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#### **1.** General information

Dichloroethane (DCE), 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP), tetrabutylammonium perchlorate (*n*Bu4NClO4), tetrabutylammonium hexafluorophosphate (*n*Bu4NPF6), platinum plate, graphite felt are all obtained from commercial sources. The electrochemical instrument is HONGSHENGFENG DPS-305BM. Column chromatography was performed on silica gel (200-300 mesh). NMR spectra were recorded in CDCl<sub>3</sub> on Avance 500 MHz spectrometers. The chemical shifts ( $\delta$ ) are reported in parts per million (ppm) relative to the residue signal of CDCl<sub>3</sub> ( $\delta$ /ppm = 7.26 for <sup>1</sup>H NMR and  $\delta$ /ppm = 77.16 for <sup>13</sup>C NMR). The following abbreviations are used for multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet. The coupling constants *J* have been given in Hertz (Hz). HRMS were obtained on an Ultima Global spectrometer with an ESI source.

#### 2. General procedure



**Procedure A:** To a 10 mL three-necked flask (Figure S1) equipped with a graphite felt (1 cm x 1.5 cm x 1 cm) anode and a platinum plate (1 cm x 1 cm x 0.1 mm) cathode was charged with substrate (0.3 mmol, 1.0 equiv), TMSNCS (0.6 mmol, 2.0 equiv),  $nBu_4NCIO_4$  (0.6 mmol, 2.0 equiv) and DCE (6.0 mL). The electrolysis was carried out at room temperature using a constant current of 5 mA under N<sub>2</sub> for 4-6 h. After completion of the reaction, the mixture was evaporated and purified by column chromatography on silica gel to give the corresponding product.

$$\begin{array}{c} \hline Ar \\ \hline R \\ + \\ TMSNCS \\ \hline nBu_4NCIO_4, DCE/HFIP (3:1) \\ \hline 5 mA, RT, N_2 \\ \end{array}$$

**Procedure B:** To a 10 mL three-necked flask (Figure S1) equipped with a graphite felt (1 cm x 1.5 cm x 1 cm) anode and a platinum plate (1 cm x 1 cm x 0.1 mm) cathode was charged with substrate (0.3 mmol, 1.0 equiv), TMSNCS (0.6 mmol, 2.0 equiv),  $nBu_4NCIO_4$  (0.6 mmol, 2.0 equiv), DCE (4.5 mL) and HFIP (1.5 mL). The electrolysis was carried out at room temperature using a constant current of 5 mA under N<sub>2</sub> for 5-6 h. After completion of the

reaction, the mixture was evaporated and purified by column chromatography on silica gel to give the corresponding product.



**Procedure C:** To a 10 mL three-necked flask (Figure S1) equipped with a graphite felt (1 cm x 1.5 cm x 1 cm) anode and a platinum plate (1 cm x 1 cm x 0.1 mm) cathode was charged with substrate (0.3 mmol, 1.0 equiv), TMSNCS (0.6 mmol, 2.0 equiv),  $nBu_4NCIO_4$  (0.3 mmol, 1.0 equiv), DCE (4.5 mL) and HFIP (1.5 mL). The electrolysis was carried out at room temperature using a constant current of 7 mA under N<sub>2</sub> for 4-9 h. After completion of the reaction, the mixture was evaporated and purified by column chromatography on silica gel to give the corresponding product.







At the beginning of the reaction

At the end of the reaction

Figure S1. Electrolysis cell for small scale reaction

# **3.** Gram-scale reaction



To a 150 mL beaker-type cell (Figure S2) equipped with a graphite felt (3 cm x 3 cm x 1.5 cm) anode and a nickel plate (3 cm x 3 cm x 0.1 mm) cathode was charged with 1 (1.85 g, 10 mmol, 1.0 equiv), TMSNCS 2 (2.63 g, 20 mmol, 2.0 equiv),  $nBu_4NCIO_4$  (6.84 g, 20 mmol, 2.0 equiv) and DCE (90 mL). The electrolysis was carried out at room temperature using a constant current of 100 mA under N<sub>2</sub> for 10 h (3.7 F/mol). After completion of the reaction, the mixture was evaporated and purified by column chromatography on silica gel with petroleum ether/dichloromethane (2:1) to give **3** as a yellow oil (1.48 g, 61% yield).





At the beginning of the reaction

At the end of the reaction



### 4. Characterization data



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 6 h (3.7 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/dichloromethane (2:1) to afford **3** (59 mg, 81% yield) as a yellow oil.

<sup>1</sup>**H** NMR (500 MHz, CDCl<sub>3</sub>) δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 4.55 (q, *J* = 7.0 Hz, 1H), 1.85 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 138.3, 132.3, 128.8, 123.1, 111.4, 47.8, 21.8.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 4 h (2.5 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/dichloromethane (2:1) to afford **4** (45 mg, 76% yield) as a yellow oil. The spectra matched with the previous report.<sup>[1]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.37 (d, *J* = 8.6 Hz, 2H), 7.33 (d, *J* = 8.6 Hz, 2H), 4.57 (q, *J* = 7.0 Hz, 1H), 1.85 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 137.8, 135.0, 129.4, 128.5, 111.4, 47.8, 21.9.

**IR** (film, cm<sup>-1</sup>) 2931, 2152, 1511, 1229.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 4 h (2.5 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/dichloromethane (2:1) to afford **5** (37 mg, 68% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.37 (dd, *J* = 8.7, 5.1 Hz, 2H), 7.08 (t, *J* = 8.5 Hz, 2H), 4.60 (q, *J* = 7.0 Hz, 1H), 1.86 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 162.9 (d, *J* = 248.6 Hz), 135.1, 129.0 (d, *J* = 8.3 Hz), 116.2 (d, *J* = 21.9 Hz), 111.6, 47.8, 22.1.

<sup>19</sup>**F NMR** (376 MHz, CDCl<sub>3</sub>) δ -112.12.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 6 h (5.2 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **6** (34 mg, 51% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.40 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 4.61 (q, *J* = 7.0 Hz, 1H), 1.88 (d, *J* = 6.9 Hz, 3H), 1.32 (s, 9H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 152.2, 136.0, 126.9, 126.1, 112.1, 48.5, 34.8, 31.3, 22.2.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 4 h (2.5 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (20:1) to afford 7 (50 mg, 75% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H** NMR (500 MHz, CDCl<sub>3</sub>) δ 7.40 (d, *J* = 8.6 Hz, 2H), 7.12 (d, *J* = 8.6 Hz, 2H), 4.61 (q, *J* = 7.0 Hz, 1H), 2.31 (s, 3H), 1.87 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 169.3, 150.9, 136.6, 128.4, 122.3, 111.6, 47.9, 22.1, 21.2. IR (film, cm<sup>-1</sup>) 2931, 2151, 1770, 1200.



The reaction was conducted following the general procedure B in a 0.3 mmol scale for 5 h (3.1 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (10:1) to afford **8** (46 mg, 75% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 4.61 (q, *J* = 7.0 Hz, 1H), 2.61 (s, 3H), 1.88 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 197.3, 144.4, 137.5, 129.2, 127.4, 111.2, 47.8, 26.8, 21.7.
HRMS (ESI-TOF) m/z: [M + Na]<sup>+</sup> calcd for C<sub>11</sub>H<sub>11</sub>NOSNa, 228.0459, found 228.0460.
IR (film, cm<sup>-1</sup>) 2930, 2152, 1683, 1266.



The reaction was conducted following the general procedure B in a 0.3 mmol scale for 6 h (3.7 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **9** (49 mg, 73% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 8.04 (d, *J* = 8.7 Hz, 2H), 7.45 (d, *J* = 8.6 Hz, 2H), 4.60 (q, *J* = 7.0 Hz, 1H), 3.91 (s, 3H), 1.86 (d, *J* = 7.2 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 166.4, 144.1, 130.7, 130.4, 127.2, 111.2, 52.3, 47.9, 21.7. HRMS (ESI-TOF) m/z: [M + Na]<sup>+</sup> calcd for C<sub>11</sub>H<sub>11</sub>NO<sub>2</sub>SNa, 244.0408, found 244.0416.



The reaction was conducted following the general procedure B in a 0.3 mmol scale for 6 h (3.7 F/mol). The residue was purified by column chromatography on silica gel with dichloromethane to afford 10 (35 mg, 61% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.70 (d, *J* = 8.4 Hz, 2H), 7.51 (d, *J* = 8.2 Hz, 2H), 4.57 (q, *J* = 7.1 Hz, 1H), 1.87 (d, *J* = 7.1 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 144.6, 133.0, 128.0, 118.2, 113.1, 110.7, 47.5, 21.5.

**HRMS** (ESI-TOF) m/z:  $[M + Na]^+$  calcd for  $C_{10}H_8N_2SNa$ , 211.0306, found 211.0309.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 4 h (2.5 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/dichloromethane (2:1) to afford **11** (46 mg, 64% yield) as a yellow oil.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.62 (d, J = 8.0 Hz, 1H), 7.51 (d, J = 7.8 Hz, 1H), 7.39 (t, J = 7.6 Hz, 1H), 7.22 (t, J = 8.4 Hz, 1H), 4.95 (q, J = 7.0 Hz, 1H), 1.91 (d, J = 7.0 Hz, 3H).
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 138.3, 133.5, 130.3, 128.3, 127.7, 124.3, 111.0, 46.8, 21.5.



The reaction was conducted following the general procedure B in a 0.3 mmol scale for 5 h (3.1 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (20:1) to afford **12** (45 mg, 79% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.73 – 7.66 (m, 2H), 7.64 (d, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 4.89 (q, *J* = 7.0 Hz, 1H), 1.93 (d, *J* = 7.1 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 142.9, 133.7, 133.5, 129.3, 127.0, 116.7, 112.0, 110.2, 44.9, 21.4.

HRMS (ESI-TOF) m/z: [M + Na]<sup>+</sup> calcd for C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>SNa, 211.0306, found 211.0307. IR (film, cm<sup>-1</sup>) 2932, 2226, 2153, 1449.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 5 h (3.1 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/dichloromethane (2:1) to afford **13** (43 mg, 59% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.50 (s, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.25 – 7.22 (m, 1H), 4.51 (q, *J* = 7.0 Hz, 1H), 1.83 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 141.5, 132.2, 130.7, 130.2, 125.9, 123.1, 111.2, 47.7, 21.9. IR (film, cm<sup>-1</sup>) 2928, 2152, 1570, 1475.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 4 h (2.5 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **14** (44 mg, 76% yield) as a yellow oil.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.30 (d, J = 8.2 Hz, 2H), 7.22 (d, J = 7.9 Hz, 2H), 4.61 (q, J = 7.0 Hz, 1H), 2.66 (q, J = 7.6 Hz, 2H), 1.88 (d, J = 7.0 Hz, 3H), 1.24 (t, J = 7.6 Hz, 3H).
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 145.4, 136.3, 128.7, 127.2, 112.1, 48.7, 28.7, 22.2, 15.5.
HRMS (ESI-TOF) m/z: [M + Na]<sup>+</sup> calcd for C<sub>11</sub>H<sub>13</sub>NSNa, 214.0666, found 214.0648.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 4 h (3.5 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **15** (50 mg, 79% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.42 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 4.62 – 4.59 (m, 1H), 4.58 (s, 2H), 1.87 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 139.4, 138.3, 129.3, 127.6, 111.6, 48.1, 45.6, 22.0.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 4 h (3.5 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **16** (65 mg, 88% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.30 (d, *J* = 7.9 Hz, 2H), 7.22 (d, *J* = 7.9 Hz, 2H), 4.61 (q, *J* = 7.0 Hz, 1H), 2.55 – 2.46 (m, 1H), 1.88 (d, *J* = 6.9 Hz, 3H), 1.88 – 1.72 (m, 4H), 1.47 – 1.36 (m, 4H), 1.31 – 1.22 (m, 2H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 149.1, 136.3, 127.6, 127.1, 112.1, 48.6, 44.3, 34.4, 26.9, 26.2, 22.2.

**HRMS** (ESI-TOF) m/z:  $[M + H]^+$  calcd for C<sub>15</sub>H<sub>20</sub>NS, 246.1316, found 246.1314.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 5 h (3.1 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **17** (30 mg, 52% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H** NMR (500 MHz, CDCl<sub>3</sub>) δ 7.40 – 7.29 (m, 5H), 4.08 (d, *J* = 9.2 Hz, 1H), 2.40 – 2.30 (m, 1H), 1.18 (d, *J* = 6.6 Hz, 3H), 0.90 (d, *J* = 6.7 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 138.5, 128.9, 128.7, 127.9, 112.1, 61.8, 33.7, 21.2, 20.6. IR (film, cm<sup>-1</sup>) 2965, 2151, 1454, 1390.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 4 h (2.5 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **18** (41 mg, 53% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.44 – 7.36 (m, 5H), 4.61 (dd, J = 8.6, 6.7 Hz, 1H), 3.49 (dt, J = 10.8, 5.9 Hz, 1H), 3.25 (ddd, J = 10.6, 7.9, 5.4 Hz, 1H), 2.74 – 2.63 (m, 2H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 136.9, 129.5, 127.6, 110.9, 51.0, 37.9, 29.6. IR (film, cm<sup>-1</sup>) 2926, 2152, 1454, 1258.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 4 h (5.2 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **19** (29 mg, 39% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.44 – 7.30 (m, 5H), 4.35 (t, *J* = 7.3 Hz, 1H), 4.07 (t, *J* = 6.6 Hz, 2H), 2.33 – 2.17 (m, 2H), 2.05 (s, 3H), 1.76 – 1.60 (m, 2H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 171.0, 137.9, 129.3, 129.2, 127.5, 111.4, 63.3, 53.1, 32.4, 26.6, 21.0.

HRMS (ESI-TOF) m/z: [M + Na]<sup>+</sup> calcd for C<sub>13</sub>H<sub>15</sub>NO<sub>2</sub>SNa, 272.0721, found 272.0724.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 7 h (6.1 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (10:1) to afford **20** (35 mg, 50% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.30 (m, 5H), 4.45 (dd, *J* = 8.8, 6.6 Hz, 1H), 3.66 (s, 3H), 2.59 – 2.42 (m, 2H), 2.41 – 2.34 (m, 2H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 172.4, 137.5, 129.4, 129.3, 127.5, 111.2, 52.4, 52.0, 31.5, 30.9.

**HRMS** (ESI-TOF) m/z:  $[M + Na]^+$  calcd for  $C_{12}H_{13}NO_2SNa$ , 258.0565, found 258.0567.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 5 h (3.1 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **21** (40 mg, 58% yield) as a yellow oil. The spectra matched with the previous report.<sup>[3]</sup>

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.53 (d, J = 8.4 Hz, 2H), 7.24 (d, J = 8.4 Hz, 2H), 4.10 (s, 2H).
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 133.5, 132.4, 130.7, 123.2, 111.6, 37.7.

**IR** (film, cm<sup>-1</sup>) 2928, 2154, 1488, 1012.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 8 h (7.0 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **22** (32 mg, 52% yield) as a yellow oil. The spectra matched with the previous report.<sup>[3]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.41 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 2H), 4.16 (s, 2H), 1.32 (s, 9H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 152.2, 131.3, 128.8, 126.2, 112.3, 38.3, 34.8, 31.3.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 6 h (3.7 F/mol). The residue was purified by column chromatography on silica gel with dichloromethane to afford **23** (63 mg, 83% yield) as a yellow oil.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.52 (d, J = 8.7 Hz, 2H), 7.41 (d, J = 8.7 Hz, 2H), 1.96 (s, 6H).
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 141.4, 132.0, 127.8, 122.8, 111.6, 56.0, 30.3.

**HRMS** (ESI-TOF) m/z:  $[M + H]^+$  calcd for C<sub>10</sub>H<sub>11</sub>BrNS, 255.9795, found 255.9801.



The reaction was conducted following the general procedure B in a 0.3 mmol scale for 5 h (3.1 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (10:1) to afford **24** (44 mg, 73% yield) as a yellow oil.

<sup>1</sup>**H** NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.08 (d, J = 7.7 Hz, 1H), 7.61 (t, J = 7.5 Hz, 1H), 7.55 – 7.43 (m, 2H), 5.02 (t, J = 3.6 Hz, 1H), 3.13 – 3.02 (m, 1H), 2.89 – 2.66 (m, 2H), 2.63 – 2.57 (m, 1H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 195.0, 138.0, 134.4, 132.2, 130.1, 129.5, 128.1, 111.1, 48.2, 33.8, 28.6.

**HRMS** (ESI-TOF) m/z:  $[M + Na]^+$  calcd for C<sub>11</sub>H<sub>9</sub>NOSNa, 226.0303, found 226.0302.

**IR** (film, cm<sup>-1</sup>) 2955, 2151, 1693, 1454.



The reaction was conducted following the general procedure B in a 0.3 mmol scale for 5 h (3.1 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (20:1) to afford **25** (55 mg, 62% yield) as a yellow oil.

<sup>1</sup>**H** NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.11 – 8.05 (m, 3H), 7.93 (d, J = 1.8 Hz, 1H), 7.56 – 7.50 (m, 3H), 7.52 (d, J = 2.3 Hz, 1H), 4.75 (q, J = 7.0 Hz, 1H), 1.96 (d, J = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 169.6, 154.4, 136.4, 135.8, 133.4, 131.4, 129.2, 127.7, 125.7, 123.8, 120.3, 111.6, 48.7, 22.3.

**HRMS** (ESI-TOF) m/z:  $[M + H]^+$  calcd for C<sub>16</sub>H<sub>13</sub>N<sub>2</sub>S<sub>2</sub>, 297.0520, found 297.0523.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 9 h (7.8 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **27** (43 mg, 41% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.58 (d, *J* = 3.7 Hz, 2H), 7.56 (d, *J* = 3.5 Hz, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.41 (d, *J* = 8.3 Hz, 2H), 4.39 (t, *J* = 8.0 Hz, 1H), 2.21 – 2.11 (m, 2H), 1.50 – 1.32 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 140.6, 139.2, 138.0, 132.1, 128.7, 128.1, 127.6, 122.1, 111.7, 53.2, 37.7, 20.8, 13.6.

**HRMS** (ESI-TOF) m/z:  $[M + Na]^+$  calcd for C<sub>17</sub>H<sub>16</sub>BrNSNa, 368.0084, found 368.0061.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 6 h (5.2 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (20:1) to afford **28** (42 mg, 51% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.23 (d, *J* = 8.5 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 3.99 (d, *J* = 8.9 Hz, 1H), 3.66 (q, *J* = 7.1 Hz, 1H), 3.61 (s, 3H), 2.39 – 2.19 (m, 1H), 1.43 (d, *J* = 7.2 Hz, 3H), 1.10 (d, *J* = 6.7 Hz, 3H), 0.83 (d, *J* = 6.7 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 174.8, 140.9, 137.4, 128.2, 128.1, 112.1, 61.6, 52.2, 45.2, 33.6, 21.2, 20.6, 18.6.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 6 h (5.2 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **29** (29 mg, 32% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.77 (s, 1H), 7.40 (s, 1H), 5.56 (t, *J* = 4.8 Hz, 1H), 2.65 (s, 3H), 2.52 (d, *J* = 5.7 Hz, 2H), 1.47 (s, 3H), 1.37 (s, 9H), 1.35 (s, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 199.7, 154.8, 154.1, 135.5, 133.3, 126.5, 124.3, 113.2, 50.7, 48.3, 43.2, 35.1, 31.4, 30.6, 30.1, 28.1.



The reaction was conducted following the general procedure C in a 0.3 mmol scale for 8 h (7.0 F/mol). The residue was purified by column chromatography on silica gel with dichloromethane to afford **30** (59 mg, 40% yield) as a yellow oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.70 (d, *J* = 8.5 Hz, 2H), 7.64 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 6.77 (d, *J* = 8.8 Hz, 2H), 5.19 (s, 2H), 4.55 (q, *J* = 7.0 Hz, 1H), 1.83 (d, *J* = 7.0 Hz, 3H), 1.69 (s, 6H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 194.2, 173.5, 159.5, 139.6, 138.6, 136.4, 136.0, 132.0, 131.3, 130.5, 129.1, 128.7, 127.4, 117.3, 111.5, 79.5, 66.8, 48.1, 25.6, 25.5, 21.9.

**HRMS** (ESI-TOF) m/z:  $[M + H]^+$  calcd for C<sub>27</sub>H<sub>25</sub>ClNO<sub>4</sub>S, 494.1193, found 494.1193.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 6 h (3.7 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **34** (30 mg, 57% yield) as a yellow oil. The spectra matched with the previous report.<sup>[3]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.27 (d, *J* = 8.3 Hz, 2H), 7.19 (d, *J* = 7.8 Hz, 2H), 4.60 (q, *J* = 6.9 Hz, 1H), 2.35 (s, 3H), 1.86 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 139.0, 136.1, 129.8, 127.1, 112.0, 48.6, 22.1, 21.3.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 6 h (3.7 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **35** (41 mg, 66% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.30 (d, J = 8.3 Hz, 2H), 7.24 (d, J = 8.3 Hz, 2H), 4.61 (q, J = 7.0 Hz, 1H), 2.96 - 2.86 (m, 1H), 1.88 (d, J = 7.0 Hz, 3H), 1.25 (d, J = 6.9 Hz, 6H).
<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 150.0, 136.4, 127.3, 127.2, 112.1, 48.6, 34.0, 23.9, 22.2.



The reaction was conducted following the general procedure A in a 0.3 mmol scale for 6 h (3.7 F/mol). The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to afford **36** (42 mg, 52% yield) as a yellow oil. The spectra matched with the previous report.<sup>[2]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.30 (d, *J* = 8.3 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 2.36 (s, 3H), 1.77 (s, 6H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 141.2, 137.6, 132.1, 129.4, 124.3, 63.8, 32.0, 21.1. HRMS (ESI-TOF) m/z: [M + H]<sup>+</sup> calcd for C<sub>11</sub>H<sub>14</sub>NS, 192.0847, found 192.0835.

## 5. Cyclic voltammetry studies

Cyclic voltammograms (CV) were performed with a CS electrochemical workstation (CS300H, CorrTest, China). CV analysis conditions: working electrode: glassy carbon disk; counter electrode: Pt wire; reference electrode: The reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution.



Figure S3. Cyclic voltammetry experiments

Solvent: DCE; scan rate: v = 100 mV/s; c = 0.05 M; electrolyte:  $n\text{Bu}_4\text{NClO}_4$  (0.1 M).



Figure S4. Cyclic voltammetry experiments

Solvent: DCE/HFIP (3:1); scan rate: v = 100 mV/s; c = 0.05 M; electrolyte:  $nBu_4NClO_4$  (0.1 M).

#### 6. Follow-up transformations



To a solution of **3** (48 mg, 0.2 mmol, 1.0 equiv),  $ZnCl_2$  (27 mg, 0.2 mmol, 1.0 equiv) in *i*-PrOH (1.0 mL) was added NaN<sub>3</sub> (33 mg, 0.5 mmol, 2.5 equiv) in one portion. The reaction mixture was stirred at 50°C for 11 h. After cooling, the mixture was acidified with 10% HCl. The aqueous layer was extracted with ethyl acetate. The combined organic layer was washed with H<sub>2</sub>O and brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and evaporated. The residue was purified by column chromatography on silica gel (DCM/MeOH = 5:1) to give the desired product **31** (46 mg, 80% yield) as a colorless oil.

<sup>1</sup>**H** NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  9.07 (s, 1H), 7.62 – 6.67 (m, 4H), 4.70 (s, 1H), 1.52 (d, J = 13.9 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 155.5, 140.6, 131.7, 128.9, 121.6, 46.5, 22.3.

HRMS (ESI-TOF) m/z: [M + Na]<sup>+</sup> calcd for C<sub>9</sub>H<sub>9</sub>BrN<sub>4</sub>SNa, 306.9629, found 306.9639.



To a solution of **3** (48 mg, 0.2 mmol, 1.0 equiv) in CH<sub>3</sub>CN (1.5 mL) was added TMSCF<sub>3</sub> (57 mg, 0.4 mmol, 2.0 equiv). The reaction mixture was stirred at room temperature for 6 h. After completion of the reaction (monitored by TLC), water (5 mL) was added to quench the reaction and the mixture was extracted with DCM. The organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and evaporated. The residue was purified by column chromatography on silica gel with petroleum ether to give the desired product **32** (47 mg, 82% yield) as a colorless oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.48 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 4.48 (q, *J* = 7.1 Hz, 1H), 1.69 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 140.6, 132.0, 130.4 (q, *J* = 307.4 Hz), 128.8, 121.9, 43.9, 22.9.
<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -40.12.



To a solution of **3** (48 mg, 0.2 mmol, 1.0 equiv), diphenylphosphine oxide (61 mg, 0.3 mmol, 1.5 equiv) in toluene (1.5 mL) was added DBU (46 mg, 0.3 mmol, 1.5 equiv). The reaction mixture was srirred at room temperature for 3 h. After completion of the reaction (monitored by TLC), the solvent was removed under vacuum. The residue was purified by column chromatography on silica gel petroleum ether/ethyl acetate (1:1) to give the desired product **33** (63 mg, 75% yield) as a colorless oil.

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.78 (dd, *J* = 12.2, 7.6 Hz, 2H), 7.70 (dd, *J* = 12.4, 7.7 Hz, 2H), 7.51 – 7.38 (m, 4H), 7.35 – 7.30 (m, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.02 (d, *J* = 8.2 Hz, 2H), 4.52 – 4.43 (m, 1H), 1.67 (d, *J* = 7.1 Hz, 3H).

<sup>13</sup>**C** NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  142.0 (d, J = 3.4 Hz), 134.2, 133.4, 132.8, 132.3 (d, J = 2.8 Hz), 131.9 (d, J = 3.0 Hz), 131.8 (d, J = 10.5 Hz), 131.4, 131.1 (d, J = 10.4 Hz), 128.8, 128.5 (q, J = 37.7 Hz), 121.1, 43.6 (d, J = 2.1 Hz), 24.4 (d, J = 4.5 Hz).

<sup>31</sup>**P NMR** (162 MHz, CDCl<sub>3</sub>) δ 41.51.

HRMS (ESI-TOF) m/z: [M + Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>18</sub>BrOPSNa, 438.9897, found 438.9894.

#### 7. Synthesis of substrates

#### General procedure for esterification (GP1)

$$R \rightarrow OH \rightarrow H_2SO_4 \rightarrow OH \rightarrow R \rightarrow OMe$$

To a solution of acid substrate (2.5 mmol, 1.0 equiv) in methanol (10 mL) was added concentrated sulfuric acid (0.05 mL) and the reaction mixture stirred at room temperature for 16 h. After concentration to remove methanol, the oil was dissolved in ethyl acetate (50 mL), washed with sat. Na<sub>2</sub>CO<sub>3</sub> (2 x 50 mL), water (50 mL), and brine (50 mL). The crude was purified via column chromatography.



Following GP1 with ibuprofen. The crude was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (20:1) to give **S1** (0.48 g, 87%) as a colorless oil. The NMR spectrum is consistent with literature report.<sup>[4]</sup>



Following GP1 with 4-phenylbutyric acid. The crude was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (10:1) to give **S2** (0.38 g, 85%) as a colorless oil. The NMR spectrum is consistent with literature report.<sup>[5]</sup>

#### General procedure for acetate protection (GP2)

$$R-OH + OO O DMAP R-OAc$$

To a round-bottom flask was added the alcohol substrate (2.5 mmol, 1.0 equiv), acetic anhydride (5 mmol, 2.0 equiv), 4-dimethylaminopyridine (0.13 mmol, 0.05 equiv) and pyridine (2.5 mL). The reaction was stirred overnight at room temperature under N<sub>2</sub>, then slowly quenched with sat. NaHCO<sub>3</sub>. The mixture was extracted with Et<sub>2</sub>O (3 x 50 mL). The organic extracts were combined and washed with sat. CuSO<sub>4</sub> (50 mL), H<sub>2</sub>O (50 mL), brine (50 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The residue was purified by column chromatograph on silica gel to afford the pure product.



Following GP2 with benzenebutanol. The crude was purified with petroleum ether/ethyl acetate (40:1) to give **S3** (0.43 g, 90%) as a colorless oil. The NMR spectrum is consistent with literature report.<sup>[6]</sup>



Following GP2 with 4-ethylphenol. The crude was purified with petroleum ether/ethyl acetate (20:1) to give **S4** (0.35 g, 85%) as a colorless oil. The NMR spectrum is consistent with literature report.<sup>[7]</sup>



To a round-bottom flask was added fenofibric acid (0.8 g, 2.5 mmol, 1.0 equiv), 1-(bromomethyl)-4-ethylbenzene (0.5 g, 2.5 mmol, 1.0 equiv), K<sub>2</sub>CO<sub>3</sub> (0.69 g, 5 mmol, 2.0

equiv) and DMF (5 mL). The reaction was stirred overnight at 130°C. The mixture was quenched with water and extracted with Et<sub>2</sub>O (3 x 50 mL). The organic layer was washed with brine (50 mL), dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated. The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to give **S5** (0.85 g, 78%) as a colorless oil.<sup>[8]</sup>

<sup>1</sup>**H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.69 (d, *J* = 8.5 Hz, 2H), 7.63 (d, *J* = 8.8 Hz, 2H), 7.44 (d, *J* = 8.5 Hz, 2H), 7.18 (d, *J* = 8.3 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 2H), 6.77 (d, *J* = 8.8 Hz, 2H), 5.17 (s, 2H), 2.60 (q, *J* = 7.6 Hz, 2H), 1.67 (s, 6H), 1.18 (t, *J* = 7.6 Hz, 3H).

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 194.2, 173.5, 159.6, 144.8, 138.4, 136.5, 132.4, 132.0, 131.2, 130.3, 128.7, 128.6, 128.1, 117.4, 79.5, 67.4, 28.6, 25.5, 15.5.



To a round-bottom flask was added 4-ethyl-2-iodobenzenamine (0.62 g, 2.5 mmol, 1.0 equiv), benzaldehyde (0.32 g, 3.0 mmol, 1.2 equiv), sulfer powder (1.9 g, 7.5 mmol, 3.0 equiv),  $CuCl_2 \cdot 2H_2O$  (43 mg, 0.25 mmol, 0.1 equiv), 1,10-phenanthroline (45 mg, 0.25 mmol, 0.1 equiv) and K<sub>2</sub>CO<sub>3</sub> (0.69 g, 5.0 mmol, 2.0 equiv) in water (25 mL). The reaction mixture was stirred at 100°C for 24 h and then cooled to room temperature and extracted with ethyl acetate. The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (100:1) to give **S6** (0.67 g, 90% yield) as a yellow solid. The NMR spectrum is consistent with literature report.<sup>[9]</sup>



To a round-bottom flask was added 4-tert-butylstyrene (0.32 g, 2 mmol, 1.0 equiv), Pb(OAc)<sub>2</sub> (0.2 mol%), HBPin (0.28 g, 2.2 mmol, 1.1 equiv), H<sub>2</sub>O (0.04 g, 2.2 mmol, 1.1 equiv), and DCM (5 mL). The reaction was stirred at room temperature for 12 h. After completion of the reaction, the mixture was evaporated, dissolved in ethyl acetate, the combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>. The combined organic layer was evaporated and purified by column chromatography on silica gel with petroleum ether/ethyl acetate (40:1) to give **S7** (0.29 g, 90% yield) as a colorless oil. The NMR spectrum is consistent with literature report.<sup>[10]</sup>



To a 100 mL round bottom flask were added Pd/C (10% Pd on activated charcoal, 97 mg, 20% w/w), 4-isopropylacetophenone (0.49 g, 3 mmol), and methanol (12 mL). The solution was sparged with H<sub>2</sub> and stirred under 1 atm H<sub>2</sub> for 48 hours. The crude reaction mixture was filtered through a pad of Celite, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The residue was purified by column chromatography on silica gel with petroleum ether to afforded **S8** (0.4 g, 90% yield) as a colorless oil. The NMR spectrum is consistent with literature report.<sup>[11]</sup>



To a round bottom flask was added 1-bromo-2-ethylbenzene (0.56 g, 3 mmol, 1.0 equiv), CuCN (0.67 g, 7.5 mmol, 2.5 equiv), and NMP (4 mL). The mixture was heated at 190°C for 20 mins. The reaction mixture was cooled to room temperature and quenched with 10% NH4OH and CHCl<sub>3</sub>. The organic layer was evaporated and purified by column chromatography on silica gel with petroleum ether/ethyl acetate (20:1) to give **S9** (0.24 g, 60% yield) as a colorless oil. The NMR spectrum is consistent with literature report.<sup>[12]</sup>

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# 9. NMR spectra





S24





100 90 fl (ppm) 170 160  $\frac{1}{70}$  $\frac{1}{40}$ 



20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2 f1 (ppm)











190 180 100 90 fl (ppm)  $\frac{1}{70}$ 





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100 90 fl (ppm)  $\frac{1}{70}$  $\frac{1}{40}$ 



























S45





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S50









CDCl<sub>3</sub>



-4.70

 $\underset{(1.51)}{\overset{1.54}{\leftarrow}}$ 









150 130 110 90 70 50 30 10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230 -2£ f1 (ppm)







-- (PP)....