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Supporting Information

## Electroreductive deoxygenative carboxylation of alkyl oxalates with CO<sub>2</sub>

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#### **1. General Information**

All glassware was oven dried at 110 °C for h and cooled down under vacuum. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Flash column chromatography was performed with silica gel (200-300 mesh). Cyclic voltammograms were recorded on a CHI660E potentiostat. <sup>1</sup>H NMR, <sup>13</sup>C NMR, and <sup>19</sup>F NMR experiments were carried out using Vnmr Mercury plus 400 MHz or Agilent DD2-600 MHz spectrometers. All chemical shifts ( $\delta$ ) are reported in ppm relative to internal tetramethyl silane (TMS, 0 ppm) for <sup>1</sup>H, CDCl<sub>3</sub> (77.16 ppm) or DMSO-*d*<sub>6</sub> (39.52 ppm) for <sup>13</sup>C. The abbreviations used for explaining the multiplicities were as follows: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet, br = broad. Coupling constants (*J*) are reported in Hz. High-resolution mass spectra (HRMS) spectra were obtained from the Thermo Fisher Q-Exactive mass spectrometer in electrospray ionization (ESI<sup>+</sup>) mode.

#### 2. Experimental Details

2.1 General procedure for the synthesis of alkyl oxalates <sup>[1]</sup>

The alcohols (5.0 mmol), pyridine (0.6 mL, 7.5 mmol, 1.5 equiv.), and 4dimethylamino pyridine (DMAP) (61 mg, 0.5 mmol, 0.1 equiv.) were dissolved in 25 mL anhydrous CH<sub>2</sub>Cl<sub>2</sub> and methyl oxalyl chloride (0.7 mL, 7.5 mmol, 1.5 equiv.) was next added slowly. The reaction mixture was stirred at room temperature until the reaction was complete (monitored by TLC). The resulting mixture was quenched with saturated NaHCO<sub>3</sub> solution and extracted with DCM ( $3 \times 30$  mL). The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. Then the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 20/1 to 10/1) to obtain the desired alkyl oxalates.

### 2.2 General procedure for the electroreductive deoxygenative carboxylation of alkyl oxalates with CO<sub>2</sub>

$$R^{O} + CO_{2} \xrightarrow{n Bu_{4}NOAc, 15 mA, 4 h}{DMF, RT, undivided cell} R-COOH$$

In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, alkyl oxalates (0.5 mmol, 1.0 equiv.) and "Bu<sub>4</sub>NOAc (151 mg, 0.5 mmol, 1.0 equiv.) were combined and added. The bottle was equipped with an aluminum plate (15.0 mm  $\times$  15.0 mm  $\times$  0.5 mm) anode and a stainless steel plate (15.0 mm  $\times$  15.0 mm  $\times$  1.0 mm) cathode and was then charged with CO<sub>2</sub>. Under the atmosphere of carbon dioxide, DMF (11 mL) was injected into the tube by a syringe. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA for 4 h. After electrolysis, the mixture was acidized by HCl solution (10 mL, 2M) and extracted by ethyl acetate for 4 times (4  $\times$  15 mL). The combined organic layers were washed with saturated saline solution (2  $\times$  15 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. Then

the residue was purified by flash column chromatography (petroleum ether/ethyl acetate/acetic acid = 10/1/0.005 to 5/1/0.005) to obtain the desired products.



#### 2.3 Procedure for the gram scale synthesis of 2

In an oven-dried undivided three-necked bottle equipped with a stir bar, methyl (1-phenylethyl) oxalate (20 mmol, 4.16 g) and "Bu<sub>4</sub>NOAc (5 mmol, 1.51 g) were combined and added. The bottle was equipped with two aluminum plates (15 mm  $\times$  30 mm  $\times$  0.5 mm) anodes and two stainless steel plates (15 mm  $\times$  30 mm  $\times$  1.0 mm) cathodes and was then charged with carbon dioxide. Under the atmosphere of carbon dioxide, DMF (75 mL) was injected into the tube by a syringe. The reaction mixture was stirred and electrolyzed at a constant current of 30 mA for 40 h. After electrolysis, the mixture was acidized by HCl solution (50 mL, 2M) and extracted by ethyl acetate for 4 times (4  $\times$  60 mL). The combined organic layers were washed with saturated saline solution (2  $\times$  30 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. Then the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 5/1) to obtain the desired product as a colorless oil (73%).

#### 2.4 Procedure for the gram scale synthesis of 27



Biprofen



In an oven-dried undivided three-necked bottle equipped with a stir bar, 1-([1,1'biphenyl]-4-yl)ethyl methyl oxalate (20 mmol, 5.69 g) and "Bu4NOAc (5 mmol, 1.51 g) was added. The bottle was equipped with two aluminum plates (15 mm × 30 mm × 0.5 mm) anodes and two stainless steel plates (15 mm × 30 mm × 1.0 mm) cathodes and was then charged with carbon dioxide. Under the atmosphere of carbon dioxide, DMF (75 mL) was injected into the tube by a syringe. The reaction mixture was stirred and electrolyzed at a constant current of 30 mA for 40 h. After electrolysis, the mixture was acidized by HCl solution (50 mL, 2M) and extracted by ethyl acetate for 4 times (4 × 60 mL). The combined organic layers were washed with saturated saline solution (2 × 30 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. Then the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 5/1) to obtain the desired biprofen as a white solid (63%). 2.5 The experimental setup for the gram scale synthesis







#### **3.** Mechanistic Investigations

#### 3.1 Cyclic voltammetry study

Cyclic voltammetry (CV) experiments were conducted in an electrolyte of <sup>*n*</sup>Bu<sub>4</sub>NOAc (0.1 M) in DMF using a glassy carbon disk working electrode (diameter, 1 mm), a Pt wire auxiliary electrode and a Ag/AgCl reference electrode. The scan rate is 100 mV/s.



Fig. S1. Cyclic voltammogram of 1,  $CO_2$  and their mixture in DMF. Conditions: <sup>*n*</sup>Bu<sub>4</sub>NOAc (0.1 M in DMF), and with (a)  $CO_2$  saturated, (b) 1 (1.0 mM), or (c) 1 (1.0 mM) +  $CO_2$  saturated. Scan rate: 100 mV/s.

#### **3.2 Radical trapping by TEMPO**



In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, methyl (1-phenylethyl) oxalate (104 mg, 0.5 mmol, 1.0 equiv.), "Bu4NOAc (151 mg, 0.5 mmol, 1.0 equiv.) and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO, 156 mg, 1.0 mmol, 2.0 equiv.) were combined and added. The bottle was equipped with an aluminum plate (15.0 mm  $\times$  15.0 mm  $\times$  0.5 mm) anode and a stainless steel plate (15.0 mm  $\times$  15.0 mm  $\times$  0.5 mm) anode and a stainless steel plate (15.0 mm  $\times$  15.0 mm  $\times$  1.0 mm) cathode and was then charged with CO<sub>2</sub>. Under the atmosphere of carbon dioxide, DMF (11 mL) was injected into the tube by a syringe. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA for 4 h. After electrolysis, the mixture was acidized by HCl solution (10 mL, 2M) and extracted by ethyl acetate for 4 times (4  $\times$  15 mL). The combined organic layers were washed with saturated saline solution (2  $\times$  15 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. The desired product was obtained by HRMS.



#### 3.3 Radical trapping by 1,1-diphenylethylene



In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, methyl (1-phenylethyl) oxalate (104 mg, 0.5 mmol, 1.0 equiv.) and "Bu<sub>4</sub>NOAc (151 mg, 0.5 mmol, 1.0 equiv.) were combined and added. The bottle was equipped with an aluminum plate (15.0 mm × 15.0 mm × 0.5 mm) anode and a stainless steel plate (15.0 mm × 15.0 mm × 15.0 mm) cathode and was then charged with CO<sub>2</sub>. Under the atmosphere of carbon dioxide, 1,1-diphenylethylene (177  $\mu$ L, 1.0 mmol, 2.0 equiv.) and DMF (11 mL) were injected respectively into the tube by syringes. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA for 4 h. After electrolysis, the mixture was acidized by HCl solution (10 mL, 2M) and extracted by ethyl acetate

for 4 times ( $4 \times 15$  mL). The combined organic layers were washed with saturated saline solution ( $2 \times 15$  mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. The desired product was obtained by flash column chromatography on silica gel and other intermediates were detected by HRMS.



#### 3.4 D-labeling experiment





In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, methyl (1-(naphthalen-1-yl)ethyl) oxalate (129 mg, 0.5 mmol, 1.0 equiv.) and <sup>*n*</sup>Bu<sub>4</sub>NOAc (151 mg, 0.5 mmol, 1.0 equiv.) were combined and added. The bottle was equipped with an aluminum plate (15.0 mm  $\times$  15.0 mm  $\times$  0.5 mm) anode and a stainless

steel plate (15.0 mm  $\times$  15.0 mm  $\times$  1.0 mm) cathode and was then charged with CO<sub>2</sub>. Under the atmosphere of carbon dioxide, D<sub>2</sub>O (45 µL, 2.5 mmol, 5.0 equiv.) and DMF (11 mL) were injected respectively into the tube by syringes. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA for 4 h. After electrolysis, the mixture was acidized by HCl solution (10 mL, 2M) and extracted by ethyl acetate for 4 times (4  $\times$  15 mL). The combined organic layers were washed with saturated saline solution (2  $\times$  15 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. The pure products were obtained by flash column chromatography on silica gel.

#### 4. Characterization of the products



**2-Phenylpropanoic acid (2)** <sup>[2]</sup>. The desired pure product was obtained in 86% yield (65 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.38 - 7.17 (m, 5H), 3.73 (q, J = 7.2 Hz, 1H), 1.51 (d, J = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.9, 139.7, 128.6, 127.6, 127.4, 45.4, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>9</sub>H<sub>11</sub>O<sub>2</sub> 151.0754. Found: 151.0753.



**2-(***p***-Tolyl)propanoic acid (3)** <sup>[2]</sup>. The desired pure product was obtained in 67% yield (55 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.20 (d, *J* = 7.8 Hz, 2H), 7.13 (d, *J* = 7.9 Hz, 2H), 3.69 (q, *J* = 7.2 Hz, 1H), 2.32 (s, 3H), 1.48 (d, *J* = 7.3 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.8, 137.0, 136.8, 129.3, 127.4, 44.9, 21.0, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> 165.0910. Found: 165.0909



**2-(***m***-Tolyl)propanoic acid (4)** <sup>[2]</sup>. The desired pure product was obtained in 58% yield (48 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.27 - 7.18 (m, 1H), 7.16 - 7.05 (m, 3H), 3.70 (q, *J* = 7.2 Hz, 1H), 2.34 (s, 3H), 1.49 (d, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.5, 139.7, 138.3, 128.6, 128.3, 128.1, 124.6, 45.2, 21.4, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> 165.0910. Found: 165.0913.

Соон

**2-(***o***-Tolyl)propanoic acid (5)** <sup>[2]</sup>. The desired pure product was obtained in 63% yield (52 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 - 7.13 (m, 4H), 3.98 (q, J = 7.1 Hz, 1H), 2.37 (s, 3H), 1.48 (d, J = 7.1 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.9, 138.3, 135.9, 130.5, 127.2, 126.5, 126.4, 41.1, 19.6, 17.5. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> 165.0910. Found: 165.0911.



**2-(4-Methoxyphenyl)propanoic acid (6)** <sup>[3]</sup>. The desired pure product was obtained in 57% yield (51 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 - 7.21 (m, 2H), 6.90 - 6.83 (m, 2H), 3.80 (s, 3H), 3.68 (q, *J* = 7.2 Hz, 1H), 1.47 (d, *J* = 6.4 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  181.2, 158.8, 131.8, 128.6, 114.0, 55.2, 44.5, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>13</sub>O<sub>3</sub> 181.0859. Found: 181.0861.



**2-(4-Fluorophenyl)propanoic acid (7)** <sup>[4]</sup>. The desired pure product was obtained in 76% yield (64 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 - 7.24 (m, 2H), 7.05 - 6.97 (m, 2H), 3.72 (q, *J* = 7.0 Hz, 1H), 1.50 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.1, 162.1 (d, *J* = 246.1 Hz), 135.4 (d, *J* = 3.2 Hz), 129.1 (d, *J* = 8.1 Hz), 115.5 (d, *J* = 21.0 Hz), 44.5, 18.2. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -115.24. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>9</sub>H<sub>10</sub>FO<sub>2</sub> 169.0659. Found: 169.0660.



**2-(4-Chlorophenyl)propanoic acid (8)** <sup>[4]</sup>. The desired pure product was obtained in 83% yield (77 mg) as a white solid. M.P. = 57 °C - 59 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 - 7.19 (m, 4H), 3.71 (q, *J* = 7.2 Hz, 1H), 1.49 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.5, 138.1, 133.3, 129.0, 128.8, 44.7, 18.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>9</sub>H<sub>10</sub>ClO<sub>2</sub> 185.0364. Found: 185.0365.



**2-(4-Bromophenyl)propanoic acid (9)** <sup>[5]</sup>. The desired pure product was obtained in 71% (81 mg) yield as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.45 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 3.70 (q, *J* = 7.2 Hz, 1H), 1.49 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.2, 138.6, 131.8, 129.3, 121.4, 44.8, 18.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>9</sub>H<sub>10</sub>BrO<sub>2</sub> 228.9859. Found: 228.9861.



**2-(4-(Trifluoromethyl)phenyl)propanoic acid (10)** <sup>[4]</sup>. The desired pure product was obtained in 88% yield (96 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.59 (d, *J* = 8.1 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 3.81 (q, *J* = 7.2 Hz, 1H), 1.54 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.1, 143.5, 129.8 (q, *J* = 30.8 Hz), 128.1, 126.8 (q, *J* = 3.2 Hz), 124.0 (q, *J* = 271.0 Hz), 45.2, 18.0. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -62.62. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>10</sub>F<sub>3</sub>O<sub>2</sub> 219.0627. Found: 219.0629.



**2-Phenylbutanoic acid (11)** <sup>[6]</sup>. The desired pure product was obtained in 88% yield (72 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.36 - 7.23 (m, 5H), 3.45 (t, *J* = 7.7 Hz, 1H), 2.17 - 1.70 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)

 $\delta$  180.3, 138.3, 128.6, 128.1, 127.4, 53.3, 26.3, 12.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> 165.0910. Found: 165.0912.



**2-Phenylpentanoic acid (12)** <sup>[2]</sup>. The desired pure product was obtained in 76% yield (68 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.38 - 7.26 (m, 5H), 3.55 (t, *J* = 7.7 Hz, 1H), 2.12 - 1.68 (m, 2H), 1.34 - 1.22 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.4, 138.5, 128.6, 128.0, 127.4, 51.3, 35.2, 20.6, 13.8. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>11</sub>H<sub>15</sub>O<sub>2</sub> 179.1067. Found: 179.1066.



**4-Ethoxy-4-oxo-2-phenylbutanoic acid (13).** The desired pure product was obtained in 74% yield (82 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.36 - 7.27 (m, 5H), 4.15 - 4.07 (m, 3H), 3.16 (dd, *J* = 16.9, 10.0 Hz, 1H), 2.67 (dd, *J* = 16.9, 5.5 Hz, 1H), 1.20 (t, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 178.5, 171.3, 137.0, 128.9, 127.9, 127.8, 60.9, 47.1, 37.4, 14.0. HRMS (ESI) m/z: [M - H]<sup>-</sup> calcd for C<sub>9</sub>H<sub>13</sub>O<sub>4</sub> 221.0808. Found: 221.0811.



**2-Cyclopropyl-2-phenylacetic acid (14)** <sup>[7]</sup>. The desired pure product was obtained in 70% yield (62 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.43 - 7.27 (m, 5H), 2.82 (d, *J* = 10.2 Hz, 1H), 1.55 - 1.42 (m, 1H), 0.75 - 0.53 (m, 2H), 0.47 - 0.15 (m, 2H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.4, 138.2, 128.6, 128.0, 127.4, 56.4, 13.8, 4.9, 4.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>11</sub>H<sub>13</sub>O<sub>2</sub> 177.0910. Found: 177.0911.

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**2-Cyclobutyl-2-phenylacetic acid (15).** The desired pure product was obtained in 66% yield (63 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.36 - 7.25 (m, 5H), 3.57 (d, *J* = 11.0 Hz, 1H), 3.06 - 2.94 (m, 1H), 2.29 - 2.17 (m, 1H), 1.95 - 1.78 (m, 4H), 1.67 - 1.53 (m, 1H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 179.8, 137.0, 128.5, 128.2, 127.4, 58.1, 38.0, 27.5, 26.3, 17.8. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>15</sub>O<sub>2</sub> 191.1067. Found: 191.1068.



**1,2,3,4-Tetrahydronaphthalene-1-carboxylic acid (16)** <sup>[8]</sup>. The desired pure product was obtained in 71% yield (63 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.25 - 7.05 (m, 4H), 3.84 (t, *J* = 5.7 Hz, 1H), 2.89 - 2.70 (m, 2H), 2.24 - 2.10 (m, 1H), 2.09 - 1.90 (m, 2H), 1.84 - 1.71 (m, 1H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  181.4, 137.3, 132.5, 129.6, 129.4, 127.1, 125.8, 44.4, 29.0, 26.4, 20.4. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>11</sub>H<sub>13</sub>O<sub>2</sub> 177.0910. Found: 177.0912.



**6,7,8,9-Tetrahydro-5H-benzo**[7]**annulene-5-carboxylic acid (17)** <sup>[8]</sup>. The desired pure product was obtained in 74% yield (70 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.19 - 7.06 (m, 4H), 3.94 (dd, J = 7.4, 2.3 Hz, 1H), 2.92 - 2.70 (m, 2H), 2.26 - 2.13 (m, 1H), 1.93 - 1.70 (m, 4H), 1.54 (d, J = 11.6 Hz, 1H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.1, 143.0, 138.7, 130.0, 129.0, 127.3, 126.2, 51.5, 36.2, 30.0, 28.6, 27.5. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>15</sub>O<sub>2</sub> 191.1067. Found: 191.1066.



**2,2-Diphenylacetic acid** (18) <sup>[8]</sup>. The desired pure product was obtained in 75% yield (80 mg) as a white solid. M.P. = 147 °C - 149 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.37 - 7.25 (m, 10H), 5.04 (s, 1H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$ 178.7, 137.9, 128.6, 127.5, 57.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>14</sub>H<sub>13</sub>O<sub>2</sub> 213.0910. Found: 213.0911.



**2-(Naphthalen-1-yl)propanoic acid (19)** <sup>[9]</sup>. The desired pure product was obtained in 90% yield (90 mg) as a white solid. M.P. =  $122 \circ C - 124 \circ C$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.11 (d, *J* = 8.3 Hz, 1H), 7.85 (dd, *J* = 34.1, 7.8 Hz, 2H), 7.62 - 7.42 (m, 4H), 4.56 (q, *J* = 7.1 Hz, 1H), 1.69 (d, *J* = 7.3 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  181.1, 135.9, 133.9, 131.3, 129.0, 128.0, 126.4, 125.7, 125.5, 124.6, 123.0, 41.0, 17.8. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>13</sub>O<sub>2</sub> 201.0910. Found: 201.0912.



**2-(Naphthalen-2-yl)propanoic acid (20)** <sup>[9]</sup>. The desired pure product was obtained in 78% yield (78 mg) as a white solid. M.P. = 129 °C - 130 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.89 - 7.74 (m, 4H), 7.54 - 7.42 (m, 3H), 3.93 (q, *J* = 7.1 Hz, 1H), 1.62 (d, *J* = 7.6 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 180.7, 137.1, 133.4, 132.7, 128.4, 127.8, 127.6, 126.3, 126.2, 125.9, 125.7, 45.5, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>13</sub>O<sub>2</sub> 201.0910. Found: 201.0911.



**1,2-Dihydroacenaphthylene-1-carboxylic acid (21)** <sup>[2]</sup>. The desired pure product was obtained in 68% yield (67 mg) as a white solid. M.P. = 256 °C - 257 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.67 (dd, *J* = 22.7, 8.1 Hz, 2H), 7.59 - 7.44 (m, 3H), 7.33 (d, *J* = 6.8 Hz, 1H), 4.62 (dd, *J* = 8.8, 4.0 Hz, 1H), 3.87 (dd, *J* = 17.4, 4.0 Hz, 1H), 3.64 (dd, *J* = 17.4, 8.7 Hz, 1H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  179.0, 142.9, 141.3, 138.1, 131.5, 128.1, 127.8, 124.1, 122.7, 120.6, 119.6, 48.2, 34.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>11</sub>O<sub>2</sub> 199.0754. Found: 199.0755.



**2-(3-((3,7-Dimethyloct-6-en-1-yl)oxy)phenyl)propanoic acid (22).** The desired pure product was obtained in 72% yield (110 mg) as colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.23 (t, *J* = 9.1 Hz, 1H), 6.98 - 6.73 (m, 3H), 5.11 (s, 1H), 4.06 - 3.89 (m, 2H), 3.70 (t, *J* = 7.7 Hz, 1H), 2.11 - 1.93 (m, 2H), 1.91 - 1.77 (m, 1H), 1.70 (d, *J* = 7.9 Hz, 5H), 1.61 (s, 3H), 1.50 (s, 2H), 1.32 - 1.14 (m, 2H), 0.99 - 0.91 (m, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  159.3, 141.2, 131.3, 129.6, 124.7, 119.7, 114.1, 113.1, 66.2, 45.3, 37.1, 36.2, 29.5, 25.7, 25.4, 19.6, 18.1, 17.7. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>19</sub>H<sub>29</sub>O<sub>3</sub> 305.2111. Found: 305.2110.



**2-(4-Isobutylphenyl)propanoic acid (23)** <sup>[8]</sup>. The desired pure product was obtained in 61% yield (63 mg) as a white solid. M.P. = 77 °C - 78 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.78 (d, J = 7.7 Hz, 2H), 7.66 (d, J = 7.7 Hz, 2H), 4.26 (q, J = 7.3 Hz, 1H), 3.00 (d, J

= 7.1 Hz, 2H), 2.45 - 2.34 (m, 1H), 2.05 (d, J = 7.1 Hz, 3H), 1.45 (d, J = 5.0 Hz, 6H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  181.1, 140.8, 136.9, 129.4, 127.3, 45.1, 45.0, 30.1, 22.4, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>19</sub>O<sub>2</sub> 207.1380. Found: 207.1381.



**2-(6-Methoxynaphthalen-2-yl)propanoic acid (24)** <sup>[8]</sup>. The desired pure product was obtained in 68% yield (78 mg) as a white solid. M.P. = 152 °C - 154 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.73 - 7.64 (m, 3H), 7.40 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.17 - 7.05 (m, 2H), 3.90 (s, 3H), 3.85 (q, *J* = 7.2 Hz, 1H), 1.58 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.7, 157.7, 134.8, 133.8, 129.3, 128.9, 127.2, 126.2, 126.1, 119.0, 105.6, 55.3, 45.2, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>14</sub>H<sub>15</sub>O<sub>3</sub> 231.1016. Found: 231.1023.



**2-(3-Phenoxyphenyl)propanoic acid** (**25**) <sup>[8]</sup>. The desired pure product was obtained in 82% yield (99 mg) as a white solid. M.P. = 169 °C - 170 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.36 - 7.23 (m, 3H), 7.14 - 6.97 (m, 5H), 6.88 (dd, *J* = 8.2, 2.4 Hz, 1H), 3.71 (q, *J* = 7.2 Hz, 1H), 1.50 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.3, 157.5, 156.9, 141.6, 129.9, 129.7, 123.4, 122.3, 119.0, 118.2, 117.5, 45.2, 18.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>15</sub>H<sub>15</sub>O<sub>3</sub> 243.1016. Found: 243.1014.



**2-(4-Cyclohexylphenyl)propanoic acid (26)** <sup>[8]</sup>. The desired pure product was obtained in 60% yield (70 mg) as a white solid. M.P. = 110 °C - 111 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.23 (d, *J* = 8.2 Hz, 2H), 7.16 (d, *J* = 8.2 Hz, 2H), 3.70 (q, *J* = 7.2 Hz, 1H), 2.54 - 2.41 (m, 1H), 1.92 - 1.67 (m, 6H), 1.49 (d, *J* = 7.2 Hz, 3H), 1.45 - 1.33 (m, 4H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.7, 147.2, 137.0, 127.4, 127.1, 44.9, 44.2, 34.4, 26.9, 26.1, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>15</sub>H<sub>21</sub>O<sub>2</sub> 233.1536. Found: 233.1535.



**2-([1,1'-Biphenyl]-4-yl)propanoic acid (27)** <sup>[8]</sup>. The desired pure product was obtained in 75% yield (85 mg) as a white solid. M.P. = 168 °C - 169 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.61 - 7.52 (m, 4H), 7.49 - 7.29 (m, 5H), 3.79 (q, *J* = 7.2 Hz, 1H), 1.55 (d, *J* = 7.0 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  180.5, 140.7, 140.4, 138.7, 128.7, 128.0, 127.4, 127.3, 127.1, 45.0, 18.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>15</sub>H<sub>15</sub>O<sub>2</sub> 227.1067. Found: 227.1066.



**2-Methyl-3-phenylpropanoic acid** (**28**) <sup>[10]</sup>. The desired pure product was obtained in 38% yield as (31 mg) a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.34 - 7.17 (m, 5H), 4.05 - 3.96 (m, 1H), 2.80 - 2.64 (m, 2H), 1.23 (d, *J* = 6.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  182.7, 139.0, 129.0, 128.4, 126.4, 41.2, 39.3, 16.4. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> 165.0910. Found: 165.0912.



**2-Phenylacetic acid (29)** <sup>[8]</sup>. The desired pure product was obtained in 64% yield (44 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.39 - 7.22 (m, 5H), 3.65 (s, 2H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  177.6, 133.2, 129.4, 128.6, 127.3, 41.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>8</sub>H<sub>9</sub>O<sub>2</sub> 137.0597. Found: 137.0598.



**2-(***p***-Tolyl)acetic acid (30)** <sup>[8]</sup>. The desired pure product was obtained in 44% yield (33 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.21 - 7.07 (m, 4H), 3.60 (s, 2H), 2.33 (s, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 178.2, 137.0, 130.2, 129.3, 129.2, 40.6, 21.1. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>9</sub>H<sub>11</sub>O<sub>2</sub> 151.0754. Found: 151.0755.



**2-(4-Methoxyphenyl)acetic acid (31)** <sup>[8]</sup>. The desired pure product was obtained in 53% yield (44 mg) as a white solid. M.P. = 84 °C - 86 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 - 7.16 (m, 2H), 6.93 - 6.82 (m, 2H), 4.62 (s, 1H), 3.80 (d, *J* = 7.1 Hz, 3H), 3.58 (s, 1H). <sup>13</sup>C NMR (151 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  173.9, 158.6, 135.0, 130.8, 128.4, 114.0, 113.9, 63.0, 55.5. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>9</sub>H<sub>11</sub>O<sub>3</sub> 167.0703. Found: 167.0705.

**2-(4-Chlorophenyl)acetic acid (32)**<sup>[11]</sup>. The desired pure product was obtained in 62% yield (53 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 - 7.17 (m, 4H), 3.61 (s, 2H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  177.6, 133.4, 131.6, 130.7, 128.8, 40.3. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>8</sub>H<sub>8</sub>ClO<sub>2</sub> 171.0207. Found: 171.0209.



**2-(4-Bromophenyl)acetic acid (33)** <sup>[11]</sup>. The desired pure product was obtained in 68% yield (73 mg) as a white solid. M.P. = 114 °C - 117 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.45 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 3.60 (s, 2H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  177.3, 132.1, 131.7, 131.1, 121.4, 40.4. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>8</sub>H<sub>8</sub>BrO<sub>2</sub> 214.9702. Found: 214.9701.



**2-(4-Cyanophenyl)acetic acid (34)**. The desired pure product was obtained in 62% yield (50 mg) as a white solid. M.P. = 145 °C - 150 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.64 (d, *J* = 8.2 Hz, 1H), 7.41 (d, *J* = 8.3 Hz, 1H), 3.73 (s, 1H), <sup>13</sup>C NMR (151 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  172.3, 141.4, 132.6, 131.1, 119.4, 110.0, 40.9. HRMS (ESI) m/z: [M - H]<sup>-</sup> calcd for C<sub>9</sub>H<sub>6</sub>NO<sub>2</sub> 160.0393. Found: 160.0393.



**2-(4-(Methoxycarbonyl)phenyl)acetic acid (35).** The desired pure product was obtained in 80% yield (78 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.01 (d, *J* = 8.4 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 3.91 (s, 2H), 3.71 (s, 2H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  176.5, 166.8, 138.3, 129.9, 129.5, 129.3, 52.1, 40.9. HRMS (ESI) m/z: [M - H]<sup>-</sup> calcd for C<sub>10</sub>H<sub>9</sub>O<sub>4</sub> 193.0495. Found: 193.0494.



**4-Phenylbutanoic acid** (**36**) <sup>[4]</sup>. The desired pure product was obtained in 28% yield (23 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 - 7.15 (m, 5H), 3.68 (t, *J* = 6.4 Hz, 2H), 2.71 (dd, *J* = 8.7, 6.7 Hz, 2H), 1.95 - 1.84 (m, 2H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  179.6, 141.8, 128.4, 128.3, 125.8, 62.1, 34.1, 32.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> 165.0910. Found: 165.0913



**2-Methyl-2-phenylpropanoic acid (37)** <sup>[12]</sup>. The desired pure product was obtained in 72% yield (59 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.43 - 7.21 (m, 5H), 1.60 (s, 6H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 183.2, 143.8, 128.4, 126.9, 125.8, 46.3, 26.2. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> 165.0910. Found: 165.0913.



**2,2-Dimethyl-3-phenylpropanoic acid** (**38**) <sup>[13]</sup>. The desired pure product was obtained in 66% yield (59 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.29 - 7.13 (m, 5H), 2.89 (s, 2H), 1.20 (s, 6H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  184.5, 137.5, 130.2, 128.0, 126.5, 45.8, 43.4, 24.6. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>11</sub>H<sub>15</sub>O<sub>2</sub> 179.1067. Found: 179.1069.



**2,2-Dimethyl-4-phenylbutanoic acid** (**39**) <sup>[14]</sup>. The desired pure product was obtained in 75% yield (72 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 - 7.12 (m, 5H), 2.71 (t, *J* = 8.7 Hz, 2H), 1.80 (t, *J* = 8.9 Hz, 2H), 1.29 (s, 6H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  173.7, 142.5, 128.4, 128.3, 125.7, 70.9, 45.7, 30.7, 29.3. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>17</sub>O<sub>2</sub> 193.1223. Found: 193.1226.

ÇH₂OH

**2,2-Dimethyl-4-phenylbutanoic acid (40).** The desired pure product was obtained in 93% yield (38 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.37 - 7.20 (m, 5H),

3.70 (d, J = 4.2 Hz, 2H), 2.95 (q, J = 6.9 Hz, 1H), 1.28 (d, J = 7.0 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  143.6, 128.6, 127.5, 126.7, 68.7, 42.4, 17.6. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>9</sub>H<sub>13</sub>O 137.0961. Found: 137.0962.



**2,2-Dimethyl-4-phenylbutanoic acid** (**41**). The desired pure product was obtained in 96% yield (85 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.91 - 7.73 (m, 4H), 7.47 - 7.29 (m, 5H), 4.13 (q, *J* = 7.2 Hz, 1H), 1.68 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  170.8, 161.8, 138.3, 134.7, 128.9, 128.9, 127.8, 127.5, 123.9, 42.9, 19.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>17</sub>H<sub>14</sub>NO<sub>4</sub> 296.0917. Found: 296.0921.



**2,2-Dimethyl-4-phenylbutanoic acid (42).** The desired pure product was obtained in 94% yield (68 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.34 - 7.21 (m, 10H), 5.10 (q, *J* = 12.5 Hz, 2H), 3.77 (q, *J* = 7.1 Hz, 1H), 1.52 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  174.3, 140.4, 136.0, 128.6, 128.4, 128.0, 127.8, 127.5, 127.1, 66.4, 45.5, 18.4. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>16</sub>H<sub>17</sub>O<sub>2</sub> 241.1223. Found: 241.1225.



**2,2-Dimethyl-4-phenylbutanoic acid (43).** The desired pure product was obtained in 91% yield (64 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.38 - 7.24 (m, 5H), 5.94 (s, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 4.04 - 3.87 (m, 2H), 3.63 (q, *J* = 7.2 Hz, 1H),

1.54 (d, J = 7.2 Hz, 3H), 1.24 (t, J = 7.2 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  174.3, 169.8, 140.9, 128.9, 127.7, 127.3, 61.4, 46.8, 41.5, 18.4, 14.0. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>18</sub>NO<sub>3</sub> 236.1281. Found: 236.1287.



**2,2-Dimethyl-4-phenylbutanoic acid** (**47**). The desired pure product was obtained in 21% yield (17 mg) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.89 - 7.78 (m, 3H), 7.67 (s, 1H), 7.53 - 7.35 (m, 3H), 2.86 (q, *J* = 7.6 Hz, 2H), 1.38 (t, *J* = 7.6 Hz, 3H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  141.8, 133.7, 131.9, 127.8, 127.6, 127.4, 127.1, 125.8, 125.5, 125.0, 29.7, 29.0, 15.5. HRMS (ESI) m/z: [M + H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>13</sub>D 159.1153. Found: 159.1155.

#### 5. References

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### 6. NMR Spectra of Products



fl (ppm)





















S32

#### <sup>19</sup>F NMR of compound 7



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

















#### <sup>19</sup>F NMR of compound **10**







110 100 f1 (ppm) 



7.3318 7.3289 7.3286 7.3197 7.3110 7.3120 7.3220 7.3020 7.2851 7.2851 7.2851 7.2775	4.1404 4.11224 4.1181 4.1181 4.0939 4.0868 4.0868 4.0803 3.1482 3.1482 3.1482 3.1392 3.1392 3.1392 3.1392 3.1392 3.12837 2.6972 2.6837 2.6612	1.2157 1.1979 1.1799
		512













 $^1\text{H}$  NMR (400 MHz, CDCl\_3) of compound 15









S46







S48





S50













S56



S57





110 100 f1 (ppm) 



S60



![](_page_60_Figure_1.jpeg)

![](_page_61_Figure_1.jpeg)

100 90 fl (ppm) 

![](_page_62_Figure_0.jpeg)

S63

![](_page_63_Figure_0.jpeg)

![](_page_64_Figure_0.jpeg)

S65

![](_page_65_Figure_0.jpeg)

![](_page_66_Figure_0.jpeg)

![](_page_66_Figure_1.jpeg)

![](_page_67_Figure_1.jpeg)

![](_page_67_Figure_3.jpeg)

#### <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of compound 42 <sup>80151</sup> <sup>80152</sup> <sup>80</sup>

![](_page_68_Figure_1.jpeg)

#### $^{13}C$ NMR (151 MHz, CDCl<sub>3</sub>) of compound **42**

|--|

- 18.4451

-174.2939

![](_page_68_Figure_5.jpeg)

![](_page_69_Figure_0.jpeg)

![](_page_69_Figure_1.jpeg)

![](_page_69_Figure_2.jpeg)

![](_page_69_Figure_3.jpeg)

<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) of compound 43

![](_page_69_Figure_5.jpeg)

![](_page_69_Figure_6.jpeg)

- 174.2780 - 169.8365

![](_page_69_Figure_7.jpeg)

- 18.3697 - 14.0307

![](_page_69_Figure_8.jpeg)

110 100 f1 (ppm) ( 

![](_page_70_Figure_0.jpeg)

![](_page_70_Figure_1.jpeg)