# Supporting Information

# Electrochemical α-Hydroxylation of Aryl Ketones with

# Methanol as the Oxygen Source

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#### **General Information**

All manipulations were carried out by standard Schlenk techniques. Unless otherwise stated, analytical grade solvents and commercially available reagents were used to conduct the reactions. Thin layer chromatography (TLC) was carried out using 0.25 - mm glass silica gel plates. Column chromatography was performed on silica gel (200–300 meshes) using petroleum ether (bp 60–90 °C) and ethyl acetate as eluents. All the new compounds were characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR, and HRMS. The known compounds were characterized by <sup>1</sup>H, <sup>13</sup>C, and <sup>19</sup>F NMR. The <sup>1</sup>H, <sup>13</sup>C, and <sup>19</sup>F NMR spectra were recorded on a Bruker 400/600 MHz NMR spectrometer. The chemical shifts ( $\delta$ ) were given in parts per million relative to internal tetramethylsilane (TMS, 0 ppm for <sup>1</sup>H NMR) and CDCl<sub>3</sub> (77.16 ppm for <sup>13</sup>C NMR). High resolution mass spectra (HRMS) were measured with a Waters Micromass GCT Premier or JEOL AccuTOF-MS.

Electrolysis experiments were performed using a dual-display potentiostat (DJS - 292B) or galvanostat. Platinum plates ( $15 \text{ mm} \times 15 \text{ mm} \times 0.3 \text{ mm}$ ) were purchased as the anodic and cathodic electrodes. Cyclic voltammograms were obtained on a CHI 605E potentiostat.

#### **General Procedure for Hydroxylation**



**Procedure A1**: In an oven-dried undivided three necked bottle (6 mL) equipped with a stir bar, the bottle was equipped with two platinum electrodes (15 mm × 15 mm × 0.3 mm). A solution of 4'-Methylpropiophenone (0.3 mmol, 45  $\mu$ L), Na<sub>2</sub>CO<sub>3</sub> (0.36 mmol, 1.2 equiv., 38.2 mg), and <sup>*n*</sup>Bu<sub>4</sub>NBr (0.45 mmol, 1.5 equiv., 145.1 mg) in a mixture of MeOH, DMF, and MeCN (2.0 mL each) was stirred under an argon atmosphere and electrolyzed at a constant current of 10 mA at 25 °C for 2.5 h. After completion of the reaction, the mixture was diluted with dichloromethane (10 mL), washed with water (3 × 5 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and the solvent was removed under reduced pressure by rotary evaporation. Then, the pure product **2** was obtained by flash column chromatography on silica gel (eluent: ethyl

acetate/petroleum ether (1:5)). The substrates **1** were subjected to Procedure A1 conditions.

#### **General Procedures for cyclic voltammetry**

Cyclic voltammetry experiments were performed in a three-electrode cell connected to a Schlenk line at room temperature. The working electrode was a steady glassy carbon disk electrode, and the counter electrode was a platinum wire. The reference was a Ag/AgCl electrode submerged in saturated aqueous KCl solution, and separated from reaction by a salt bridge. The scan rate was 0.1 V/s, ranging from 0 V to 2.5 V.

As shown in Figures 2 ( $\mathbf{a}$  and  $\mathbf{b}$ ), the two substrates are difficult to oxidize directly. The initial oxidation potential of TBABr is 0.75 V in the Figure 2 ( $\mathbf{c}$ ). In addition, the cyclic voltammetry curve remains essentially unchanged with the addition of 1a. Therefore,  $\mathbf{1a}$  may not participate in the direct oxidation process of the reaction.



Figure 1 cyclic voltammetry (CV) experiments



Figure 2 Cyclic voltammetry (CV) experiments

## **Experiment on capturing Intermediate products**

Under standard reaction conditions, the intermediates of the reaction were detected by high resolution mass spectra (**HRMS**), and the following intermediates were successfully detected (in Figure 3, Figure 4).



**Figure 3 Intermediate products** 



Figure 4 Intermediate products by detecting of HRMS

#### **Control experiment**

A series of control experiments were conducted to explore the mechanism of the reaction (Table 1). Firstly, 2,2,6,6-Tetramethyl-1-piperidinyloxy (TEMPO, 2.0 equiv.) and Butylated Hydroxytoluene (BHT, 2.0 equiv.) were separately added to the standard reaction system, and the reaction was inhibited, as shown in Entries 1 and 2. Next, to prove the source of hydroxyl groups, oxygen - containing reagents in the reaction system were controlled. Without DMF or MeCN, the reaction proceeded smoothly (Entries 3, 5). But the reaction could not take place in the absence of MeOH (Entry 4). Consequently, methanol is necessary in the reaction (Entries 3-5). Besides, when H<sub>2</sub><sup>18</sup>O was added to the standard reaction system, 3-VII was not detected by HRMS (Entry 6).



Table 1			
Entry	Variation of standard conditions	<b>Yield(%)</b> <sup>[b]</sup>	
1	add 2.0 equiv. TEMPO	n.d.	
2	add 2.0 equiv. BHT	trace	
3	MeOH: MeCN = 3.0 : 3.0 mL	24	
4	DMF: MeCN = 3.0 : 3.0 mL	n.d.	
5	MeOH: DMF = 3.0 : 3.0 mL	61	

[a]: no detected the **3-VII** by HRMS. [b]: Isolated product.

## **Deuteration Experiment of the Product**



**Procedure A1**: In an oven-dried undivided three necked bottle (6 mL) equipped with a stir bar, a solution of **2a** (0.3 mmol, 49.2 mg), Na<sub>2</sub>CO<sub>3</sub> (0.2 mmol, 1.0 equiv., 21.3 mg), NaOMe (0.2 mmol, 1.0 equiv., 10.8 mg) in a mixture of MeOH, DMF, and MeCN (5.0 mL) was stirred under N<sub>2</sub> at 40 °C for 12 h. After completion of the reaction, the mixture was directly analyzed by <sup>1</sup>H NMR spectroscopy. Degree of deuteration: 100%.



#### General procedure for the gram-scale reaction

#### a) The equipment for gram-scale reaction

The gram-scale reaction equipment was composed of a direct current instrument, platinum electrodes, a magnetic stirrer and a solvent bottle (Figure 5).



Figure 5 Gram-scale reaction equipment b) Synthesis of 2-hydroxy-1-(p-tolyl)propan-1-one

In an oven-dried undivided bottle (100 mL) equipped with a stir bar, 4'-Methylpropiophenone **1a** (15.0 mmol, 2.24 mL), "Bu<sub>4</sub>NBr (22.5 mmol, 7.25 g), Na<sub>2</sub>CO<sub>3</sub> (18.0 mmol, 1.91 g), DMF (30 mL), MeCN (30 mL), and MeOH (30 mL) were added. The bottle was equipped with two platinum electrodes (30 mm × 30 mm × 0.3 mm), one as the anode and the other as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 80 mA under an Ar atmosphere at room temperature for 15 h. Finally, after completion of the reaction, the pure product (yield 75%, yellow liquid, 1.85 g) was obtained by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 5 : 1).



# Figure 6 Synthesis of 2-hydroxy-1-(p-tolyl)propan-1-one c) Synthesis of 2-hydroxy-1,2-diphenylethan-1-one

In an oven - dried undivided bottle (100 mL) equipped with a stir bar, 2-Phenylacetophenone (15.0 mmol, 2.94 g), <sup>*n*</sup>Bu<sub>4</sub>NBr (18.0 mmol, 5.80 g), Na<sub>2</sub>CO<sub>3</sub> (18.0 mmol, 1.91 g), DMF (30 mL), MeCN (30 mL), and MeOH (30 mL) were added. The bottle was equipped with two platinum electrodes (30 mm  $\times$  30 mm  $\times$  0.3 mm), one as the anode and the other as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 50 mA under an Ar atmosphere at room temperature for 15 h. Finally, after completion of the reaction, the pure product (yield 33%, light yellow solid, 1.06 g) was obtained by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 5 : 1).



Figure 7 Synthesis of 2-hydroxy-1,2-diphenylethan-1-one

#### **Synthetic Applications**



At room temperature, TFA (0.02 mmol) and 3Å molecular sieve (100 mg) were added to a stirred solution of  $\alpha$ -hydroxyl ketone **2s** (0.2 mmol) and ethylenediamine (0.2 mmol) in DCE (2 mL). Subsequently, the reaction mixture was continuously stirred at 30 °C in an oil bath in an air atmosphere for 12 h, monitored by TLC. After completion of the reaction, the mixture was cooled to room temperature, diluted with ethyl acetate (10 mL), washed with saturated NaCl solution (3 × 5 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and the solvent was removed by rotary evaporator. The pure product (yield 83%, white solid, 38.4 mg) was obtained by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 9 : 1).

#### **Synthesis of Diphenyl Acetone Substrates**

In an oven-dried undivided 100 mL bottle equipped with a stir bar, 'BuOK (5.0 equiv.) and PhI (5.0 mmol) were added first. Then, acetophenone (1.2 equiv.) and anhydrous DMF (50 mL) were added. The reaction mixture was stirred under an Ar atmosphere at 60 °C for 24 hours. After the reaction was completed, the reaction mixture was extracted, washed with appropriate solvents, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and the product was separated by column chromatography. However, during the separation process, the product was easily mixed with acetophenone. Pure benzophenone products can be obtained by recrystallization at -78 °C.



Figure 8 Synthesis of diphenyl acetone raw materials

#### **Characterization of Products**



**Reaction conditions:** substrate (45  $\mu$ L, 0.3 mmol), "Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-hydroxy-1-(p-tolyl)propan-1-one* (2a). The Product 2a was purified by silica gel column chromatography (PE/EA = 5:1), colorless oil was obtained in 83% (41.0 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.76 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 5.06 (q, *J* = 7.0 Hz, 1H), 2.36 (s, 3H), 1.37 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 202.0, 145.1, 130.8, 129.7, 128.9, 69.3, 22.6, 21.9.



**Reaction conditions:** substrate (46.2  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-hydroxy-1-(m-tolyl)propan-1-one* (2b): The Product 2b was purified by silica gel column chromatography (PE/EA = 5 : 1), light yellow oil was obtained in 90% (44.2 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.66 (s, 1H), 7.63 (d, *J* = 7.5 Hz, 1H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 5.07 (q, *J* = 7.0 Hz, 1H), 3.42 (s, 1H), 2.34 (s, 3H), 1.36 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 202.6, 138.8, 134.8, 133.4, 129.2, 128.8, 125.9, 69.4, 22.4, 21.4.



**Reaction conditions:** substrate (50.6  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(4-ethylphenyl)-2-hydroxypropan-1-one* (2c). The Product 2c was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 88% (46.8 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-d) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.2 Hz, 2H), 5.06 (q, *J* = 7.0 Hz, 1H), 3.40 (s, 1H), 2.64 (q, *J* = 7.6 Hz, 2H), 1.37 (d, *J* = 7.0 Hz, 3H), 1.19 (t, *J* = 7.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 202.0, 151.2, 131.0, 129.0, 128.4, 69.3, 29.1, 22.5, 15.2.



**Reaction conditions:** substrate (45.6  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-hydroxy-1-(3-methoxyphenyl)propan-1-one* (2d). The Product 2d was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 89% (48.0 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.42–7.37 (m, 2H), 7.36–7.29 (m, 1H), 7.11–7.06 (m, 1H), 5.06 (q, *J* = 7.0 Hz, 1H), 3.79 (s, 3H), 3.26 (s, 1H), 1.37 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 202.4, 160.0, 134.7, 129.9, 121.2, 120.4, 113.1, 69.5, 55.6, 22.5.



**Reaction conditions:** substrate (72.1 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode, platinum as cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(4-(benzyloxy)phenyl)-2-hydroxypropan-1-one* (2e). The Product 2e was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 50% (38.5 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-d)  $\delta$  7.88–7.78 (m, 2H), 7.38–7.23 (m, 5H), 7.00–6.90 (m, 2H), 5.06 (s, 2H), 5.01 (q, *J* = 7.0 Hz, 1H), 1.36 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  200.7, 163.4, 136.0, 131.1, 128.8, 128.4, 127.6, 126.3, 115.0, 70.3, 69.0, 22.7. HRMS (ESI) exact mass calculated for [C<sub>16</sub>H<sub>16</sub>O<sub>3</sub> + K]<sup>+</sup> : 295.0731, found: 295.0732.



**Reaction conditions:** substrate (42  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(4-fluorophenyl)-2-hydroxypropan-1-one* (2f). The Product 2f was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 96% (48.3 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.93–7.88 (m, 2H), 7.17–7.07 (m, 2H), 5.06 (q, J = 7.0 Hz, 1H), 1.38 (d, J = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 200.90, 166.30 (d, *J* = 256.5 Hz), 131.50 (d, *J* = 9.4 Hz), 129.83 (d, *J* = 3.1 Hz), 116.28 (d, *J* = 22.0 Hz), 69.34, 22.44.

<sup>19</sup>F NMR (376 MHz, Chloroform-*d*)  $\delta$  –103.1.



**Reaction conditions:** substrate (50.6 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(4-chlorophenyl)-2-hydroxypropan-1-one* (2g). The Product 2g was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 89% (49.2 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-d)  $\delta$  7.82–7.77 (m, 2H), 7.43–7.37 (m, 2H), 5.04 (q, J = 7.0 Hz, 1H), 3.25 (s, 1H), 1.37 (d, J = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.3, 140.6, 131.8, 130.1, 129.3, 69.4, 22.3.



**Reaction conditions:** substrate (63.9 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(4-bromophenyl)-2-hydroxypropan-1-one* (2h). The Product 2h was purified by silica gel column chromatography (PE/EA = 5 : 1), yellow oil was obtained in 90% (61.5 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-d)  $\delta$  7.75–7.68 (m, 2H), 7.60–7.54 (m, 2H), 5.04 (q, J = 7.1 Hz, 1H), 3.20 (s, 1H), 1.36 (d, J = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.5, 132.3, 132.2, 130.2, 129.3, 69.4, 22.3.



**Reaction conditions:** substrate (42.5  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(3-fluorophenyl)-2-hydroxypropan-1-one* (2i). The Product 2i was purified by silica gel column chromatography (PE/EA = 5 : 1), yellow oil was obtained in 65% (32.6 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.62 (d, *J* = 7.7 Hz, 1H), 7.57–7.53 (m, 1H), 7.44–7.39 (m, 1H), 7.27–7.22 (m, 1H), 5.04 (q, *J* = 7.0 Hz, 1H), 3.47 (s, 1H), 1.38 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  201.2, 163.0 (d, J = 252.5 Hz), 135.5 (d, J = 10.1 Hz), 130.7 (d, J = 8.1 Hz), 124.5 (d, J = 3.0 Hz), 121.0 (d, J = 20.2 Hz), 115.4 (d, J = 20.2 Hz), 69.5, 22.1.

<sup>19</sup>F NMR (376 MHz, Chloroform-*d*)  $\delta$  –111.0.



**Reaction conditions:** substrate (50.6 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(3-chlorophenyl)-2-hydroxypropan-1-one* (2j). The Product 2j was purified by silica gel column chromatography (PE / EA = 5 : 1), yellow oil was obtained in 64% (34.9 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.83 (t, *J* = 1.8 Hz, 1H), 7.73–7.70 (m, 1H), 7.53–7.50 (m, 1H), 7.38 (t, *J* = 7.9 Hz, 1H), 5.04 (q, *J* = 7.0 Hz, 1H), 1.37 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.4, 135.4, 135.1, 134.0, 130.3, 128.8, 126.8, 69.6, 22.2.



**Reaction conditions:** substrate (63.9 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(3-bromophenyl)-2-hydroxypropan-1-one* (2k). The Product 2k was purified by silica gel column chromatography (PE/EA = 5 : 1), yellow oil was obtained in 73% (50.1 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.99 (t, J = 1.7 Hz, 1H), 7.78–7.74 (m, 1H), 7.69–7.64 (m, 1H), 7.31 (t, J = 7.9 Hz, 1H), 5.04 (q, J = 7.0 Hz, 1H), 3.28 (s, 1H), 1.37 (d, J = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.3, 136.9, 135.3, 131.7, 130.5, 127.2, 123.3, 69.6, 22.2.



**Reaction conditions:** substrate (41.4  $\mu$ L, 0.3 mmol), "Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(2-fluorophenyl)-2-hydroxypropan-1-one* (21). The Product 21 was purified by silica gel column chromatography (PE/EA = 5 : 1), yellow oil was obtained in 77% (38.4 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.88–7.84 (m, 1H), 7.54–7.49 (m, 1H), 7.23–7.20 (m, 1H), 7.12–7.07 (m, 1H), 5.00–4.94 (m, 1H), 3.57 (s, 1H), 1.33 (q, *J* = 4.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  200.9, 161.5 (d, J = 252.5 Hz), 135.6 (d, J = 10.1 Hz), 131.1 (d, J = 3.0 Hz), 124.9 (d, J = 3.0 Hz), 122.2 (d, J = 13.1 Hz), 116,7(d, J = 30.3 Hz), 72.8, 20.7.

<sup>19</sup>F NMR (376 MHz, Chloroform-*d*)  $\delta$  -108.3.



**Reaction conditions:** substrate (43.8  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(3,4-difluorophenyl)-2-hydroxypropan-1-one* (2m). The Product 2m was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 90% (50.3 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.74–7.69 (m, 1H), 7.66–7.63 (m, 1H), 7.23 (t, J = 8.0 Hz, 1H), 5.02 (q, J = 7.0 Hz, 1H), 3.57 (s, 1H), 1.37 (d, J = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 200.0, 161.7 (d, J = 255.3 Hz), 135.7 (d, J = 9.2 Hz), 131.2 (d, J = 2.7 Hz), 125.0 (d, J = 3.3 Hz), 122.3 (d, J = 13.3 Hz), 116.9 (d, J = 23.5 Hz). 69.4, 22.0.

<sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -127.9, -128.0, -135.2, -135.3.

HRMS (ESI) exact mass calculated for  $[C_9H_8F_2O_2 + H]^+$ : 187.0565, found: 187.0562.



**Reaction conditions:** substrate (43.5  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-hydroxy-1-phenylbutan-1-one* (2n). The Product 2n was purified by silica gel column chromatography (PE/EA = 5:1), colorless oil was obtained in 77% (37.8 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.85–7.83 (m, 2H), 7.56–7.52 (m, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 5.00–4.98 (m, 1H), 3.25 (s, 1H), 1.93–1.83 (m, 1H), 1.59–1.49 (m, 1H), 0.87 (t, *J* = 7.4 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 202.2, 134.0, 133.9, 129.0, 128.6, 74.1, 28.9, 9.0.



**Reaction conditions:** substrate (56.1  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-hydroxy-1-(p-tolyl)pentan-1-one* (20). The Product 20 was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 54% (31.0 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 4.99–4.96 (m, 1H), 3.22 (s, 1H), 2.35 (s, 3H), 1.77–1.71 (m, 1H), 1.51–1.40 (m, 2H), 1.38–1.31 (m, 1H), 0.84 (t, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.8, 145.0, 131.2, 129.6, 128.7, 72.9, 38.3, 21.9, 18.3, 14.0.

HRMS (ESI) exact mass calculated for  $[C_{12}H_{16}O_2 + H]^+$ : 193.1223, found: 193.1227.



**Reaction conditions:** substrate (55.2  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-hydroxy-1-phenylhexan-1-one* (2p). The Product 2p was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 41% (23.6 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 7.5 Hz, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 5.01–4.99 (m, 1H), 3.31 (s, 1H), 1.83–1.74 (m, 1H), 1.51–1.37 (m, 2H), 1.33–1.16 (m, 3H), 0.78 (t, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 202.3, 134.0, 133.8, 128.9, 128.6, 73.2, 35.7, 27.2, 22.6, 14.0<sup>-</sup>

HRMS (ESI) exact mass calculated for  $[C_{12}H_{16}O_2 + H]^+$ : 193.1223, found: 193.1221.



**Reaction conditions:** substrate (73.9 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-hydroxy-1-phenylundecan-1-one* (2q). The Product 2q was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 34% (26.2 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 8.1 Hz, 2H), 7.54 (t, *J* = 7.3 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 5.01–4.99 (m, 1H), 3.26 (s, 1H), 1.82–1.73 (m, 1H), 1.51–1.39 (m, 2H), 1.26–1.18 (m, 13H), 0.79 (t, *J* = 6.7 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 202.3, 134.0, 133.8, 129.0, 128.6, 73.2, 36.0, 32.0, 29.6, 29.5, 29.5, 29.4, 25.0, 22.8, 14.2.

HRMS (ESI) exact mass calculated for  $[C_{17}H_{26}O_2 + Na]^+$  : 285.1825 , found: 285.1827.



**Reaction conditions:** substrate (63.1 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-hydroxy-1,3-diphenylpropan-1-one* (2r). The Product 2r was purified by silica gel column chromatography (PE/EA = 5 : 1), light yellow solid was obtained in 63% (42.5 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.83–7.81 (m, 2H), 7.53–7.51 (m, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.17–7.11 (m, 3H), 7.04–7.02 (m, 2H), 5.25–5.22 (m, 1H), 3.54 (s, 1H), 3.12–3.07 (m, 1H), 2.82–2.77 (m, 1H).

<sup>13</sup>C NMR (151 MHz, Chloroform-*d*) δ 201.0, 136.5, 134.0, 133.9, 129.5, 129.0, 128.7, 128.3, 126.8, 73.8, 41.9.

HRMS (ESI) exact mass calculated for  $[C_{15}H_{14}O_2 + Na]^+$  : 249.0886 , found: 249.0887.



**Reaction conditions:** substrate (58.8 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-hydroxy-1,2-diphenylethan-1-one* (2s). The Product 2s was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 70% (44.7 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.84–7.82 (m, 2H), 7.43 (t, *J* = 7.4 Hz, 1H), 7.31 (t, *J* = 7.7 Hz, 2H), 7.27–7.22 (m, 4H), 7.20–7.16 (m, 1H), 5.88 (s, 1H), 4.43 (s, 1H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 199.1, 139.1, 134.0, 133.6, 129.3, 129.2, 128.8, 128.7, 127.9, 76.3.



**Reaction conditions:** substrate (63.1 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-hydroxy-2-phenyl-1-(p-tolyl)ethan-1-one* (2t). The Product 2t was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 56% (37.5 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 8.2 Hz, 2H), 7.23 (q, *J* = 8.0 Hz, 4H), 7.19–7.13 (m, 1H), 7.09 (d, *J* = 8.1 Hz, 2H), 5.84 (s, 1H), 4.13 (s, 1H), 2.25 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 198.5, 145.1, 139.4, 131.0, 129.5, 129.4, 129.2, 128.6, 127.8, 76.1, 21.8.

HRMS (ESI) exact mass calculated for  $[C_{15}H_{14}O_2 + Na]^+$  : 249.0886 , found: 249.0885.



**Reaction conditions:** substrate (67.2 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *1-(4-ethylphenyl)-2-hydroxy-2-phenylethan-1-one* (2u). The Product 2u was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 52% (37.2 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.28–7.18 (m, 5H), 7.14 (d, *J* = 8.2 Hz, 2H), 5.85 (s, 1H), 2.57 (q, *J* = 7.6 Hz, 2H), 1.13 (t, *J* = 7.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 198.6, 151.2, 139.4, 131.2, 129.5, 129.2, 128.6, 128.3, 127.9, 76.2, 29.1, 15.0.

HRMS (ESI) exact mass calculated for  $[C_{16}H_{16}O_2 + Na]^+$  : 263.1042 , found: 263.1036.



**Reaction conditions:** substrate (71.4 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-hydroxy-1-(4-isopropylphenyl)-2-phenylethan-1-one* (2v). The Product 2v was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 43% (32.8 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.29–7.24 (m, 4H), 7.22–7.18 (m, 3H), 5.85 (s, 1H), 2.88–2.78 (m, 1H), 1.14 (d, *J* = 6.9 Hz, 6H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 198.5, 155.8, 139.5, 131.3, 129.6, 129.3, 128.6, 127.9, 127.0, 76.2, 34.4, 23.6.

HRMS (ESI) exact mass calculated for  $[C_{17}H_{18}O_2 + H]^+$ : 255.1380, found: 255.1372.



**Reaction conditions:** substrate (64.2 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *1-(4-fluorophenyl)-2-hydroxy-2-phenylethan-1-one* (2w). The Product 2w was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 36% (24.3 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.86–7.83 (m, 2H), 7.23–7.15 (m, 5H), 6.94 (t, J = 8.5 Hz, 2H), 5.81 (s, 1H), 4.31 (s, 1H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.4, 166.0 (d, *J* = 262.6 Hz), 138.9, 132.0 (d, *J* = 10.1 Hz), 129.9 (d, *J* = 3.0 Hz), 129.2, 128.7, 127.8, 116.0 (d, *J* = 20.2 Hz), 76.2.
<sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ –103.0.

HRMS (ESI) exact mass calculated for  $[C_{14}H_{11}FO_2 + Na]^+:253.0635$  , found: 253.0637.



**Reaction conditions:** substrate (69.0 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *1-(4-chlorophenyl)-2-hydroxy-2-phenylethan-1-one* (2x). The Product 2x was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 33% (24.0 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.77 (d, *J* = 8.6 Hz, 2H), 7.28 (d, *J* = 8.6 Hz, 2H), 7.26–7.22 (m, 4H), 7.20 (s, 1H), 5.82 (s, 1H), 4.38 (s, 1H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.9, 140.6, 138.8, 131.9, 130.6, 129.4, 129.2, 128.9, 127.9, 76.4.



**Reaction conditions:** substrate (63.1 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h.

*2-hydroxy-1-phenyl-2-(p-tolyl)ethan-1-one* (2y). The Product 2y was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 26% (17.6 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.87–7.77 (m, 2H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.28 (t, *J* = 7.7 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 2H), 7.02 (d, *J* = 7.9 Hz, 2H), 5.83 (s, 1H), 4.21–3.68 (m, 1H), 2.18 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 199.1, 138.5, 136.2, 133.9, 133.6, 129.9, 129.2, 128.7, 127.8, 76.1, 21.2.



**Reaction conditions:** substrate (67.2 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-(4-ethylphenyl)-2-hydroxy-1-phenylethan-1-one* (2z). The Product 2z was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 40% (28.9 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.86–7.84 (m, 2H), 7.46–7.42 (m, 1H), 7.34–7.30 (m, 2H), 7.18 (d, *J* = 3.1 Hz, 1H), 7.16 (d, *J* = 1.8 Hz, 1H), 7.08 (d, *J* = 8.2 Hz, 2H), 5.86 (s, 1H), 4.42 (s, 1H), 2.52 (q, J = 7.6 Hz, 2H), 1.11 (t, *J* = 7.6 Hz, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  199.2, 144.9, 136.4, 134.0, 133.7, 129.3, 128.8, 127.9, 76.1, 28.7, 15.4.

HRMS (ESI) exact mass calculated for  $[C_{16}H_{16}O_2 + H]^+$ : 241.1223, found: 241.1228.



**Reaction conditions:** substrate (71.4 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-hydroxy-2-(4-isopropylphenyl)-1-phenylethan-1-one* (2aa). The Product 2aa was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 44% (33.1 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.86–7.84 (m, 2H), 7.46–7.42 (m, 1H), 7.32 (t, J = 7.7 Hz, 2H), 7.17 (d, J = 7.5 Hz, 2H), 7.10 (d, J = 8.2 Hz, 2H), 5.86 (s, 1H), 2.28–2.73 (m, 1H), 1.11 (m, 6H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 199.1, 149.4, 136.5, 134.0, 133.7, 129.3, 128.8, 127.8, 127.4, 76.1, 33.9, 23.9.

HRMS (ESI) exact mass calculated for  $[C_{17}H_{18}O_2 + K]^+$ : 293.0939, found: 293.0938.



**Reaction conditions:** substrate (75.6 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-(4-(tert-butyl) phenyl)-2-hydroxy-1-phenylethan-1-one* (2ab). The Product 2ab was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 34% (27.7 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.89–7.83 (m, 2H), 7.49–7.41 (m, 1H), 7.33 (t, J = 7.7 Hz, 2H), 7.29–7.23 (m, 2H), 7.20–7.16 (m, 2H), 5.87 (s, 1H), 4.20 (d, J = 177.0 Hz, 1H), 1.18 (s, 9H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 199.0, 151.6, 135.9, 133.8, 133.6, 129.2, 128.7, 127.4, 126.1, 75.9, 34.6, 31.2.

HRMS (ESI) exact mass calculated for  $[C_{18}H_{20}O_2 + H]^+$ : 269.1536, found: 269.1537.



**Reaction conditions:** substrate (67.8 mg, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-hydroxy-2-(4-methoxyphenyl)-1-phenylethan-1-one* (2ac). The Product 2ac was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 38% (27.3 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.83–7.80 (m, 2H), 7.44–7.39 (m, 1H), 7.29 (t, *J* = 7.7 Hz, 2H), 7.18–7.14 (m, 2H), 6.77–6.73 (m, 2H), 5.83 (s, 1H), 3.65 (s, 3H). <sup>13</sup>C NMR (151 MHz, Chloroform-*d*) δ 199.1, 159.8, 133.9, 133.6, 131.3, 129.2, 129.2, 128.7, 114.6, 75.7, 55.3. HRMS (ESI) exact mass calculated for  $[C_{15}H_{14}O_3 + Na]^+:265.0835$  , found: 265.0837.



**Reaction conditions:** substrate (76.8 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-hydroxy-1,2-bis(4-methoxyphenyl)ethan-1-one* (2ad). The Product 2ad was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 45% (36.5 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 8.9 Hz, 2H), 7.16 (d, *J* = 8.7 Hz, 2H), 6.78–6.73 (m, 4H), 5.76 (s, 1H), 3.71 (s, 3H), 3.65 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.4, 164.0, 159.7, 131.9, 131.6, 129.1, 126.4, 114.6, 114.0, 75.3, 55.5, 55.3.



**Reaction conditions:** substrate (64.2 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, Ar, platinum as anode, platinum as cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h.

**2-(4-fluorophenyl)-2-hydroxy-1-phenylethan-1-one** (2ae). The Product 2ae was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 36% (25.0 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.87–7.75 (m, 2H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.29 (t, *J* = 7.7 Hz, 2H), 7.25–7.17 (m, 2H), 6.89 (t, *J* = 8.6 Hz, 2H), 5.86 (s, 1H), 4.16 (s, 1H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 198.8, 162.7 (d, *J* = 252.5 Hz) 135.0 (d, *J* = 3.0 Hz), 134.0, 133.4, 129.6 (d, *J* = 8.1 Hz), 129.1, 128.8, 116.1 (d, *J* = 20.2 Hz), 75.4.
<sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ –112.9.

HRMS (ESI) exact mass calculated for  $[C_{14}H_{11}FO_2 + K]^+$  : 269.0375 , found: 269.0383.



**Reaction conditions:** substrate (69.0, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol),  $Na_{2}CO_{3}$  (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h.

2-(4-chlorophenyl)-2-hydroxy-1-phenylethan-1-one (2af). The Product 2af was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 27% (19.5 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.80 (d, *J* = 7.3 Hz, 2H), 7.44 (t, *J* = 7.4 Hz, 1H), 7.35-7.28 (m, 2H), 7.19 (s, 4H), 5.85 (s, 1H), 4.73–3.51 (m, 1H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 198.7, 137.6, 134.6, 134.2, 133.4, 129.4, 129.2, 129.2, 128.9, 75.5.

HRMS (ESI) exact mass calculated for  $[C_{14}H_{11}ClO_2 + Na]^+: 269.0340$  , found: 269.0340.



**Reaction conditions:** substrate (82.3 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), MeOH: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h.

2-(4-bromophenyl)-2-hydroxy-1-phenylethan-1-one (2ag). The Product 2ag was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 21% (18.2 mg) isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.83–7.78 (m, 2H), 7.48–7.43 (m, 1H), 7.37 (d, *J* = 1.8 Hz, 1H), 7.36–7.29 (m, 3H), 7.15–7.10 (m, 2H), 5.84 (s, 1H), 4.43 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 198.6, 138.1, 134.3, 133.4, 132.4, 129.5, 129.2,

HRMS (ESI) exact mass calculated for  $[C_{14}H_{11}BrO_2 + Na]^+$ : 312.9834 , found: 312.9831.



128.9, 122.8, 75.6.

**Reaction conditions:** substrate (45  $\mu$ L, 0.3 mmol), "Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), CD<sub>3</sub>OD: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

2-(hydroxy-d)-1-(p-tolyl)propan-1-one-2-d (2ah): The Product 2ah was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 72% (35.9 mg) isolated yield. The 89% deuterium incorporation was ascertained by means of <sup>1</sup>H NMR.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.76 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 2.36 (s, 3H), 1.36 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.9, 145.1, 130.7, 129.6, 128.8, 68.3–69.3 (m, 1C), 22.4, 21.8.

HRMS (ESI) exact mass calculated for  $[C_{10}H_{11}DO_2 + H]^+: 166.0973$  , found: 166.0983.



**Reaction conditions:** substrate (50.6 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), CD<sub>3</sub>OD: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(4-chlorophenyl)-2-(hydroxy-d)propan-1-one-2-d* (2ai): The Product 2ai was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 46% (25.7 mg) isolated yield. The 95% deuterium incorporation was ascertained by means of <sup>1</sup>H NMR.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.80 (d, *J* = 8.5 Hz, 2H), 7.41 (d, *J* = 8.5 Hz, 2H), 2.93 (s, 1H), 1.36 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.3, 140.6, 131.8, 130.1, 129.4, 68.8–69.4 (m, 1C), 22.2.

HRMS (ESI) exact mass calculated for  $[C_9H_8DClO_2\ +\ H]^+$  : 186.0427 , found: 186.0435.



**Reaction conditions:** substrate (50.6 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), CD<sub>3</sub>OD: MeCN: DMF = 2.0 : 2.0 : 2.0 mL,

platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*1-(3-chlorophenyl)-2-(hydroxy-d)propan-1-one-2-d* (2aj): The Product 2aj was purified by silica gel column chromatography (PE/EA = 5 : 1), colorless oil was obtained in 43% (24.0 mg) isolated yield. The 91% deuterium incorporation was ascertained by means of <sup>1</sup>H NMR.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.84 (t, *J* = 1.9 Hz, 1H), 7.74–7.71 (m, 1H), 7.54–7.51 (m, 1H), 7.39 (t, *J* = 7.9 Hz, 1H), 1.37 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.3, 135.3, 134.9, 133.9, 130.2, 128.7, 126.7, 69.5–68.9 (m, 1C), 22.0.

HRMS (ESI) exact mass calculated for  $[\mathrm{C_9H_8DClO_2}+\mathrm{H}]^+:186.0427$  , found: 186.0437.



**Reaction conditions:** substrate (63.1 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), CD<sub>3</sub>OD: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

*2-(hydroxy-d)-1,3-diphenylpropan-1-one-2-d* (2ak): The Product 2ak was purified by silica gel column chromatography (PE/EA = 5:1), white solid was obtained in 60% (41.5 mg) isolated yield. The 94% deuterium incorporation was ascertained by means of <sup>1</sup>H NMR.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.88–7.80 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.20–7.12 (m, 3H), 7.04 (d, *J* = 6.5 Hz, 2H), 3.11 (d, *J* = 14.2 Hz, 1H), 2.81 (d, *J* = 14.2 Hz, 1H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 199.12, 136.40, 133.90, 133.69, 129.26, 128.75, 127.75, 127.32, 76.47–74.03 (m, 1C), 33.88, 23.90.

HRMS (ESI) exact mass calculated for  $[C_{15}H_{11}DO_2 + K]^+:226.0688$  , found: 226.0686.

**Reaction conditions:** substrate (56.1  $\mu$ L, 0.3 mmol), <sup>*n*</sup>Bu<sub>4</sub>NBr (145.1 mg, 0.45 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), CD<sub>3</sub>OD: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2.5 h.

2-(hydroxy-d)-1-(p-tolyl)pentan-1-one-2-d (2al): The Product 2al was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 61% (35.3 mg) isolated yield. The 85% deuterium incorporation was ascertained by means of <sup>1</sup>H NMR.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.74 (d, J = 8.2 Hz, 2H), 7.23 (d, J = 8.1 Hz, 2H), 2.36 (s, 3H), 1.79–1.69 (m, 1H), 1.49–1.41 (m, 2H), 1.39–1.31 (m, 1H), 0.84 (t, J = 7.1 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 201.9, 145.1, 131.2, 129.7, 128.8, 72.3–72.9 (m, 1C), 38.2, 21.9, 18.3, 14.0.

HRMS (ESI) exact mass calculated for  $[C_{12}H_{15}DO_2 + K]^+:232.0845$  , found: 232.0840.



**Reaction conditions:** substrate (58.8 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), CD<sub>3</sub>OD: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h. *2-(hydroxy-d)-1,2-diphenylethan-1-one-2-d* (2am): The Product 2am was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 50%

(32.1 mg) isolated yield. The 87% deuterium incorporation was ascertained by means of <sup>1</sup>H NMR.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.87–7.81 (m, 2H), 7.44 (t, *J* = 7.4 Hz, 1H), 7.32 (t, *J* = 7.8 Hz, 2H), 7.28–7.16 (m, 5H), 4.35 (s, 1H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 199.1, 139.1, 134.0, 133.6, 129.2, 128.8, 128.7, 127.9, 75.7–76.3 (m, 1C).

HRMS (ESI) exact mass calculated for  $[C_{14}H_{11}DO_2 + H]^+:214.0973$  , found: 214.0981.



**Reaction conditions:** substrate (71.4 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), CD<sub>3</sub>OD: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h.

2-(hydroxy-d)-2-(4-isopropylphenyl)-1-phenylethan-1-one-2-d (2an): The Product 2an was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 40% (30.6 mg) isolated yield. The 96% deuterium incorporation was ascertained by means of <sup>1</sup>H NMR.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.84 (d, *J* = 7.2 Hz, 2H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.30 (t, *J* = 7.7 Hz, 2H), 7.18–7.16 (m, 2H), 7.08 (d, *J* = 8.1 Hz, 2H), 3.81 (s, 1H), 2.80–2.70 (m, 1H), 1.11–1.09 (m, 6H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 199.1, 149.4, 136.4, 133.9, 133.7, 129.3, 128.7, 127.7, 127.3, 75.4-76.0 (m, 1C), 33.9, 23.9.

HRMS (ESI) exact mass calculated for  $[C_{17}H_{17}DO_2 + H]^+$  : 294.1001 , found: 294.1005.



**Reaction conditions:** substrate (76.8 mg, 0.3 mmol),  ${}^{n}Bu_{4}NBr$  (116.1 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (38.2 mg, 0.36 mmol), CD<sub>3</sub>OD: MeCN: DMF = 2.0 : 2.0 : 2.0 mL, platinum as anode and cathode, undivided cell, constant current = 10 mA, r.t., Ar, 2 h.

*2-(hydroxy-d)-1,2-bis(4-methoxyphenyl)ethan-1-one-2-d* (2ao) : The Product 2ao was purified by silica gel column chromatography (PE/EA = 5 : 1), white solid was obtained in 43% (35.3 mg) isolated yield. The 93% deuterium incorporation was ascertained by means of <sup>1</sup>H NMR.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.86–7.81 (m, 2H), 7.21–7.86 (m, 2H), 6.82–6.76 (m, 4H), 3.76–3.67 (m, 6H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.4, 164.1, 159.7, 131.9, 131.6, 129.1, 126.4, 114.6, 114.0, 74.7–75.3 (m, 1C), 55.6, 55.3.

HRMS (ESI) exact mass calculated for  $[C_{16}H_{15}DO_4 + H]^+:274.1184$  , found: 274.1191.



5,6-diphenyl-2,3-dihydropyrazine (2ap). The Product 2ap was purified by silica gel

column chromatography (PE/EA = 9:1), white solid was obtained in 83% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.38–7.31 (m, 4H), 7.26–7.24 (m, 2H), 7.20–7.18 (m, 4H), 3.63 (s, 4H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 160.5, 137.9, 129.8, 128.3, 128.0, 46.0.



*2-(4-methoxyphenyl)-1-phenylethan-1-one* (2-1). The Product 2-1 was purified by silica gel column chromatography (PE/EA = 20 : 1), white solid was obtained in 43% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.03–7.96 (m, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.18 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 4.22 (s, 2H), 3.78 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 198.0, 158.7, 136.7, 133.2, 130.6, 128.7, 128.7, 126.6, 114.3, 55.4, 44.7.



*2-(4-ethylphenyl)-1-phenylethan-1-one* (2-2). The Product 2-2 was purified by silica gel column chromatography (PE/EA = 20 : 1), white solid was obtained in 48% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.05–7.97 (m, 2H), 7.59–7.50 (m, 1H), 7.44 (t, J = 7.6 Hz, 2H), 7.23–7.11 (m, 4H), 4.25 (s, 2H), 2.62 (q, J = 7.6 Hz, 2H), 1.21 (t, J = 7.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.9, 142.9, 136.8, 133.2, 131.8, 129.5, 128.7, 128.7, 128.3, 45.2, 28.6, 15.6.



*2-(4-isopropylphenyl)-1-phenylethan-1-one* (2-3). The Product 2-3 was purified by silica gel column chromatography (PE/EA = 20 : 1), white solid was obtained in 40% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.03–8.01 (m, 2H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.19 (s, 4H), 4.25 (s, 2H), 2.93–2.82 (m, 1H), 1.23 (d, *J* = 6.9 Hz, 6H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.9, 147.5, 136.8, 133.2, 131.9, 129.5, 128.8, 128.7, 126.9, 45.2, 33.9, 24.1.



*2-(4-fluorophenyl)-1-phenylethan-1-one* (2-4). The Product 2-4 was purified by silica gel column chromatography (PE/EA = 20 : 1), white solid was obtained in 45% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.01–7.99 (m, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.24–7.20 (m, 2H), 7.01 (t, *J* = 8.7 Hz, 2H), 4.26 (s, 2H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$ 197.50, 162.02 (d, J = 245.1 Hz), 136.59, 133.42, 131.17 (d, J = 8.0 Hz), 130.26 (d, J = 3.3 Hz), 128.83, 128.64, 115.63 (d, J = 21.4 Hz), 44.63.

<sup>19</sup>F NMR (376 MHz, Chloroform-*d*)  $\delta$  –116.0.



*2-(4-chlorophenyl)-1-phenylethan-1-one* (2-5). The Product 2-5 was purified by silica gel column chromatography (PE/EA = 20 : 1), white solid was obtained in 50% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 7.3 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.3 Hz, 2H), 4.25 (s, 2H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.2, 136.5, 133.5, 133.0, 133.0, 131.0, 128.9, 128.8, 128.6, 44.8.



*2-(4-bromophenyl)-1-phenylethan-1-one* (2-6). The Product 2-6 was purified by silica gel column chromatography (PE/EA = 20 : 1), white solid was obtained in 46% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.02–7.95 (m, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.50–7.42 (m, 4H), 7.13 (d, *J* = 8.3 Hz, 2H), 4.24 (s, 2H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.1, 136.5, 133.6, 133.5, 131.9, 131.4, 128.8, 128.6, 121.1, 44.9.



*1-phenyl-2-(p-tolyl)ethan-1-one* (2-7). The Product 2-7 was purified by silica gel column chromatography (PE / EA = 20 : 1), white solid was obtained in 50% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.03–7.97 (m, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.18–7.09 (m, 4H), 4.24 (s, 2H), 2.31 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.9, 136.7, 136.6, 133.2, 131.5, 129.5, 129.4, 128.7, 128.7, 45.3, 21.2.



*2-phenyl-1-(p-tolyl)ethan-1-one* (2-8). The Product 2-8 was purified by silica gel column chromatography (PE/EA = 20 : 1), white solid was obtained in 30% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.91 (d, *J* = 8.2 Hz, 2H), 7.33–7.29 (m, 2H), 7.26–7.21 (m, 5H), 4.24 (s, 2H), 2.39 (s, 3H).

<sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 197.4, 144.1, 134.9, 134.2, 129.5, 129.4, 128.9, 128.7, 126.9, 45.5, 21.7.



*1-(4-ethylphenyl)-2-phenylethan-1-one* (2-9). The Product 2-9 was purified by silica gel column chromatography (PE/EA = 20 : 1), white solid was obtained in 51% isolated yield.

<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.35–7.20 (m, 7H), 4.25 (s, 2H), 2.69 (q, *J* = 7.6 Hz, 2H), 1.24 (t, *J* = 7.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 197.4, 150.3, 134.9, 134.4, 129.0, 128.8, 128.3, 126.9, 45.6, 29.1, 15.3.

# **NMR Spectra of Products**



<sup>220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2</sup> f1 (ppm)

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## Compound 2b, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

666 64 65 64 65 73 73 73 73 66 60 60 60 60 60 60 60 60 60 60 72 72 72 72 72 72 72 72 72 72 72 72 72	42	34	37 35	
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			$\sim$	





Compound 2b, <sup>13</sup>C NMR (101 MHz, Chloroform-d)

- 202.64	138.85 134.83 133.44 133.44 129.18 128.77 128.77	- 69.39	22.41	
ОН				

220 210 200 190 180 170 180 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2 f1 (ppm) Delete the blank page.

## Compound 2c, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

79 77 25 23	09 07 05	40 67 65 65 63 63 719 119 119
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$\checkmark \lor$	$\sim$	







151.21	131.01 128.97 128.44	69.27	29.10 22.52 15.17
	$\sim$		215



- 201.99




S37





ÒН







#### Compound 2f, <sup>1</sup>H NMR (400 MHz, Chloroform-d)

7.93 7.92 7.92 7.90 7.13 7.13 7.13 7.13 7.13 7.13 7.13 7.13	<pre>{1.39 1.37</pre>



210 200

190 180





170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 f1 (ppm)

-10

20 10 0

#### Compound 2f, <sup>19</sup>F NMR (376 MHz, Chloroform-*d*)









Compound 2i, <sup>19</sup>F NMR (376 MHz, Chloroform-d)



50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -2: f1 (ppm)













#### Compound 2k, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

7.99 7.77 7.77 7.77 7.77 7.77 7.77 7.77	3.28	1.38 1.36
	Ι	$\checkmark$





## Compound 2l, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

888888888888888888888888888888888888888	32 32 34
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# Compound 2l, <sup>13</sup>C NMR (101 MHz, Chloroform-d)

- 72.91

- 20.82



mp.	Juna	,	$\sim$	-			1
93 40	72 63	19 22	66 90	34	5	6 L 0 R	5
162. 160.	135.	<u>.</u> 6	124.	122.	122.	116	2
57			$\overline{\checkmark}$	2			د



# Compound 2l, <sup>19</sup>F NMR (376 MHz, Chloroform-*d*)





Compound 2m, <sup>1</sup> H NMR (4	400 MHz,	, Chlorot	form-d)
7.72 7.72 7.72 7.72 7.72 7.75 66 7.66 7.66 7.66 7.66 7.66 7.66 7	4.00 0.03 1.09 0.03	3.57	1.38
	$\checkmark$	ï	$\mathbf{\nabla}$







-127.0 -127.5 -128.0 -128.5 -129.0 -129.5 -130.0 -130.5 -131.0 -131.5 -132.0 -132.5 -133.0 -133.5 -134.0 -134.5 -135.0 -135.5 -136.0 f1 (ppm)

#### Compound 2n, <sup>1</sup>H NMR (400 MHz, Chloroform-d)

7.85 7.7.85 7.7.55 7.557 7.7.557 7.7.557 7.7.557 7.7.557 7.7.5577 7.7.55777 7.7.5577777777	- 3.25	1.91 1.92 1.92 1.93 1.94 1.95 1
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fl (ppm)





<sup>220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2</sup> f1 (ppm)

# **Compound 2p, <sup>1</sup>H NMR (400 MHz, Chloroform-***d***)**









### Compound 2r, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

83333	12234571356773967	22255203	7 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2











S56

#### Compound 2t, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

-		
7.72 7.72 7.126 7.128 7.128 7.14 7.15 7.16 7.16 7.16 7.108 7.108 7.108 7.108 7.108 7.108 7.108 7.108	4.13	2.25
	I	1







- 198.52 145.10 139.36 129.47 129.16 129.16 129.16 129.16 127.83 75.10	- 21.82
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220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2 f1 (ppm)



S58



#### Compound 2w, <sup>1</sup>H NMR (400 MHz, Chloroform-d)

80	82	84	83	23	22	20	19	18	17	16	16	15	96	94	92	8	31
<u>∼</u> ı	ΝI	∼.	∼.	∼.	∼.	∼.	∼.	∼.	∼.	∼	∼.	∼.	ю.	<u>ن</u>	ю.	Ω.	4
_	-	_		-		-			L.	5	-	-	• -	2	_		









Compound 2x, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)





S63















S69



Compound 2ae, <sup>19</sup>F NMR (376 MHz, Chloroform-d)



50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -2: f1 (ppm)



220 210 200 150 150 170 180 150 140 130 120 110 100 50 50 50 40 30 20 10 0 -10 -2 11 (ppm)
## Compound 2ag, <sup>1</sup>H NMR (400 MHz, Chloroform-d)



## Compound 2ah <sup>1</sup>H NMR (400 MHz, Chloroform-d)

27. 124 125 125	.09 .05 .03	.36	.36
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210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)











## Compound 2ak <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

50 00	3		5	$\mathcal{O}$	5	$\mathcal{O}$	_	6	6			5	2	$\mathcal{O}$	5	$\mathcal{O}$		9	5	4		$\sim$	9	3	6
$\infty \infty$	$\infty$	5	5	5	4	4	4	<u> </u>	÷	-	-	-	—	-	0	0	0	N	2	Ċ.	-	-	Ó	×	5
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							_					-	-	~~	_	_	_		$\checkmark$	_		_	1	6	_





## Compound 2al <sup>1</sup>H NMR (400 MHz, Chloroform-d)





Compound 2al <sup>13</sup>C NMR (101 MHz, Chloroform-d)







72.92 72.72 72.50 72.28	
$\sim$	

-- 38.20 -> 21.89 -> 13.98







Compound 2ao <sup>1</sup>H NMR (400 MHz, Chloroform-d)





220 210 200 190 180 170 180 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2 f1 (ppm)



100 90 f1 (ppm) -10 





 -10

180 170









Compound 2-4, <sup>19</sup>F NMR (376 MHz, Chloroform-*d*)







Compound 2-6, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

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Compound 2-7, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)



Compound 2-8, <sup>1</sup>H NMR (400 MHz, Chloroform-d)



Compound 2-9, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)





Compound 2-9, <sup>13</sup>C NMR (101 MHz, Chloroform-d)





