Supporting Information for

## Poly(ethylene glycol) dimethyl ether mediated oxidative scission of aromatic olefins to carbonyl compounds by molecular oxygen

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#### 1. General information

Unless otherwise noted, all reagents, catalysts and solvents were purchased from commercial suppliers and used without further purification. Column chromatography was performed with silica gel (200-300 mesh). NMR spectra were recorded on Bruker AVANCE III (400 MHz) spectrometers. CDCl<sub>3</sub> was the solvent used for the NMR analysis, with tetramethylsilane as the internal standard. Chemical shifts were reported upfield to TMS (0.00 ppm) for <sup>1</sup>H NMR and relative to CDCl<sub>3</sub> (77.0 ppm) for <sup>13</sup>C NMR. HPLC analysis was conducted on an Agilent 1200 Series instrument with  $5C_{18}$ -MS-II Packed Column (4.6 mm I.D. × 250 mm). The substrates were prepared according to reported method <sup>[1]</sup> or commercially available.

#### 2. General procedure for oxidative scission of aromatic olefin



The corresponding aromatic olefin **1** (0.5 mmol), PEGDME (1 mL) were added to a 10 mL Schlenk tube. The tube was evacuated and filled with oxygen three times. The mixture was stirred at 110 °C for 8 hours under O<sub>2</sub> atmosphere using a balloon. After cooling, the mixture was subjected to silica gel column chromatography (PE: EA = 15:1) to give the product **2**.

#### 3. Gram-scale synthesis of 2a



The *gem*-diphenylethylene (**1a**, 1.80 g, 10 mmol), PEGDME (20 mL) were added to a 50 mL of round-bottomed flask equipped with a three-way jointer. The flask was then evacuated and filled with oxygen three times. The mixture was stirred at 110 °C for 10 hours under O<sub>2</sub> atmosphere using a balloon. After cooling, the mixture was subjected to silica gel column chromatography (PE: EA = 15:1) to give the product **2a** (1.75 g, 96% yield).

#### 4. Synthesis of compound 3a<sup>[2]</sup>



To a stirred solution of benzophenone **2a** (0.91 g, 5 mmol) in tertiary butanol (6.2 mL) was added portionwise trimethylsulfonium iodide (2.04 g, 10 mmol) and crushed potassium hydroxide (1.7 g, 30 mmol) subsequently at 30 °C. The resulting mixture was heated to 40-50 °C and stirred for 12 h. After the reaction was complete, tertiary butanol was removed. The residue was dissolved in a mixture of water (10 mL) and dichloromethane (20 mL). After phase separation, aqueous phase was extracted with dichloromethane (2 × 5 mL). The combined organic phase was washed with water (2 × 10 mL), saturated brine (10 mL), and dried over Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent, **3a** was obtained, which was used for the subsequent oxidative scission directly.

#### 5. Analytical data of the products

Benzophenone (2a, CAS: 119-61-9<sup>[3]</sup>)



White solid; 99% yield (90.2 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.86-7.84 (m, 4H), 7.66-7.62 (m, 2H), 7.55-7.51 (m, 4H); <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 196.8, 137.6, 132.5, 130.1, 128.3.

2-Methylbenzophenone (**2b**, CAS: 131-58-8<sup>[3]</sup>)



Colorless liquid; 98% yield (95.2 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.86-7.84 (m, 2H), 7.65-7.61 (m, 1H), 7.52-7.42 (m, 3H), 7.37-7.28 (m, 3H), 2.38 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 198.7, 138.6, 137.7, 136.8, 133.2,

131.0, 130.3, 130.2, 128.6, 128.5, 125.2, 20.0.

3-Methylbenzophenone (2c, CAS: 643-65-2<sup>[3]</sup>)



Colorless oil; 98% yield (96.2 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.86-7.84 (m, 2H), 7.68 (s, 1H), 7.65-7.61 (m, 2H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.46-7.38 (m, 2H), 2.47(s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  =

197.0, 138.2, 137.8, 137.7, 133.2, 132.4, 130.5, 130.1, 128.3, 128.1, 127.4, 21.4.

4-Methylbenzophenone (2d, CAS: 134-84-9<sup>[3]</sup>)



White solid; 98% yield (96.2 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.84-7.82 (m, 2H), 7.78 (d, J = 8.0 Hz, 2H), 7.64-7.60 (m, 1H), 7.54-7.50 (m, 2H), 7.33 (d, J = 7.6 Hz, 2H), 2.49 (s, 3H); <sup>13</sup>C NMR (100

MHz, CDCl<sub>3</sub>): *δ* = 196.6, 143.3, 137.9, 134.9, 132.2, 130.4, 123.0, 129.0, 128.3, 21.7.

2-Methoxybenzophenone (2e, CAS: 2553-04-0<sup>[3]</sup>)



OMe Colorless oil; 99% yield (105.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.87-7.85 (m, 2H), 7.61-7.57 (m, 1H), 7.53-7.45 (m, 3H), 7.40 (dd,  $J_1$  = 7.2 Hz,  $J_2$  = 1.6 Hz, 1H), 7.10-7.03 (m, 2H), 3.76 (s, 3H); <sup>13</sup>C NMR (100 MHz,

 $CDCl_3$ ):  $\delta = 196.5, 157.4, 137.8, 133.0, 131.9, 129.9, 129.6, 128.8, 128.3, 120.5, 111.5, 55.6.$ 

3-Methoxybenzophenone (**2f**, CAS:  $6136-67-0^{[4]}$ )



Colorless oil; 98% yield (104.0 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.86-7.84 (m, 2H), 7.66-7.61 (m, 1H), 7.55-7.51 (m, 2H), 7.45-7.37 (m, 3H), 7.20-7.17 (m, 1H), 3.91 (s, 3H); <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$ 

= 196.6, 159.6, 138.9, 137.6, 132.5, 130.1, 129.3, 128.3, 122.9, 118.9, 114.3, 55.5.

4-Methoxybenzophenone (2g, CAS: 611-94-9<sup>[3]</sup>)



Colorless oil; 99% yield (105.1 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.89-7.86 (m, 2H), 7.82-7.79 (m, 2H), 7.63-7.59 (m, 1H), 7.54-7.50 (m, 2H), 7.03-6.99 (m, 2H), 3.93 (s, 3H); <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$ 

= 195.6, 163.2, 138.3, 132.6, 131.9, 130.2, 129.8, 128.2, 113.6, 55.5.

2-Fluorobenzophenone (**2h**, CAS: 342-24-5<sup>[3]</sup>)



Colorless oil; 91% yield (91.1 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.89 (d, J = 8.0 Hz, 2H), 7.67-7.50 (m, 5H), 7.31 (td, J<sub>1</sub> = 7.6 Hz, J<sub>2</sub> = 0.8 Hz, 1H), 7.23-7.18 (m, 1H); <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 193.5, 160.1 (d,

J = 250.8 Hz), 137.4, 133.5, 133.1 (d, J = 8.3 Hz), 130.8 (d, J = 2.9 Hz), 129.8, 128.5, 127.0 (d, J = 14.7 Hz), 124.3 (d, J = 3.6 Hz), 116.3 (d, J = 21.6 Hz). <sup>19</sup>F NMR (CDCl<sub>3</sub>, 376 MHz): δ -111.0. 4-Fluorobenzophenone (**2i**, CAS: 345-83-5<sup>[3]</sup>)



Yellow oil; 99% yield (99.1 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.91-7.86 (m, 2H), 7.82-7.80 (m, 2H), 7.63 (tt,  $J_1$  = 6.8 Hz,  $J_2$  = 1.3 Hz, 1H), 7.55-7.51 (m, 2H), 7.23-7.17 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 195.3, 165.4 (d, J = 252.6 Hz), 137.5, 133.8 (d, J = 3.0 Hz), 132.7 (d, J = 9.1 Hz), 132.5, 129.9, 128.4, 115.5 (d, J = 21.7 Hz). <sup>19</sup>**F NMR** (CDCl<sub>3</sub>, 376 MHz):  $\delta$  -105.89.

2-Chlorobenzophenone (2j, CAS: 5162-03-8<sup>[3]</sup>)



Colorless oil; 91% yield (98.6 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.87-7.85 (m, 2H), 7.67-7.63 (m, 1H), 7.53-7.46 (m, 4H), 7.44-7.41 (m, 2H); <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 195.4, 138.6, 136.5, 133.8, 131.3, 131.2, 130.1,

130.1, 129.2, 128.6, 126.7.

4-Chlorobenzophenone (2k, CAS: 134-85-0<sup>[3]</sup>)



White solid; 97% yield (105.1 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.83-7.79 (m, 4H), 7.67-7.63 (m, 1H), 7.56-7.49 (m, 4H); <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 195.6, 138.9, 137.2, 135.9, 132.7, 131.5, 130.0,

128.7, 128.4.

2-Bromobenzophenone (21, CAS: 13047-06-8<sup>[3]</sup>)



Colorless oil; 90% yield (117.5 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.87-7.85 (m, 2H), 7.71-7.63 (m, 2H), 7.53-7.45 (m, 3H), 7.43-7.38 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 195.9, 140.7, 136.1, 133.8, 133.2, 131.2, 130.3,

129.0, 128.7, 127.2, 119.6.

2-(TrifluoroMethyl)benzophenone (**2m**, CAS: 727-99-1<sup>[3]</sup>)



CF<sub>3</sub> White solid; 73% yield (103.8 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.84-7.82 (m, 3H), 7.68-7.63 (m, 3H), 7.53-7.49 (m, 2H), 7.45-7.42 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 195.6, 138.3, 136.4, 133.9, 131.4, 130.3, 129.8,

128.6, 128.1, 126.7 (q, *J* = 4.6 Hz), 125.0, 122.3(q, *J* = 272.3 Hz); <sup>19</sup>**F NMR** (CDCl<sub>3</sub>, 376 MHz): δ -57.99.

3-(TrifluoroMethyl)benzophenone (2n, CAS: 728-81-4<sup>[5]</sup>)



White solid; 99% yield (123.9 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$ = 8.11 (s, 1H), 8.03 (d, J = 8 Hz, 1H), 7.90 (d, J = 7.6 Hz, 1H), 7.86-7.83 (m, 2H), 7.70-7.66 (m, 2H), 7.58-7.55 (m, 2H); <sup>13</sup>C NMR (100

MHz, CDCl<sub>3</sub>): δ = 195.3, 138.3, 136.7, 133.2, 133.1, 130.8, 130.1, 129.0, 128.9 (q, *J* = 3.0 Hz), 128.6, 126.7 (q, *J* = 3.7 Hz), 125.1 (q, *J* = 271.0 Hz); <sup>19</sup>F NMR (CDCl<sub>3</sub>, 376 MHz): δ -62.70.

(3,5-Dimethylphenyl)(phenyl)methanone (20, CAS: 13319-70-5<sup>[6]</sup>)



137.7, 134.1, 132.3, 130.1, 128.3 127.9, 21.3.

2,4-Difluorobenzophenone (**2p**, CAS: 85068-35-5<sup>[6]</sup>)



White solid; 90% yield (98.2 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.87-7.84 (m, 2H), 7.68-7.62 (m, 2H), 7.54-7.51 (m, 2H), 7.08-7.03 (m, 1H), 6.95 (ddd,  $J_1$  = 10.0 Hz,  $J_2$  = 8.8 Hz,  $J_3$  = 2.4 Hz, 1H); <sup>13</sup>C NMR

(100 MHz, CDCl<sub>3</sub>):  $\delta$  = 192.4, 166.2 (dd, J = 253.1Hz, 11.6 Hz), 162.2 (dd, J = 254.4Hz, 11.7 Hz), 137.4, 133.5, 132.5 (dd, J = 10.2 Hz, 4.3 Hz), 129.7, 128.5, 123.3 (dd, J = 14.6 Hz, 3.8 Hz), 111.9 (dd, J = 21.4 Hz, 3.7 Hz), 104.7 (t, J = 25.4 Hz); <sup>19</sup>**F NMR** (CDCl<sub>3</sub>, 376 MHz):  $\delta$  -103.7 (d, J = 10.4 Hz), -105.8 (d, J = 10.4 Hz).

(2-Fluoro-4-methoxyphenyl)(phenyl)methanone (2q, CAS: 1156360-90-5<sup>[7]</sup>)

White solid; 90% yield (103.6 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta = 7.85-7.83$  (m, 2H), 7.64-7.60 (m, 2H), 7.52-7.49 (m, 2H), 6.83 (dd,  $J_I = 8.6$  Hz,  $J_2 = 2.4$  Hz, 1H), 6.71 (dd,  $J_I = 12.0$  Hz,  $J_2 = 2.4$  Hz, 1H), 3.92 (s, 3H).; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta = 192.9$ , 163.9 (d, J = 11.2 Hz), 163.2 (d, J = 252.6 Hz), 138.3, 132.9, 132.7 (d, J = 4.4 Hz), 129.6 (d, J = 1.3 Hz), 128.3, 119.3 (d, J = 13.8 Hz), 110.3 (d, J = 2.9 Hz), 101.9 (d, J = 25.6 Hz), 55.9.

(2,6-Difluorophenyl)(p-tolyl)methanone (2r, known compound<sup>[8]</sup>)

Me F Colourless oil; 86% yield (99.9 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$ = 7.81 (d, J = 8.0 Hz, 2H), 7.48 (tt, J<sub>1</sub> = 8.4 Hz, J<sub>2</sub> = 6.4 Hz, 1H), 7.33 (d, J = 8.0 Hz, 2H), 7.07-7.01 (m, 2H), 2.48 (s, 3H); <sup>13</sup>C NMR (100

MHz, CDCl<sub>3</sub>):  $\delta$  = 188.5, 161.0 (d, J = 249.6 Hz), 158.5 (d, J = 249.9 Hz), 145.4, 134.5, 131.7 (t, J = 9.8 Hz), 129.8, 129.5, 117.3, 112.0 (d, J = 25.1 Hz), 111.9 (dd, J = 18.0 Hz, 4.1 Hz), 21.9; <sup>19</sup>**F NMR** (CDCl<sub>3</sub>, 376 MHz):  $\delta$  -111.9.

4,4'-Dimethoxybenzophenone (2s, CAS: 90-96-0<sup>[9]</sup>)



113.5, 55.5.

(4-Chlorophenyl)(3-(trifluoromethyl)phenyl)methanone (2t, CAS: 91503-65-0<sup>[10]</sup>)



White solid; 97% yield (138.1 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta = 8.08$  (s, 1H), 7.99 (d, J = 7.6 Hz, 1H), 7.91 (d, J = 7.6 Hz, 1H), 7.81-7.78 (m, 2H), 7.69 (t, J = 8.0 Hz, 1H), 7.56-7.52(m,

2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 194.0, 139.6, 137.9, 135.0, 133.0, 131.4, 131.0 (q, *J* = 32.9 Hz), 129.1, 129.1, 129.0, 126.6 (q, *J* = 3.8 Hz), 125.0 (q, *J* = 270.9 Hz). <sup>19</sup>F NMR (CDCl<sub>3</sub>, 376 MHz):  $\delta$  -62.8.

3,5-Dimethyl-4'-(trifluoromethyl)benzophenone (2u, known compound<sup>[11]</sup>)

$$F_{3}C$$

$$Me$$

$$F_{3}C$$

$$Me$$

$$Me$$

$$F_{3}C$$

$$Me$$

$$Me$$

$$(s, 1H), 2.43 (s, 6H); {}^{13}C NMR (100 MHz, CDCl_{3}): \delta = 196.0, 141.1, 00 MHz, CDCl_{3}): \delta = 196.0,$$

138.3, 136.9, 134.8, 133.6 (q, *J* = 32.5 Hz), 130.1, 127.9, 125.3 (q, *J* = 3.7 Hz), 122.4 (q, *J* = 271.0 Hz), 21.24.

2-Naphthyl Phenyl Ketone (2v, CAS: 644-13-3<sup>[12]</sup>)



White solid; 81% yield (95.0 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.32 (s, 1H), 8.00-7.96 (m, 4H), 7.92-7.90 (m, 2H), 7.69-7.64 (m, 2H), 7.62-7.55 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 196.8, 137.9,

135.3, 134.8, 132.4, 132.3, 131.9, 130.2, 130.1, 129.5, 128.4, 128.3, 127.8, 126.8, 125.8.

3-Benzoylthiophene (2w, CAS: 6453-99-2<sup>[6]</sup>)



CDCl<sub>3</sub>):  $\delta$  = 190.1, 141.3, 138.6, 134.0, 132.4, 129.4, 128.6, 128.4, 126.3.

2-Benzoylpyridine (2x, CAS: 91-02-1<sup>[9]</sup>)



Slightly yellow solid; 95% yield (87.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$ = 8.71 (d, J = 4.8 Hz, 1H), 8.09-8.02 (m, 3H), 7.90-7.85 (m, 1H), 7.61-7.56 (m, 1H), 7.50-7.45 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 193.9, 155.0,

148.6, 137.1, 136.3, 132.9, 131.0, 128.2, 126.2, 124.6.

9-Fluorenone (2y, CAS: 486-25-9<sup>[12]</sup>)



Yellow solid; 87% yield (78.7 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.68 (d, J = 7.2 Hz, 2H), 7.53-7.48 (m, 4H), 7.31 (td,  $J_1$  = 7.2 Hz,  $J_2$  = 1.6 Hz, 2H); <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 193.9, 144.4, 134.7, 134.1, 129.1, 124.3, 120.3.

Thioxanthen-9-one (2z, CAS: 492-22-8<sup>[12]</sup>)



Pale yellow solid; 85% yield (90.2 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.65 (dd,  $J_1$  = 8.0 Hz,  $J_2$  = 1.2 Hz, 2H), 7.67-7.59 (m, 4H), 7.53-7.49 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 180.0, 137.3, 132.3, 129.9, 129.2,

126.3, 126.0.

Acetophenone (2aa, CAS: 98-86-2<sup>[13]</sup>)



Colourless oil; 99% yield (59.5 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.93-7.91 (m, 2H), 7.52 (tt,  $J_1$  = 6.8 Hz,  $J_2$  = 1.2 Hz, 1H), 7.44-7.40 (m, 1H), 2.55 (s, 3H); <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 198.0, 137.1, 133.1, 128.5, 128.3, 26.5.

Cyclopropyl Phenyl Ketone (2ab, CAS: 3481-02-5<sup>[13]</sup>)



Colourless oil; 93% yield (68.0 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.05-8.03 (m, 2H), 7.59-7.55 (m, 1H), 7.50-7.46 (m, 2H), 2.72-2.66 (m, 1H), 1.29-1.24 (m, 2H), 1.07-1.03 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 200.6,

138.0, 132.7, 128.5, 128.0, 17.1, 11.7.

4-Bromobenzaldehyde (2ac, CAS: 1122-91-4<sup>[13]</sup>)



White solid; 92% and 89% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta = 10.02$ -10.01 (m, 1H), 7.80-7.77 (m, 2H), 7.54-7.51 (m, 2H), 7.74-7.71 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta = 191.1$ , 135.1, 132.5, 131.0, 129.8.

Benzaldehyde (2ad, CAS: 100-52-7<sup>[13]</sup>)



Colorless liquid; 95% and 90% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 10.00-9.99 (m, 1H), 7.86-7.84 (m, 2H), 7.61-7.57 (m, 1H), 7.51-7.47 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 192.3, 136.4, 134.4, 129.7, 129.0. Terephthalaldehyde (2ae CAS: 623-27-8<sup>[14]</sup>)



White solid; 83% yield (55.4 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 10.18-10.17 (m, 2H), 8.10-8.09 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 191.5, 140.0, 130.2.

2-Methylbenzaldehyde (2ae', CAS: 529-20-4<sup>[14]</sup>)

Colorless liquid; 82% yield (49.4 mg). <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 10.28 (s, 1H), 7.81 (d, J = 7.6 Hz, 1H), 7.49 (td,  $J_1$  = 7.2 Hz,  $J_2$  = 1.2 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.27 (d, J = 7.6 Hz, 1H), 2.68(s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):

 $\delta = 192.8, 140.6, 134.1, 133.7, 132.1, 131.8, 126.3, 19.6.$ 

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### 6. NMR spectra of the products

<sup>1</sup>H & <sup>13</sup>C NMR of 2a













### <sup>1</sup>H & <sup>13</sup>C NMR of 2d



<sup>1</sup>H & <sup>13</sup>C & <sup>19</sup>F NMR of 2e



## $\begin{array}{c} 7.863\\ 7.863\\ 7.850\\ 7.850\\ 7.850\\ 7.656\\ 7.656\\ 7.650\\ 7.651\\ 7.610\\ 7.611\\ 7.610\\ 7.613\\ 7.616\\ 7.613\\ 7.616\\ 7.613\\ 7.616\\ 7.613\\ 7.616\\ 7.613\\ 7.616\\ 7.7391\\ 7.395\\ 7.7391\\ 7.395\\ 7.7391\\ 7.395\\ 7.7391\\ 7.395\\ 7.7391\\ 7.395\\ 7.7391\\ 7.1391\\ 7.395\\ 7.7391\\ 7.1391\\ 7.1395\\ 7.736\\$



### <sup>1</sup>H & <sup>13</sup>C NMR of 2g

# $\begin{array}{c} 7.894\\ 7.887\\ 7.882\\ 7.882\\ 7.882\\ 7.882\\ 7.865\\ 7.865\\ 7.865\\ 7.796\\ 7.791\\ 7.791\\ 7.791\\ 7.791\\ 7.791\\ 7.7592\\ 7.592\\ 7.552\\ 7.552\\ 7.552\\ 7.553\\ 7.552\\ 7.553\\ 7$



### <sup>1</sup>H & <sup>13</sup>C & <sup>19</sup>F NMR of 2h











### <sup>1</sup>H & <sup>13</sup>C NMR of 2k



200 180 160 140 120 100 80 60 40 20 0 f1 (ppm)

### <sup>1</sup>H & <sup>13</sup>C NMR of 2l



#### <sup>1</sup>H & <sup>13</sup>C & <sup>19</sup>F NMR of 2m















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### <sup>1</sup>H & <sup>13</sup>C & <sup>19</sup>F NMR of 2p

## $\begin{array}{c} 7.866\\ 7.863\\ 7.863\\ 7.845\\ 7.845\\ 7.845\\ 7.845\\ 7.670\\ 7.673\\ 7.659\\ 7.673\\ 7.673\\ 7.673\\ 7.673\\ 7.618\\ 7.544\\ 7.551\\ 7.673\\ 7.673\\ 7.673\\ 7.673\\ 7.051\\ 7.073\\ 7.$





<sup>1</sup>H & <sup>13</sup>C NMR of 2q





<sup>1</sup>H & <sup>13</sup>C & <sup>19</sup>F NMR of 2r





-2.477





### <sup>1</sup>H & <sup>13</sup>C & <sup>19</sup>F NMR of 2t













<sup>1</sup>H & <sup>13</sup>C NMR of 2v





<sup>1</sup>H & <sup>13</sup>C NMR of 2w





<sup>1</sup>H & <sup>13</sup>C NMR of 2x







<sup>1</sup>H & <sup>13</sup>C NMR of 2y

7.684 7.5166 7.517 7.518 7.517 7.518 7.517 7.518 7.517 7.518 7.517 7.518 7.517 7.518 7.517 7.518 7.517 7.518 7.729 7.518 7.739 7.518 7.749 7.7329 7.749 7.7329 7.749 7.7329 7.749 7.7329 7.





<sup>1</sup>H & <sup>13</sup>C NMR of 2z

# 8.665 8.662 8.662 8.645







<sup>1</sup>H & <sup>13</sup>C NMR of 2ab







### <sup>1</sup>H & <sup>13</sup>C NMR of 2ac

















<sup>1</sup>H & <sup>13</sup>C NMR of 2ae'





