

Supporting Information

Visible-light-mediated iodine-catalyzed α -hydroxylation of α -methylene ketones under aerobic conditions

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Table of Contents

1. General information.....	S2
2. Screening of the reaction conditions.....	S2-S3
3. General procedure for the synthesis of α -hydroxy ketones.....	S3
4. Gram-scale synthesis of α -hydroxy ketone 2a	S3
5. Free radical-trapping experiment.....	S4
6. Characterization data of 2a-2t	S5-S10
7. References.....	S10
8. Copies of NMR spectra of 2a-2t	S11-S30

1. General information

¹H NMR spectra were recorded on a Varian Mercury-400 Plus or Agilent Technologies DD2 (600 MHz) spectrometer. ¹³C NMR spectra were recorded on an Agilent Technologies DD2 (600 MHz) spectrometer. High-resolution mass spectra (HRMS) were performed on a Thermo Orbitrap Elite instrument with an ESI source. Melting points were measured on an XT4A apparatus (uncorrected). The reactions were monitored by TLC on silica gel plates (GF254). Reagents and solvents were commercially available, and were used without further purification. The reaction mixtures were purified by column chromatography over silica gel (PE-EtOAc).

2. Screening of the reaction conditions^a

Entry	Iodine source (mol%)	Base (mol%)	Solvent	Yield (%) ^b
1	I ₂ (20)	Na ₂ CO ₃ (50)	MeOH	0
2	I ₂ (20)	K ₂ CO ₃ (50)	MeOH	0
3	I ₂ (20)	Cs ₂ CO ₃ (50)	MeOH	0
4	I ₂ (20)	NaOAc (50)	MeOH	0
5	I ₂ (20)	KOAc (50)	MeOH	0
6	I ₂ (20)	K ₃ PO ₄ (50)	MeOH	68
7	I ₂ (20)	KO ^t Bu (50)	MeOH	35
8	I ₂ (20)	NaHCO ₃ (50)	MeOH	72
9	I ₂ (20)	Et ₃ N (50)	MeOH	trace
10	I ₂ (20)	DBU (50)	MeOH	trace
11	I ₂ (20)	DABCO (50)	MeOH	77
12	I ₂ (20)	DMAP (50)	MeOH	54
13	KI (20)	DABCO (50)	MeOH	0
14	NaI (20)	DABCO (50)	MeOH	0
15	TBAI (20)	DABCO (50)	MeOH	0
16	I ₂ (20)	DABCO (50)	EtOH	56
17	I ₂ (20)	DABCO (50)	i-PrOH	40
18	I ₂ (20)	DABCO (50)	H ₂ O	6
19	I ₂ (20)	DABCO (50)	DMSO	0

20	I ₂ (20)	DABCO (50)	DMF	0
21	I ₂ (20)	DABCO (50)	MeCN	0
22	I ₂ (20)	DABCO (50)	EtOAc	0
23	I ₂ (20)	DABCO (50)	Et ₂ O	trace
24	I ₂ (20)	DABCO (50)	THF	trace
25	I ₂ (20)	DABCO (50)	dioxane	0
26	I ₂ (20)	DABCO (50)	DCE	0
27	I ₂ (20)	DABCO (50)	toluene	0
28	I ₂ (20)	DABCO (50)	hexane	trace
29	I ₂ (10)	DABCO (10)	MeOH	24
30	I ₂ (10)	DABCO (20)	MeOH	56
31	I ₂ (10)	DABCO (30)	MeOH	22
32	I ₂ (10)	DABCO (50)	MeOH	17
33	I ₂ (20)	DABCO (10)	MeOH	trace
34	I ₂ (20)	DABCO (20)	MeOH	20
35	I ₂ (20)	DABCO (30)	MeOH	59
36	I ₂ (20)	DABCO (40)	MeOH	64
37	I ₂ (20)	DABCO (60)	MeOH	74
38	I ₂ (15)	DABCO (35)	MeOH	73
39 ^c	I ₂ (20)	DABCO (50)	MeOH	58

^aReaction conditions: **1a** (0.3 mmol), iodine source, and base in solvent (2 mL) were stirred in air atmosphere with irradiation by a 23 W compact fluorescent lamp (CFL) at room temperature for 24 h. ^bIsolated yield. ^c12 W CFL was used.

3. General procedure for the synthesis of α -hydroxy ketones

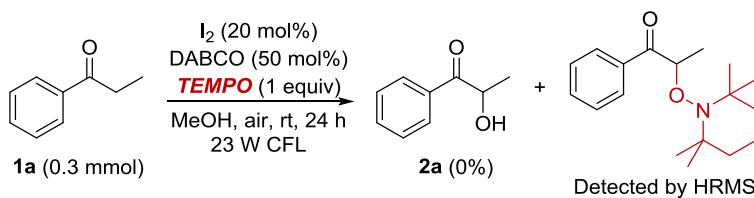
Ketone **1** (0.3 mmol), I₂ (15.2 mg, 0.06 mmol, 20 mol%), DABCO (16.8 mg, 0.15 mmol, 50 mol%) and MeOH (2 mL) were added into a 5 mL glass vial. The reaction mixture was stirred at room temperature under air atmosphere (connected to air with an injection needle) and irradiated by a 23 W compact fluorescent lamp (CFL) placed at a distance of ca. 6 cm. After completion of the reaction (monitored by TLC), the reaction mixture was purified by column chromatography on silica gel (PE-EtOAc, 10:1) to afford pure product **2**.

4. Gram-scale synthesis of α -hydroxy ketone **2a**

Ketone **1a** (1.34 g, 10.0 mmol), I₂ (0.51 g, 2.0 mmol, 20 mol%), DABCO (0.56 g, 5.0 mmol, 50 mol%) and MeOH (15 mL) were charged into a 50 mL round-bottom flask. The reaction mixture was stirred

at room temperature under air atmosphere (open flask) and irradiated by a 23 W compact fluorescent lamp (CFL). After completion of the reaction (monitored by TLC), the reaction mixture was diluted with H₂O (20 mL), and added saturated Na₂S₂O₃ until the disappearance of umber. Then the mixture was extracted with CH₂Cl₂ (20 mL × 3). The combined organic phase was dried over MgSO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (PE-EtOAc, 10:1) to afford pure product **2a** (0.92 g, 61%).

5. Free radical-trapping experiment



Ketone **1a** (40.6 mg, 0.3 mmol), I₂ (15.2 mg, 0.06 mmol, 20 mol%), DABCO (16.8 mg, 0.15 mmol, 50 mol%) and MeOH (2 mL) were added into a 5 mL glass vial. The reaction mixture was stirred at room temperature under air atmosphere (connected to air with an injection needle) and irradiated by a 23 W CFL for 24 h. After completion of the reaction, no desired product **2a** was detected by TLC as well as HRMS, indicating that the reaction was inhibited completely. Meanwhile, a free radical-trapping adduct of TEMPO with carbon radical **B** was observed by HRMS analysis of the reaction solution (Figure S1).

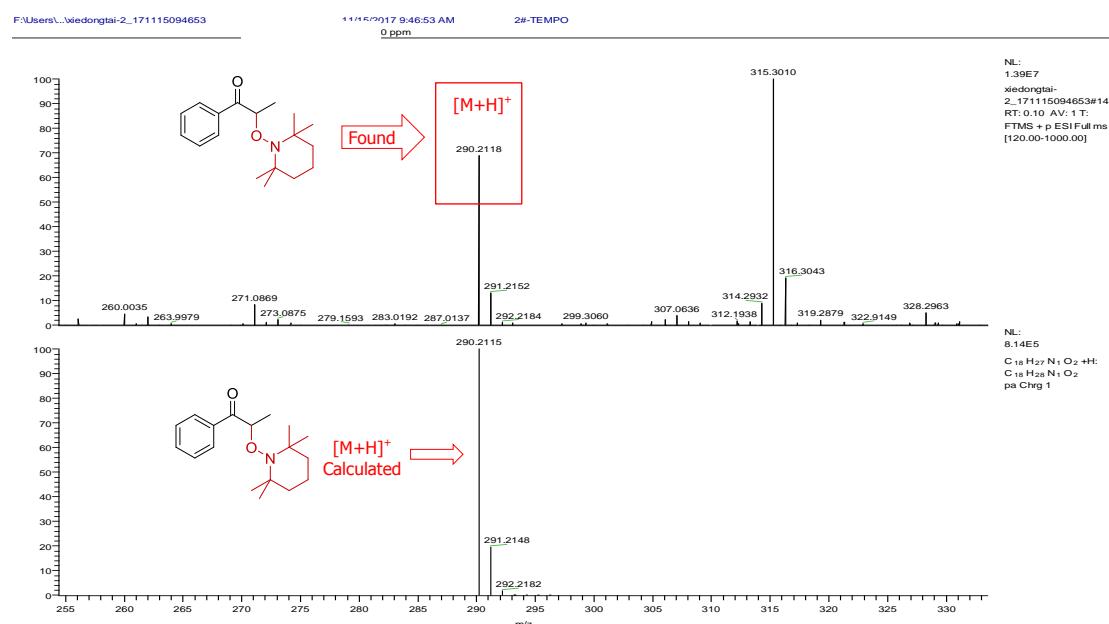
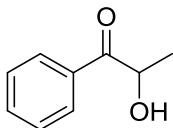


Figure S1. HRMS analysis of the adduct of carbon radical **B** with TEMPO

6. Characterization data of 2a–2t

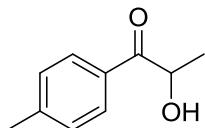


2-hydroxy-1-phenylpropan-1-one (2a)^[1]

TLC (PE/EtOAc = 5/1) R_f = 0.32; colorless oil; yield: 34.5 mg (77%).

¹H NMR (600 MHz, CDCl₃): δ 7.92 (d, J = 8.4 Hz, 2H), 7.61 (t, J = 7.2 Hz, 1H), 7.49 (t, J = 7.2 Hz, 2H), 5.16–5.14 (m, 1H), 3.79 (d, J = 4.2 Hz, 1H), 1.44 (d, J = 7.2 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 202.3, 133.9, 133.3, 128.8, 128.6, 69.3, 22.3.

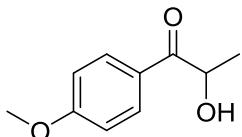


2-hydroxy-1-p-tolylpropan-1-one (2b)^[1]

TLC (PE/EtOAc = 5/1) R_f = 0.31; pale yellow oil; yield: 30.5 mg (62%).

¹H NMR (600 MHz, CDCl₃): δ 7.82 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 7.8 Hz, 2H), 5.15–5.10 (m, 1H), 3.80 (d, J = 6.6 Hz, 1H), 2.43 (s, 3H), 1.43 (d, J = 7.2 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 201.9, 145.0, 130.7, 129.5, 128.7, 69.1, 22.4, 21.7.

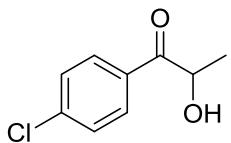


2-hydroxy-1-(4-methoxyphenyl)propan-1-one (2c)^[1]

TLC (PE/EtOAc = 5/1) R_f = 0.19; pale yellow oil; yield: 29.8 mg (55%).

¹H NMR (600 MHz, CDCl₃): δ 7.91 (d, J = 8.4 Hz, 2H), 6.97 (d, J = 8.0 Hz, 2H), 5.12–5.07 (m, 1H), 3.88 (s, 3H), 3.82 (d, J = 6.0 Hz, 1H), 1.44 (d, J = 7.2 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 200.6, 164.1, 131.0, 126.1, 114.1, 68.8, 55.5, 22.6.

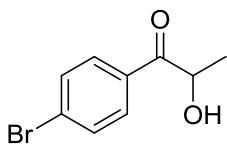


1-(4-chlorophenyl)-2-hydroxypropan-1-one (2d)^[1]

TLC (PE/EtOAc = 5/1) R_f = 0.25; white solid; yield: 41.5 mg (75%); m.p. 120–123 °C.

¹H NMR (600 MHz, CDCl₃): δ 7.86 (d, J = 8.4 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H), 5.13–5.08 (m, 1H), 3.69 (d, J = 6.0 Hz, 1H), 1.43 (d, J = 7.2 Hz, 3H)

¹³C NMR (150 MHz, CDCl₃): δ 201.2, 140.5, 131.6, 130.0, 129.2, 69.3, 22.2.

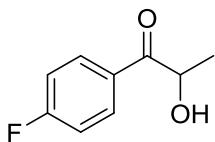


1-(4-bromophenyl)-2-hydroxypropan-1-one (2e)^[1]

TLC (PE/EtOAc = 5/1) R_f = 0.25; white solid; yield: 56.6 mg (82%); m.p. 153–156 °C.

^1H NMR (600 MHz, CDCl₃): δ 7.79 (d, J = 8.4 Hz, 2H), 7.65 (d, J = 7.8 Hz, 2H), 5.12–5.08 (m, 1H), 3.67 (d, J = 6.0 Hz, 1H), 1.43 (d, J = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, CDCl₃): δ 201.4, 132.2, 132.0, 130.0, 129.2, 69.3, 22.2.

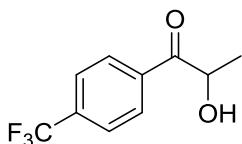


1-(4-fluorophenyl)-2-hydroxypropan-1-one (2f)^[2]

TLC (PE/EtOAc = 5/1) R_f = 0.26; colorless oil; yield: 36.2 mg (72%).

^1H NMR (600 MHz, CDCl₃): δ 7.98–7.95 (m, 2H), 7.19–7.16 (m, 2H), 5.14–5.09 (m, 1H), 3.71 (d, J = 6.0 Hz, 1H), 1.44 (d, J = 6.6 Hz, 3H).

^{13}C NMR (150 MHz, CDCl₃): δ 200.7, 166.1 (d, J = 255.2 Hz), 131.3 (d, J = 9.5 Hz), 129.7 (d, J = 3.2 Hz), 116.1 (d, J = 21.9 Hz), 69.2, 22.3.

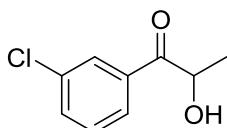


2-hydroxy-1-(4-(trifluoromethyl)phenyl)propan-1-one (2g)^[3]

TLC (PE/EtOAc = 5/1) R_f = 0.26; colorless oil; yield: 45.0 mg (61%).

^1H NMR (600 MHz, CDCl₃): δ 8.03 (d, J = 8.4 Hz, 2H), 7.77 (d, J = 7.8 Hz, 2H), 5.19–5.15 (m, 1H), 3.64 (d, J = 5.4 Hz, 1H), 1.45 (d, J = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, CDCl₃): δ 201.5, 136.2, 135.1 (q, J = 32.7 Hz), 128.9, 125.9 (q, J = 3.8 Hz), 123.3 (q, J = 271.2 Hz), 69.6, 21.8.

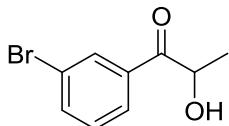


1-(3-chlorophenyl)-2-hydroxypropan-1-one (2h)^[1]

TLC (PE/EtOAc = 5/1) R_f = 0.29; pale yellow oil; yield: 35.4 mg (64%).

¹H NMR (400 MHz, CDCl₃): δ 7.91 (s, 1H), 7.80 (d, J = 7.6 Hz, 1H), 7.60 (d, J = 8.0 Hz, 1H), 7.46 (t, J = 7.6 Hz, 1H), 5.15–5.09 (m, 1H), 3.68 (d, J = 6.4 Hz, 1H), 1.45 (d, J = 6.8 Hz, 3H)

¹³C NMR (150 MHz, CDCl₃): δ 201.2, 135.3, 134.9, 133.9, 130.2, 128.7, 126.6, 69.5, 22.1.

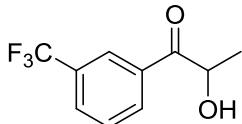


1-(3-bromophenyl)-2-hydroxypropan-1-one (2i)^[4]

TLC (PE/EtOAc = 5/1) R_f = 0.29; pale yellow oil; yield: 48.3 mg (70%).

¹H NMR (600 MHz, CDCl₃): δ 8.06 (s, 1H), 7.83 (d, J = 7.8 Hz, 1H), 7.74 (d, J = 7.8 Hz, 1H), 7.38 (t, J = 7.8 Hz, 1H), 5.12–5.08 (m, 1H), 3.64 (d, J = 6.6 Hz, 1H), 1.44 (d, J = 7.2 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 201.2, 136.8, 135.2, 131.6, 130.4, 127.1, 123.2, 69.4, 22.1.

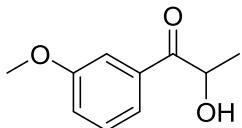


2-hydroxy-1-(3-(trifluoromethyl)phenyl)propan-1-one (2j)^[2]

TLC (PE/EtOAc = 5/1) R_f = 0.26; pale yellow oil; yield: 37.8 mg (51%).

¹H NMR (600 MHz, CDCl₃): δ 8.19 (s, 1H), 8.10 (d, J = 7.8 Hz, 1H), 7.88 (d, J = 7.8 Hz, 1H), 7.66 (t, J = 7.8 Hz, 1H), 5.19–5.15 (m, 1H), 3.65 (d, J = 6.0 Hz, 1H), 1.46 (d, J = 7.2 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 201.2, 134.0, 131.7, 131.6 (d, J = 33.2 Hz), 130.3 (q, J = 3.6 Hz), 129.6, 125.4 (q, J = 3.8 Hz), 123.4 (d, J = 273.5 Hz), 69.5, 22.0.

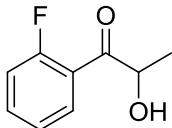


2-hydroxy-1-(3-methoxyphenyl)propan-1-one (2k)^[5]

TLC (PE/EtOAc = 5/1) R_f = 0.26; pale yellow oil; yield: 30.8 mg (57%).

¹H NMR (600 MHz, CDCl₃): δ 7.47–7.46 (m, 2H), 7.39 (t, J = 7.8 Hz, 1H), 7.15 (d, J = 7.8 Hz, 1H), 5.15–5.10 (m, 1H), 3.86 (s, 3H), 3.75 (d, J = 6.6 Hz, 1H), 1.44 (d, J = 7.2 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 202.2, 159.9, 134.7, 129.8, 121.1, 120.3, 113.0, 69.4, 55.5, 22.3.

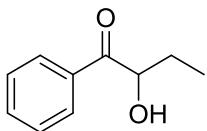


1-(2-fluorophenyl)-2-hydroxypropan-1-one (2l)^[1]

TLC (PE/EtOAc = 5/1) R_f = 0.40; pale yellow oil; yield: 28.3 mg (56%).

¹H NMR (600 MHz, CDCl₃): δ 7.93 (t, *J* = 7.8 Hz, 1H), 7.60–7.57 (m, 1H), 7.29 (t, *J* = 7.8 Hz, 1H), 7.18–7.15 (m, 1H), 5.06–5.03 (m, 1H), 3.77 (s, 1H), 1.40 (d, *J* = 7.2 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃) δ: 200.9 (d, *J* = 4.5 Hz), 161.5 (d, *J* = 254.0 Hz), 135.5 (d, *J* = 9.2 Hz), 131.1 (d, *J* = 2.7 Hz), 124.9 (d, *J* = 3.3 Hz), 122.2 (d, *J* = 13.5 Hz), 116.7 (d, *J* = 23.3 Hz), 72.7 (d, *J* = 9.2 Hz), 20.7.

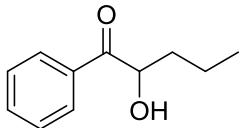


2-hydroxy-1-phenylbutan-1-one (2m) ^[4]

TLC (PE/EtOAc = 5/1) R_f = 0.40; pale yellow oil; yield: 28.6 mg (58%).

¹H NMR (400 MHz, CDCl₃): δ 7.92 (d, *J* = 7.6 Hz, 2H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 5.09–5.05 (m, 1H), 3.71 (d, *J* = 6.0 Hz, 1H), 1.99–1.93 (m, 1H), 1.67–1.57 (m, 1H), 0.94 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 202.1, 133.9, 133.7, 128.8, 128.5, 73.9, 28.8, 8.8.

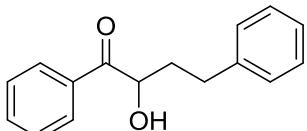


2-hydroxy-1-phenylpentan-1-one (2n) ^[4]

TLC (PE/EtOAc = 5/1) R_f = 0.49; pale yellow oil; yield: 28.7 mg (54%).

¹H NMR (600 MHz, CDCl₃): δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.61 (t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.2 Hz, 2H), 5.09–5.06 (m, 1H), 3.69 (d, *J* = 6.6 Hz, 1H), 1.84–1.79 (m, 1H), 1.54–1.51 (m, 2H), 1.45–1.40 (m, 1H), 0.91 (t, *J* = 6.0 Hz, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 202.2, 133.9, 133.7, 128.8, 128.5, 72.9, 38.0, 18.2, 13.8.

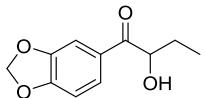


2-hydroxy-1,4-diphenylbutan-1-one (2o) ^[1]

TLC (PE/EtOAc = 5/1) R_f = 0.45; pale yellow oil; yield: 36.3 mg (50%).

¹H NMR (600 MHz, CDCl₃): δ 7.78–7.60 (m, 2H), 7.60–7.57 (m, 1H), 7.46–7.43 (m, 2H), 7.29–7.26 (m, 2H), 7.21–7.17 (m, 3H), 5.05–5.02 (m, 1H), 3.75 (d, *J* = 6.0 Hz, 1H), 2.88–2.76 (m, 2H), 2.19–2.13 (m, 1H), 1.85–1.80 (m, 1H).

¹³C NMR (150 MHz, CDCl₃): δ 201.9, 141.0, 133.9, 133.4, 128.8, 128.6, 128.5, 128.4, 126.1, 72.2, 37.7, 31.3.



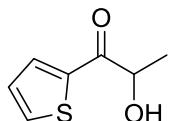
1-(benzo[d][1,3]dioxol-5-yl)-2-hydroxybutan-1-one (2p)

TLC (PE/EtOAc = 5/1) R_f = 0.28; white solid; yield: 15.2 mg (24%); m.p. 76–78 °C.

^1H NMR (600 MHz, CDCl₃): δ 7.50 (dd, J = 8.4, 1.8 Hz, 1H), 7.39 (d, J = 1.8 Hz, 1H), 6.87 (d, J = 8.4 Hz, 1H), 6.06 (s, 2H), 4.96–4.93 (m, 1H), 3.69 (d, J = 6.6 Hz, 1H), 1.96–1.89 (m, 1H), 1.63–1.56 (m, 1H), 0.93 (t, J = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, CDCl₃): δ 199.9, 152.5, 148.3, 128.2, 124.9, 108.2, 108.1, 102.0, 73.6, 29.2, 8.9.

HRMS (ESI): *m/z* [M+H]⁺ calcd for C₁₁H₁₃O₄: 209.0808; found: 209.0806.

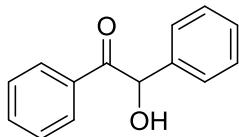


2-hydroxy-1-(thiophen-2-yl)propan-1-one (2q) ^[6]

TLC (PE/EtOAc = 5/1) R_f = 0.21; pale yellow oil; yield: 29.6 mg (63%).

^1H NMR (600 MHz, CDCl₃): δ 7.76 (d, J = 3.6 Hz, 1H), 7.73 (d, J = 4.8 Hz, 1H), 7.19–7.18 (m, 1H), 4.98–4.95 (m, 1H), 3.56 (s, 1H), 1.53 (d, J = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, CDCl₃): δ 194.9, 139.5, 134.7, 133.0, 128.3, 70.2, 23.1.

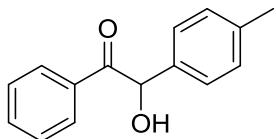


2-hydroxy-1,2-diphenylethan-1-one (2r) ^[6]

TLC (PE/EtOAc = 5/1) R_f = 0.33; white solid; yield: 24.3 mg (38%); m.p. 131–134 °C. (lit.^[2] 136–137 °C)

^1H NMR (400 MHz, CDCl₃): δ 7.93–7.90 (m, 2H), 7.54–7.50 (m, 1H), 7.42–7.38 (m, 2H), 7.34–7.27 (m, 5H), 5.96 (d, J = 6.0 Hz, 1H), 4.56 (d, J = 6.4 Hz, 1H).

^{13}C NMR (150 MHz, CDCl₃): δ 198.9, 139.0, 133.9, 133.5, 129.12, 129.10, 128.7, 128.6, 127.7, 76.2.

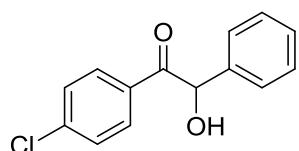


2-hydroxy-1-phenyl-2-(p-tolyl)ethan-1-one (2s) ^[7]

TLC (PE/EtOAc = 5/1) R_f = 0.36; white solid; yield: 30.5 mg (45%); m.p. 110–113 °C. (lit.^[9] 116–117 °C)

¹H NMR (600 MHz, CDCl₃): δ 7.91–7.90 (m, 2H), 7.52–7.49 (m, 1H), 7.40–7.37 (m, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 7.8 Hz, 2H), 5.92 (s, 1H), 2.28 (s, 3H).

¹³C NMR (150 MHz, CDCl₃): δ 199.0, 138.4, 136.0, 133.8, 133.5, 129.8, 129.1, 128.6, 127.6, 76.0, 21.1.



1-(4-chlorophenyl)-2-hydroxy-2-phenylethan-1-one (2t) [8]

TLC (PE/EtOAc = 5/1) R_f = 0.31; white solid; yield: 25.2 mg (34%); m.p. 89–91 °C. (lit.^[9] 88–89 °C)

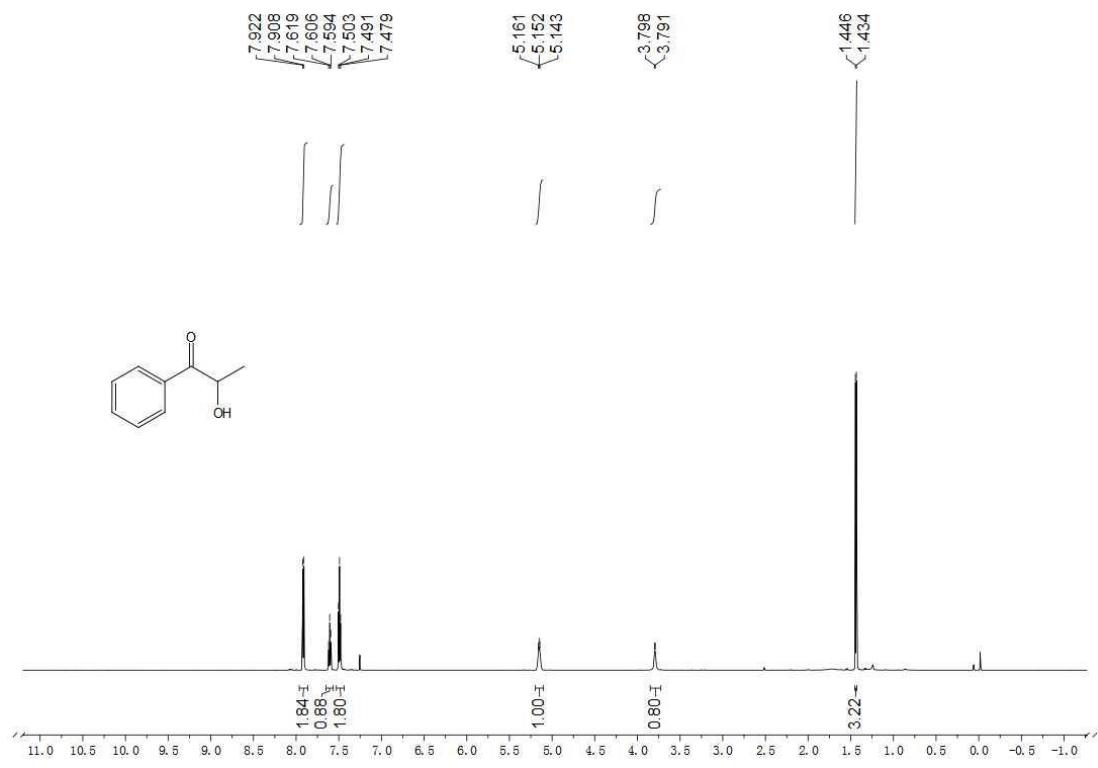
¹H NMR (600 MHz, CDCl₃): δ 7.85–7.83 (m, 2H), 7.37–7.35 (m, 2H), 7.33–7.27 (m, 5H), 5.89 (d, *J* = 6.0 Hz, 1H), 4.47 (d, *J* = 5.4 Hz, 1H).

¹³C NMR (150 MHz, CDCl₃): δ 197.8, 140.4, 138.7, 131.7, 130.5, 129.2, 129.1, 128.8, 127.7, 76.3.

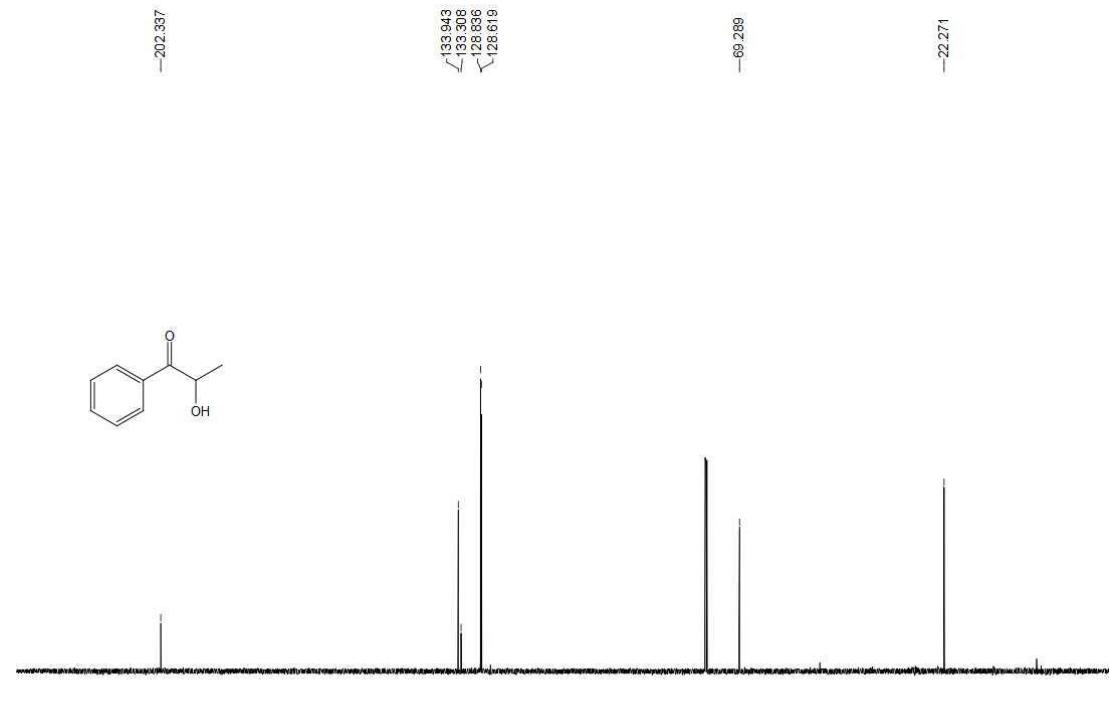
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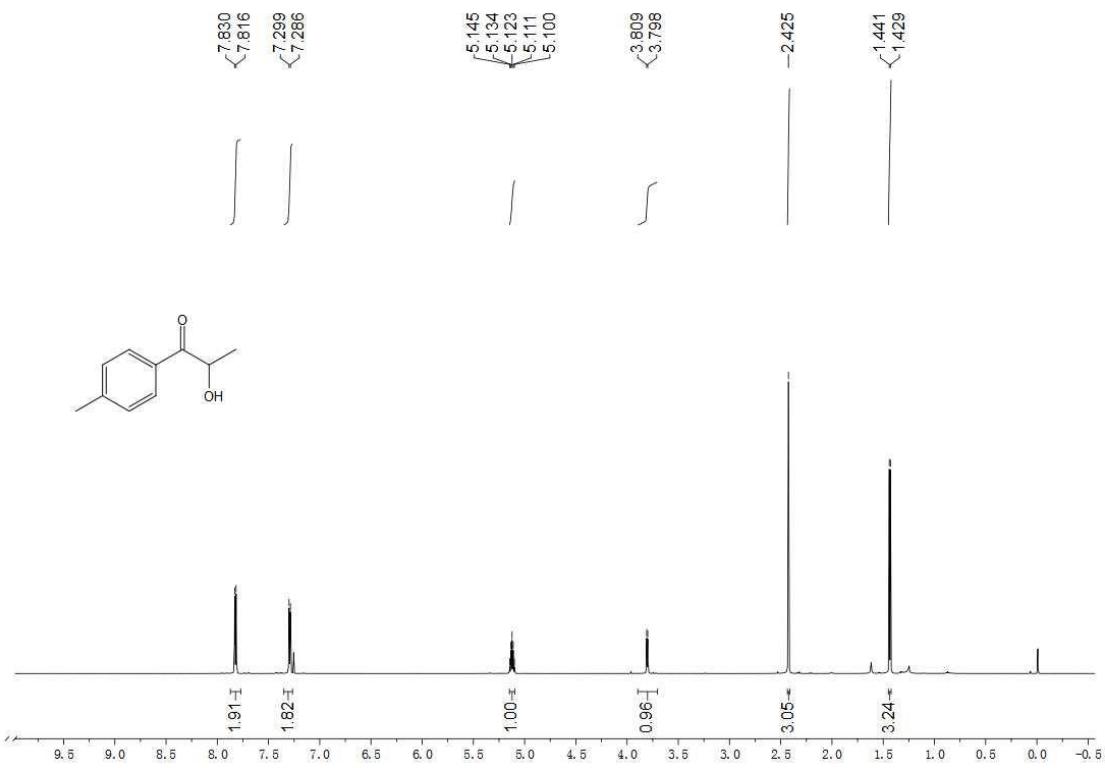
8. Copies of NMR spectra of 2a–2t



¹H NMR of 2a



¹³C NMR of 2a



¹H NMR of **2b**



¹³C NMR of **2b**

