 1H), 6.90 (d,  $J = 7.8$  Hz, 1H), 3.88 (dq,  $J = 14.5, 7.3$  Hz, 1H), 3.69 (dq,  $J = 14.3, 7.2$  Hz, 1H), 2.84 (dq,  $J = 15.1, 10.7$  Hz, 1H), 2.64 (dq,  $J = 15.1, 10.5$  Hz, 1H), 1.40 (s, 3H), 1.25 (t,  $J = 7.2$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.08, 141.92, 31.23, 128.45, 125.26 (q,  $J = 278.3$  Hz),

123.72, 122.39, 108.61, 44.29 (q,  $J = 1.9$  Hz), 40.68 (q,  $J = 28.3$  Hz), 34.78, 25.12, 12.26.<sup>[7]</sup>

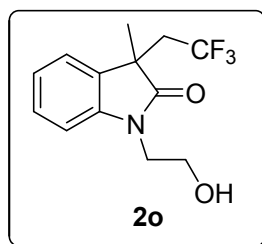


1-Ethyl-3-methyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2m**). White solid, 63mg, 77%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.30 – 7.22 (m, 2H), 7.09 – 7.01 (m, 2H), 4.63 (hept,  $J = 7.0$  Hz, 1H), 2.84 (dq,  $J = 15.1, 10.7$  Hz, 1H), 2.62 (dq,  $J = 15.1, 10.4$  Hz, 1H), 1.48 (dd,  $J = 7.1, 5.0$  Hz, 6H), 1.38 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.16, 141.57, 131.41, 128.20, 125.26 (q,  $J = 278.4$  Hz), 123.74, 122.02, 110.14, 44.08 (q,  $J = 1.8$  Hz), 43.98, 40.78 (q,  $J = 28.1$  Hz), 25.33, 19.21, 19.03.<sup>[7]</sup>

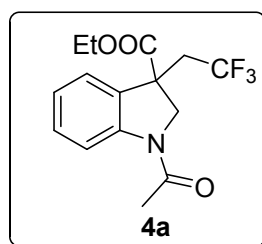


3-Methyl-1-phenyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2n**). White solid, 61mg, 67%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.53 (dd,  $J = 8.6, 6.6$  Hz, 2H), 7.45 – 7.36 (m, 3H), 7.32 (dd,  $J = 7.4, 1.2$  Hz, 1H), 7.26 – 7.19 (m, 1H), 7.12 (td,  $J = 7.6, 1.1$  Hz, 1H), 6.87 – 6.80 (m, 1H), 2.96 (dq,  $J = 15.1, 10.7$  Hz, 1H), 2.72 (dq,  $J = 15.1, 10.4$  Hz, 1H), 1.53 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 178.00, 142.96, 134.35, 130.69, 129.70, 128.47, 128.29, 126.64, 125.33 (q,  $J = 278.3$  Hz), 123.79, 123.09,

109.79, 44.53 (q,  $J = 2.2$  Hz), 41.09 (q,  $J = 28.2$  Hz), 25.47.<sup>[4]</sup>

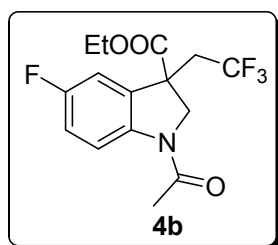


1-(2-Hydroxyethyl)-3-methyl-3-(2,2,2-trifluoroethyl)indolin-2-one (**2o**). White solid, 58mg, 71%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.33 – 7.28 (m, 1H), 7.26 (d,  $J = 7.1$  Hz, 1H), 7.09 (td,  $J = 7.6, 0.9$  Hz, 1H), 6.98 (d,  $J = 7.9$  Hz, 1H), 4.01 – 3.80 (m, 4H), 2.85 (dq,  $J = 15.2, 10.7$  Hz, 1H), 2.74 – 2.56 (m, 2H), 1.41 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 179.59, 142.25, 130.98, 128.56, 125.23 (q,  $J = 278.6$  Hz), 123.68, 122.82, 109.01, 60.32, 44.37 (q,  $J = 2.6$  Hz), 43.08, 40.70 (q,  $J = 28.0$  Hz), 25.19.



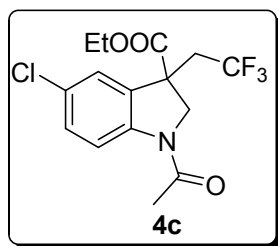
Ethyl 1-methyl-3-(2,2,2-trifluoroethyl)indoline-3-carboxylate (**4a**). White solid, 62mg, 66%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.21 (d,  $J = 8.1$  Hz, 1H), 7.38 – 7.34 (m, 1H), 7.31 (td,  $J = 7.8, 1.3$  Hz, 1H), 7.08 (td,  $J = 7.6, 1.1$  Hz, 1H), 4.86 (d,  $J = 11.2$  Hz, 1H), 4.21 (qd,  $J = 7.1, 1.7$  Hz, 2H), 4.05 (d,  $J = 11.2$  Hz, 1H), 3.35 (dq,  $J = 15.3, 10.7$  Hz, 1H), 2.66 –

2.48 (m, 1H), 2.30 (s, 3H), 1.26 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 170.18, 168.51, 141.83, 130.33, 130.13, 125.52 (q,  $J = 278.4$  Hz), 124.11, 123.76, 117.53, 62.52, 55.13, 50.91, 41.03 (q,  $J = 28.0$  Hz), 24.22, 13.88.  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ): -61.73. IR:  $\nu = 2986, 1733, 1667, 1481, 1402, 1255, 1219, 1129, 754\text{cm}^{-1}$ . HRMS (ESI): calcd for  $\text{C}_{15}\text{H}_{17}\text{F}_3\text{NO}_3^+$   $[\text{M}+\text{H}]^+$ : 316.1160; found: 316.1160.

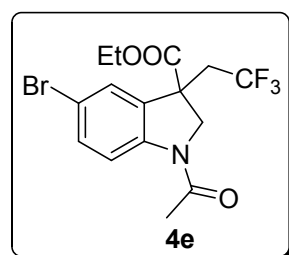


Ethyl 1-acetyl-5-fluoro-3-(2,2,2-trifluoroethyl)indoline-3-carboxylate (**4b**). White solid, 62mg, 62%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.18 (dd,  $J = 8.9, 4.8$  Hz, 1H), 7.07 (dd,  $J = 8.1, 2.7$  Hz, 1H), 7.01 (td,  $J = 8.8, 2.7$  Hz, 1H), 4.87 (d,  $J = 11.2$  Hz, 1H), 4.23 (qq,  $J = 7.0, 3.6$  Hz, 2H), 4.08 (d,  $J = 11.2$  Hz, 1H), 3.27 (dq,  $J = 15.2, 10.6$  Hz, 1H), 2.57 (dq,  $J = 15.2, 10.0$  Hz, 1H), 2.29 (s, 3H), 1.29 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 169.66, 168.25, 159.27 (d,  $J = 243.9$  Hz), 138.06, 131.92

(d,  $J = 8.0$  Hz), 125.32 (q,  $J = 278.7$  Hz), 118.51 (d,  $J = 8.0$  Hz), 116.67 (d,  $J = 22.7$  Hz), 111.21 (d,  $J = 25.2$  Hz), 62.81, 55.33, 50.84, 40.95 (q,  $J = 28.4$  Hz), 23.95, 13.87.  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ): -61.75, -117.69. IR:  $\nu = 2986, 2159, 1738, 1668, 1487, 1401, 1318, 1257, 1224, 1149, 1128\text{cm}^{-1}$ . HRMS (ESI): calcd for  $\text{C}_{15}\text{H}_{16}\text{F}_4\text{NO}_3^+$   $[\text{M}+\text{H}]^+$ : 334.1066; found: 334.1061.

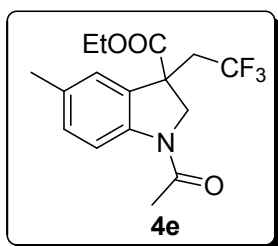


Ethyl 1-acetyl-5-chloro-3-(2,2,2-trifluoroethyl)indoline-3-carboxylate (**4c**). White solid, 80mg, 79%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.15 (d,  $J = 8.6$  Hz, 1H), 7.32 (d,  $J = 2.2$  Hz, 1H), 7.27 (dd,  $J = 8.7, 2.2$  Hz, 1H), 4.86 (d,  $J = 11.2$  Hz, 1H), 4.24 (qq,  $J = 7.5, 3.6$  Hz, 2H), 4.07 (d,  $J = 11.2$  Hz, 1H), 3.29 (dq,  $J = 15.2, 10.6$  Hz, 1H), 2.56 (dq,  $J = 15.2, 10.0$  Hz, 1H), 2.29 (s, 3H), 1.29 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 169.63, 168.48, 140.51, 131.97, 130.15, 128.96, 125.31 (d,  $J = 278.6$  Hz), 124.08, 118.46, 62.85, 55.24, 50.83, 40.98 (q,  $J = 28.3$  Hz), 24.06, 13.88.  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ): -61.72. IR:  $\nu = 2922, 2159, 1734, 1700, 1477, 1393, 1255, 1219, 1171, 1133, 826\text{cm}^{-1}$ . HRMS (ESI): calcd for  $\text{C}_{15}\text{H}_{16}\text{ClF}_3\text{NO}_3^+$   $[\text{M}+\text{H}]^+$ : 350.0771; found: 350.0771.



Ethyl 1-acetyl-5-bromo-3-(2,2,2-trifluoroethyl)indoline-3-carboxylate (**4e**). Pale white solid, 70mg, 59%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.10 (d,  $J = 8.6$  Hz, 1H), 7.46 (d,  $J = 2.0$  Hz, 1H), 7.42 (dd,  $J = 8.7, 2.1$  Hz, 1H), 4.85 (d,  $J = 11.2$  Hz, 1H), 4.24 (qq,  $J = 6.8, 3.6$  Hz, 2H), 4.07 (d,  $J = 11.2$  Hz, 1H), 3.29 (dq,  $J = 15.3, 10.6$  Hz, 1H), 2.56 (dq,  $J = 15.3, 10.0$  Hz, 1H), 2.29 (s, 3H), 1.30 (d,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 169.63, 168.53, 140.99, 133.07, 132.32, 126.97, 125.31

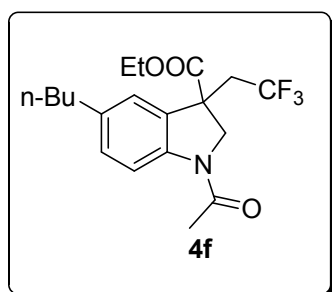
(d,  $J = 278.7$  Hz), 118.89, 116.25, 62.87, 55.18, 50.79, 41.00 (q,  $J = 28.5$  Hz), 24.11, 13.89.  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ): -61.71. IR:  $\nu = 2986, 2161, 1734, 1669, 1475, 1392, 1255, 1218, 1132, 825\text{cm}^{-1}$ . HRMS (ESI): calcd for  $\text{C}_{15}\text{H}_{16}\text{BrF}_3\text{NO}_3^+$   $[\text{M}+\text{H}]^+$ : 361.0266; found: 361.0261.



Ethyl 1-acetyl-5-methyl-3-(2,2,2-trifluoroethyl)indoline-3-carboxylate (**4e**). White solid, 68mg, 69%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.07 (d,  $J = 8.2$  Hz, 1H), 7.13 (d,  $J = 1.7$  Hz, 1H), 7.13 – 7.09 (m, 1H), 4.83 (d,  $J = 11.2$  Hz, 1H), 4.25 – 4.18 (m, 2H), 4.03 (d,  $J = 11.2$  Hz, 1H),

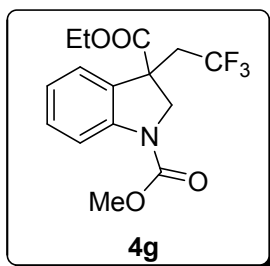


3.34 (dq,  $J = 15.3, 10.8$  Hz, 1H), 2.52 (dq,  $J = 15.3, 10.1$  Hz, 1H), 2.33 (s, 3H), 2.28 (s, 3H), 1.27 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 170.28, 168.18, 139.56, 133.90, 130.65, 130.46, 125.54 (q,  $J = 279.3$  Hz), 124.15, 117.24, 62.47, 55.24, 50.85, 41.04 (q,  $J = 28.1$  Hz), 24.10, 21.09, 13.89.  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ): -61.68. IR:  $\nu = 2924, 2159, 1733, 1663, 1491, 1396, 1255, 1131, 1015, 824\text{cm}^{-1}$ . HRMS (ESI): calcd for  $\text{C}_{16}\text{H}_{19}\text{F}_3\text{NO}_3^+$   $[\text{M}+\text{H}]^+$ : 330.1317; found: 330.1315.



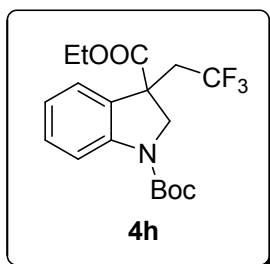
Ethyl 1-acetyl-5-butyl-3-(2,2,2-trifluoroethyl)indoline-3-carboxylate (**4f**). White solid, 79mg, 71%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.08 (d,  $J = 8.2$  Hz, 1H), 7.14 (d,  $J = 1.7$  Hz, 1H), 7.11 (dd,  $J = 8.3, 1.8$  Hz, 1H), 4.84 (d,  $J = 11.2$  Hz, 1H), 4.20 (qd,  $J = 7.1, 1.9$  Hz, 2H), 4.03 (d,  $J = 11.1$  Hz, 1H), 3.35 (dq,  $J = 15.3, 10.8$  Hz, 1H), 2.58 (t,  $J = 7.7$  Hz, 2H), 2.56–2.46 (m, 1H), 2.28 (s, 3H), 1.62–1.50 (m, 2H), 1.34 (p,  $J = 7.4$  Hz, 2H), 1.26 (t,  $J = 7.1$  Hz, 3H), 0.92 (t,  $J = 7.3$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 170.25,

168.16, 139.71, 139.06, 130.35, 130.09, 125.57 (q,  $J = 278.7$  Hz), 123.52, 117.22, 62.38, 55.30, 50.89, 40.99 (q,  $J = 28.0$  Hz), 35.23, 33.77, 24.06, 22.20, 13.91, 13.87.  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ): -61.75. IR:  $\nu = 2957, 2928, 2859, 1735, 1666, 1491, 1399, 1255, 1131, 1014, 834\text{cm}^{-1}$ . HRMS (ESI): calcd for  $\text{C}_{19}\text{H}_{25}\text{F}_3\text{NO}_3^+$   $[\text{M}+\text{H}]^+$ : 372.1787; found: 372.1779.



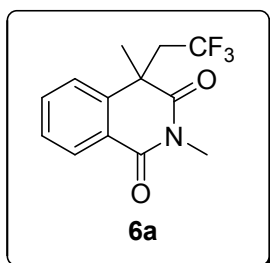
3-Ethyl 1-methyl 3-(2,2,2-trifluoroethyl)indoline-1,3-dicarboxylate (**4g**). White solid, 71.5mg, 72%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.90 (s, 1H), 7.34 (dd,  $J = 7.7, 1.2$  Hz, 1H), 7.29 (d,  $J = 7.8$  Hz, 1H), 7.02 (td,  $J = 7.6, 1.1$  Hz, 1H), 4.83 (d,  $J = 12.0$  Hz, 1H), 4.20 (q,  $J = 7.1$  Hz, 2H), 4.03 (d,  $J = 12.5$  Hz, 1H), 3.86 (s, 3H), 3.29 (dq,  $J = 15.2, 10.7$  Hz, 1H), 2.54 (dq,  $J = 15.2, 10.1$  Hz, 1H), 1.25 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 170.42, 153.15, 141.68, 130.06, 125.49 (q,  $J = 278.7\text{Hz}$ ),

124.11, 123.06, 115.26, 62.34, 53.89, 52.82, 50.48, 41.25 (q,  $J = 28.2$  Hz), 13.88.  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ): -61.52. IR:  $\nu = 2959, 1712, 1487, 1443, 1396, 1254, 1217, 1130, 1064, 1015, 751\text{cm}^{-1}$ . HRMS (ESI): calcd for  $\text{C}_{15}\text{H}_{17}\text{F}_3\text{NO}_4^+$   $[\text{M}+\text{H}]^+$ : 332.1110; found: 332.1107.



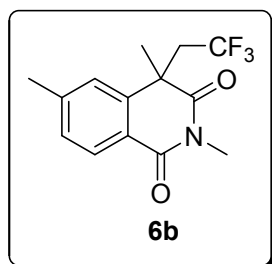
1-Tert-butyl 3-ethyl 3-(2,2,2-trifluoroethyl)indoline-1,3-dicarboxylate (**4h**). White solid, 80.5mg, 72%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.87 (s, 1H), 7.32 (dd,  $J = 7.7, 1.3$  Hz, 1H), 7.30–7.25 (m, 1H), 6.98 (td,  $J = 7.5, 1.1$  Hz, 1H), 4.76 (d,  $J = 12.1$  Hz, 1H), 4.20 (q,  $J = 7.1$  Hz, 2H), 3.99 (d,  $J = 12.1$  Hz, 1H), 3.27 (dq,  $J = 15.2, 10.7$  Hz, 1H), 2.54 (dq,  $J = 15.2, 10.1$  Hz, 1H), 1.58 (s, 9H), 1.26 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 170.61, 151.99, 129.93, 125.52 (q,  $J = 278.5$  Hz),

123.99, 122.60, 62.26, 54.05, 54.02, 41.32 (q,  $J = 28.0$  Hz), 28.41, 13.90.  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ): -61.64. IR:  $\nu = 2982, 2161, 1704, 1485, 1392, 1254, 1130, 1018, 751\text{cm}^{-1}$ . HRMS (ESI): calcd for  $\text{C}_{18}\text{H}_{23}\text{F}_3\text{NO}_4^+$   $[\text{M}+\text{H}]^+$ : 374.1579; found: 374.1580.

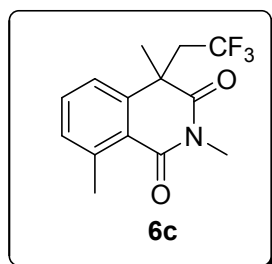


2,4-Dimethyl-4-(2,2,2-trifluoroethyl)isoquinoline-1,3(2H,4H)-dione (**6a**). White solid, 54mg, 66%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.29 (dd,  $J = 7.9, 1.4$  Hz, 1H), 7.67 (td,  $J = 7.6, 1.5$  Hz, 1H), 7.49 (ddd,  $J =$

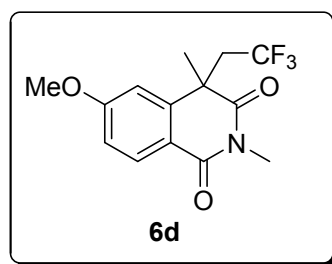
8.1, 7.4, 1.1 Hz, 1H), 7.43 (dd,  $J = 7.9, 1.2$  Hz, 1H), 3.41 (s, 3H), 3.41 – 3.28 (m, 1H), 2.81 (dq,  $J = 15.1, 9.8$  Hz, 1H), 1.67 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 174.57, 163.74, 140.37, 133.82, 129.30, 128.07, 125.65, 124.22, 123.58, 44.76, 44.49, 44.22, 43.57, 43.55, 31.18, 27.42.<sup>[8]</sup>



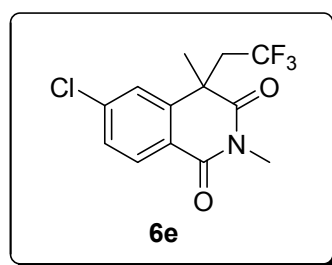
2,4,6-Trimethyl-4-(2,2,2-trifluoroethyl)isoquinoline-1,3(2H,4H)-dione (**6b**). White solid, 59mg, 69%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.16 (d,  $J = 8.0$  Hz, 1H), 7.31 – 7.26 (m, 1H), 7.20 (s, 1H), 3.40 (s, 3H), 3.33 (dq,  $J = 15.1, 9.7$  Hz, 1H), 2.79 (dq,  $J = 15.1, 9.7$  Hz, 1H), 2.46 (s, 3H), 1.65 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 174.73, 163.78, 144.75, 140.39, 129.30, 129.17, 126.00, 124.99 (q,  $J = 278.8$  Hz), 121.74, 44.33 (q,  $J = 27.7$  Hz), 43.49 (q,  $J = 2.1$  Hz), 31.20, 27.33, 21.93.<sup>[8]</sup>



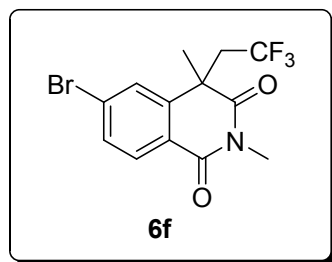
2,4,8-Trimethyl-4-(2,2,2-trifluoroethyl)isoquinoline-1,3(2H,4H)-dione (**6c**). White solid, 62mg, 72%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.50 (t,  $J = 7.7$  Hz, 1H), 7.33 – 7.25 (m, 2H), 3.30-3.41 (m, 4H), 2.87 – 2.68 (m, 4H), 1.66 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 174.26, 164.24, 143.04, 141.70, 132.58, 132.06, 125.05 (q,  $J = 278.7$  Hz), 123.96, 122.56, 44.66 (q,  $J = 27.7$  Hz), 43.60 (q,  $J = 2.5$  Hz), 31.63, 27.40, 24.11.<sup>[9]</sup>



6-Methoxy-2,4-dimethyl-4-(2,2,2-trifluoroethyl)isoquinoline-1,3(2H,4H)-dione (**6d**). White solid, 40mg, 44%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.24 (d,  $J = 8.8$  Hz, 1H), 7.00 (dd,  $J = 8.8, 2.4$  Hz, 1H), 6.85 (d,  $J = 2.4$  Hz, 1H), 3.91 (s, 3H), 3.39 (s, 3H), 3.33 (dq,  $J = 15.1, 9.7$  Hz, 1H), 2.76 (dq,  $J = 15.1, 9.7$  Hz, 1H), 1.65 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 174.64, 163.97, 163.40, 142.57, 131.68, 124.97 (q,  $J = 278.8$  Hz), 117.23, 113.60, 111.16, 55.64, 44.41 (q,  $J = 27.5$  Hz), 43.74 (q,  $J = 2.4$  Hz), 31.30, 27.28.<sup>[9]</sup>

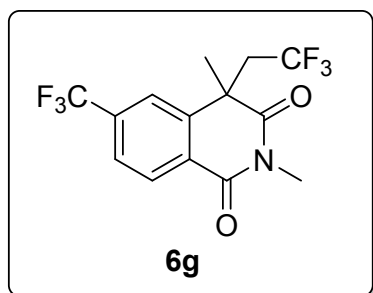


6-Chloro-2,4-dimethyl-4-(2,2,2-trifluoroethyl)isoquinoline-1,3(2H,4H)-dione (**6e**). White solid, 61mg, 67%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.23 (d,  $J = 8.4$  Hz, 1H), 7.47 (dd,  $J = 8.5, 2.0$  Hz, 1H), 7.41 (d,  $J = 2.0$  Hz, 1H), 3.40 (s, 3H), 3.39 – 3.29 (dq,  $J = 15.2, 9.7$  Hz, 1H), 2.77 (dq,  $J = 15.2, 9.7$  Hz, 1H), 1.67 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 173.91, 162.90, 142.06, 140.51, 130.92, 128.78, 125.90, 124.81 (q,  $J = 278.5$  Hz), 122.76, 44.40 (q,  $J = 27.7$  Hz), 43.57 (q,  $J = 2.4$  Hz), 31.01, 27.51.<sup>[9]</sup>



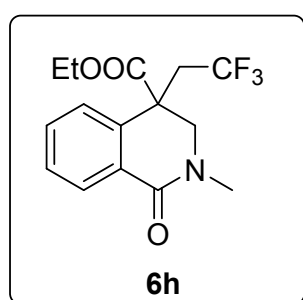
6-Bromo-2,4-dimethyl-4-(2,2,2-trifluoroethyl)isoquinoline-1,3(2H,4H)-dione (**6f**). White solid, 51mg, 49%.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.15 (d,  $J = 8.4$  Hz, 1H), 7.63 (dd,  $J = 8.5, 1.8$  Hz, 1H), 7.58 (d,  $J = 1.8$  Hz, 1H), 3.40 (s, 3H), 3.35 (dq,  $J = 15.1, 9.6$  Hz, 1H), 2.77 (dq,  $J = 15.1, 9.6$  Hz, 1H), 1.67 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ): 173.84, 163.03, 142.12, 131.71,

130.91, 129.09, 128.90, 124.81 (q,  $J = 278.5$  Hz), 123.18, 44.40 (q,  $J = 27.6$  Hz), 43.51 (q,  $J = 1.7$  Hz), 31.01, 27.52.<sup>[9]</sup>



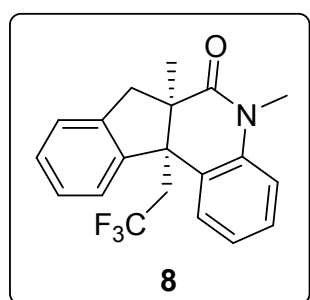
2,4-Dimethyl-4-(2,2,2-trifluoroethyl)-6-(trifluoromethyl)isoquinoline-1,3(2H,4H)-dione (**6g**). White solid, 42mg, 41%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.43 (d,  $J = 8.2$  Hz, 1H), 7.75 (dd,  $J = 8.3, 1.6$  Hz, 1H), 7.67 (s, 1H), 3.48 – 3.35 (m, 4H), 2.83 (dq,  $J = 15.2, 9.6$  Hz, 1H), 1.70 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): 173.75, 162.58, 135.46 (q,  $J = 32.9$  Hz), 130.25, 127.15, 124.91 (q,  $J = 3.2$  Hz), 124.76 (q,  $J = 278.7$  Hz), 123.23 (q,  $J = 273.3$  Hz), 122.85 (q,  $J = 3.9$  Hz),

44.40 (q,  $J = 27.7$  Hz), 43.70 (q,  $J = 1.9$  Hz), 31.00, 27.65.<sup>[9]</sup>



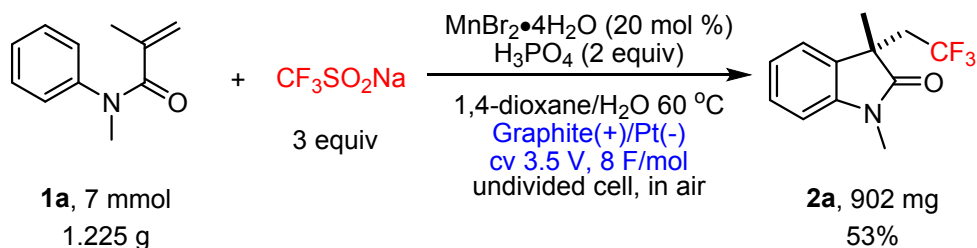
Ethyl 1-acetyl-5-fluoro-3-(2,2,2-trifluoroethyl)indoline-3-carboxylate (**6h**). White solid, 44mg, 46%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.18 (dd,  $J = 7.7, 1.5$  Hz, 1H), 7.50 (td,  $J = 7.5, 1.7$  Hz, 1H), 7.45 (td,  $J = 7.5, 1.5$  Hz, 1H), 7.38 (dd,  $J = 7.8, 1.3$  Hz, 1H), 4.30 (q,  $J = 7.1$  Hz, 2H), 4.21 (d,  $J = 13.4$  Hz, 1H), 3.60 (d,  $J = 13.4$  Hz, 1H), 3.27 – 3.10 (m, 4H), 2.60 (dq,  $J = 15.4, 10.1$  Hz, 1H), 1.31 (t,  $J = 7.1$  Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): 170.26, 163.41, 137.75, 132.37, 129.41, 128.78, 128.28, 125.32 (q,  $J = 278.8$  Hz),

124.31, 62.43, 51.50, 46.54 (q,  $J = 1.6$  Hz), 39.40 (q,  $J = 28.6$  Hz), 35.01, 13.94. <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>): -60.99. IR:  $\nu = 2984, 2159, 1732, 1657, 1258, 1222, 1178, 1131, 1094, 700$ cm<sup>-1</sup>. HRMS (ESI): calcd for C<sub>15</sub>H<sub>17</sub>F<sub>3</sub>NO<sub>3</sub><sup>+</sup> [M+H]<sup>+</sup>: 316.1160; found: 316.1158.



(6aR,11bR)-5,6a-dimethyl-11b-(2,2,2-trifluoroethyl)-5,6a,7,11b-tetrahydro-6H-indeno[2,1-c]quinolin-6-one (**8**). White solid, 33mg, 32%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.43 (d,  $J = 7.5$  Hz, 1H), 7.36 – 7.31 (m, 1H), 7.25 – 7.18 (m, 3H), 7.04 (dd,  $J = 7.9, 1.5$  Hz, 1H), 6.99 (dd,  $J = 8.3, 1.1$  Hz, 1H), 6.93 (td,  $J = 7.5, 1.1$  Hz, 1H), 3.45 (s, 3H), 3.09 (d,  $J = 15.1$  Hz, 1H), 2.98 (dq,  $J = 15.4, 11.1$  Hz, 1H), 2.78 (d,  $J = 15.1$  Hz, 1H), 2.70 (dq,  $J = 15.4, 10.3$  Hz, 1H), 1.35 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): 172.23, 146.54, 140.83, 139.49,

129.31, 128.23, 127.57, 126.80, 126.18 (q,  $J = 278.3$  Hz), 125.38, 124.58, 123.39, 123.09, 114.57, 52.68, 52.56, 44.42, 39.29 (q,  $J = 27.5$  Hz), 30.17, 18.69. HRMS (ESI): calcd for C<sub>20</sub>H<sub>19</sub>F<sub>3</sub>NO<sub>3</sub><sup>+</sup> [M+H]<sup>+</sup>: 346.1413; found: 346.1412.



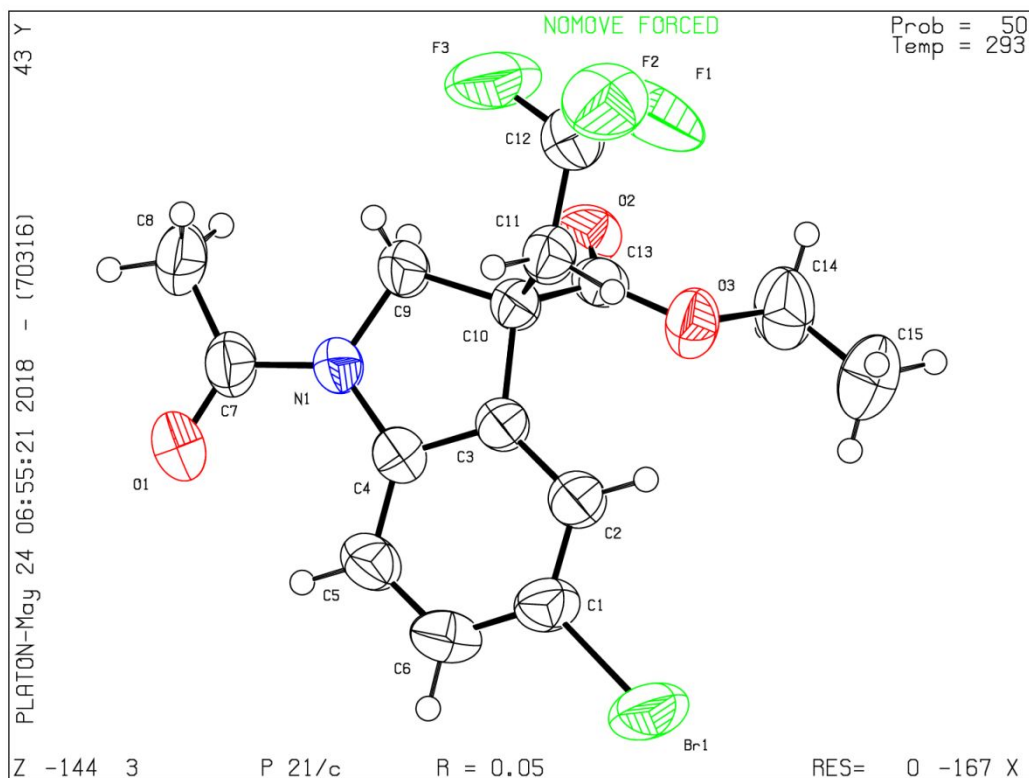
*Procedure for the gram scale electrochemical experiments:* To a 50 mL beaker with a stir bar was charged with 7 mmol **1a**, 10.5 mmol Langlois reagent and 0.35 mmol MnBr<sub>2</sub>, followed by 30 mL dioxane, 3 mL H<sub>2</sub>O, and 14 mmol H<sub>3</sub>PO<sub>4</sub>. Two electrodes (Graphite SK-50 used as the anode and Platinum as the cathode) were set up in the beaker. The resulting mixture was electrolyzed at constant voltage mode with 3.5 V under 60 °C. After 4 F/mol electric quantity, another 10.5 mmol Langlois reagent was added. After a total of 8 F/mol electric quantity, the reaction mixture was concentrated under reduced pressure. The residue was chromatographed through silica gel eluting with PE/EA to give the desired product **2a**.

## 5. References

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## 6. X-ray single crystal data

Crystal structure data of **4d**:



CCDC number: 1890462

Bond precision: C-C = 0.0048 Å Wavelength=0.71073

Cell: a=8.9486(4) b=17.7085(8) c=10.5821(5)

alpha=90 beta=104.565(5) gamma=90

Temperature: 293 K

Calculated Reported

Volume 1623.02(13) 1623.01(13)

Space group P 21/c P 21/c

Hall group -P 2ybc -P 2ybc

Moiety formula C<sub>15</sub> H<sub>15</sub> Br F<sub>3</sub> N O<sub>3</sub> ?

Sum formula C<sub>15</sub> H<sub>15</sub> Br F<sub>3</sub> N O<sub>3</sub> C<sub>15</sub> H<sub>15</sub> Br F<sub>3</sub> N O<sub>3</sub>

Mr 394.18 394.19

D<sub>x,g</sub> cm<sup>-3</sup> 1.613 1.613

Z 4 4

Mu (mm<sup>-1</sup>) 2.576 2.576

F<sub>000</sub> 792.0 792.0

F<sub>000</sub>' 791.34

h,k,l<sub>max</sub> 11,22,13 11,22,13

N<sub>ref</sub> 3720 3713

T<sub>min</sub>,T<sub>max</sub> 0.466,0.734 0.540,1.000

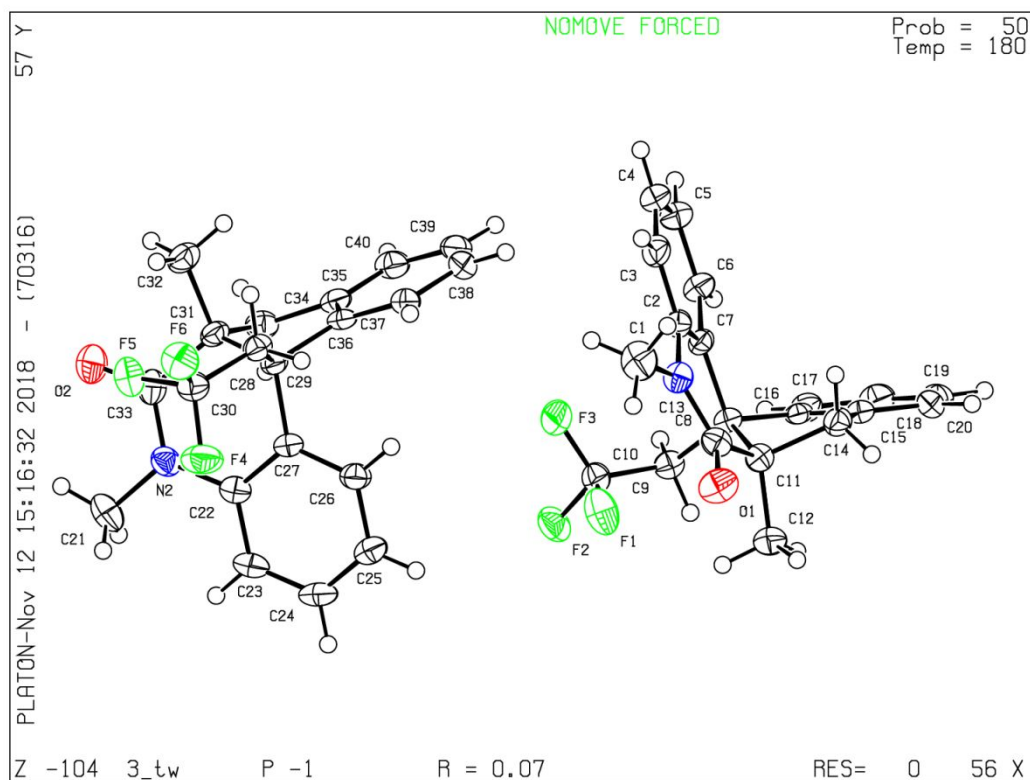
T<sub>min</sub>' 0.273

Correction method= # Reported T Limits: T<sub>min</sub>=0.540 T<sub>max</sub>=1.000

AbsCorr = MULTI-SCAN

Data completeness= 0.998 Theta(max)= 27.484  
 R(reflections)= 0.0484( 2170) wR2(reflections)= 0.1342( 3713)  
 S = 1.032 Npar= 210

Crystal structure data of **8**:



CCDC number: 1885350  
 Bond precision: C-C = 0.0054 Å Wavelength=1.54184  
 Cell: a=10.8613(6) b=12.2821(6) c=13.7407(8)  
 alpha=74.040(5) beta=71.906(5) gamma=78.776(3)  
 Temperature: 180 K  
 Calculated Reported  
 Volume 1663.19(17) 1663.19(17)  
 Space group P -1 P -1  
 Hall group -P 1 -P 1  
 Moiety formula C<sub>20</sub> H<sub>18</sub> F<sub>3</sub> N O ?  
 Sum formula C<sub>20</sub> H<sub>18</sub> F<sub>3</sub> N O C<sub>20</sub> H<sub>18</sub> F<sub>3</sub> N O  
 Mr 345.35 345.35  
 D<sub>x</sub>, g cm<sup>-3</sup> 1.379 1.379  
 Z 4 4  
 Mu (mm<sup>-1</sup>) 0.909 0.909  
 F<sub>000</sub> 720.0 720.0  
 F<sub>000</sub>' 722.54  
 h,k,l<sub>max</sub> 12,14,16 12,14,16

Nref 5872 5863

Tmin,Tmax 0.804,0.873 0.551,1.000

Tmin' 0.664

Correction method= # Reported T Limits: Tmin=0.551 Tmax=1.000

AbsCorr = MULTI-SCAN

Data completeness= 0.998 Theta(max)= 66.594

R(reflections)= 0.0700( 4423) wR2(reflections)= 0.1837( 5863)

S = 1.067 Npar= 456

## 7. $^1\text{H}$ NMR, $^{13}\text{C}$ NMR and $^{19}\text{F}$ NMR Spectra

