# Selective oxidation of alkenes to carbonyls under

# mild conditions

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# **Supporting Information**

# **Table of contents**

Ge	neral Information	2
1.	Experimental Section	2
2.	Characterization of Products	7
3.	References	20
4.	Copies of <sup>1</sup> H and <sup>13</sup> C NMR Spectra	21

# **General Information**

All reagents and deuterated solvents were commercially available and used without further purification. All products were separated by silica gel (200-300 mesh) column chromatography with petroleum ether (PE) (60-90°C) and ethyl acetate (EA). <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker Advance 500 spectrometer at ambient temperature with CDCl<sub>3</sub> or CD<sub>3</sub>SOCD<sub>3</sub> as solvent and tetramethylsilane (TMS) as the internal standard. Analytical thin layer chromatography (TLC) was performed on Merk precoated TLC (silica gel 60 F254) plates. Compounds for HRMS were analyzed by positive mode electrospray ionization (ESI) using Agilent 6530 QTOF mass spectrometer. The photoreactor (PL-SX100A) and condenser (CW-5000) were purchased from Beijing Princess Technology Co., Ltd. The Schlenk tube used in photocatalysis was purchased from Beijing Synthware Glass.



Figure S1. The photoreactor and condenser used for selective oxidation of alkenes to carbonyls (Distance from the light source to the reaction tube: 2.0 cm; Power: 10 W).

# **1. Experimental Section**

# 1.1 Optimization of reaction conditions

 Table S1. Optimization of light source <sup>a</sup>

	1,4-dioxane (1) H <sub>2</sub> O, LED, O <sub>2</sub> 1a	00 mol%) , rt, 12 h	*	52 OH	
	T : 14	A 11:4:	Time (h)	Yield (%) <sup>b</sup>	
Entry	Light source	Additive		3	52
1	420–425 nm	1,4-Dioxane	12	trace	trace
2	410–415 nm	1,4-Dioxane	12	21	trace
3	400–405 nm	1,4-Dioxane	12	44	trace
4	395–400 nm	1,4-Dioxane	12	39	10
5	360–365 nm	1,4-Dioxane	12	33	37
6	-	1,4-Dioxane	12	0	0

<sup>a</sup> Reaction conditions: **1a** (0.5 mmol), 1,4-dioxane (100 mol%), H<sub>2</sub>O (3.0 mL), LED, rt, O<sub>2</sub> (balloon), 12 h. <sup>b</sup> Isolated yields.

 Table S2. Optimization of additive<sup>a</sup>

		additive (100 mol%) 400-405 nm LED H <sub>2</sub> O, O <sub>2</sub> , rt, 12 h 3	+			
Entry	Light source	Additive	Time (h) -	Yield	Yield (%) <sup>b</sup>	
	8			3	52	
1	400–405 nm	1,4-Dioxane	12	44	trace	
2	400–405 nm	1,2-Diethoxyethan	12	18	trace	
3	400–405 nm	Bis(2-ethoxyethyl)ether	12	28	trace	
4	400–405 nm	1,2-Dimethoxyethane	12	46	10	
5	400–405 nm	Bis(2-methoxy ethyl)ether	12	52	trace	
6	400–405 nm	Tetrahydrofuran	12	56	trace	
7	400–405 nm	γ-Valerolactone	12	0	0	
8	400–405 nm	3,4-Epoxytetrahydrofuran	12	0	0	
9	400–405 nm	2,5-Dimethyl tetrahydrofuran	12	42	trace	
10	400–405 nm	2-Methyl tetrahydrofuran	12	55	trace	
11	400–405 nm	Tetrahydrofurfurylchloride	12	32	trace	
12	400–405 nm		12	0	0	

 $^a$  Reaction conditions: 1a (0.5 mmol), additive (100 mol%), H\_2O (3.0 mL), 400-405 nm LED, rt, O\_2 (balloon), 12 h.  $^b$  Isolated yields.

Table S3.	. Optimization	of reaction	time,	additive	dosage and	atmosphere <sup><i>a</i></sup>
	1		,		0	1

0		(x mol%) 5 nm LED 20, rt	+		
	18	3		Vield (%)	
Entry	Light source	Additive	Time (h)	3	<u>52</u>
1	400–405 nm	Tetrahydrofuran	12	56	trace
2	400–405 nm	Tetrahydrofuran	18	81	12
3	400–405 nm	Tetrahydrofuran	24	71	22
4	400–405 nm	Tetrahydrofuran	36	49	42
5	400–405 nm	Tetrahydrofuran	48	28	59
6	400–405 nm	Tetrahydrofuran	60	trace	81
7°	400–405 nm	Tetrahydrofuran	18	82	9
8 <sup>d</sup>	400–405 nm	Tetrahydrofuran	18	70	9
9e	400–405 nm	Tetrahydrofuran	18	80	15
$10^{c,f}$	400–405 nm	Tetrahydrofuran	18	36	trace
11 <sup>c,g</sup>	400–405 nm	Tetrahydrofuran	18	0	0

<sup>a</sup> Reaction conditions: **1a** (0.5 mmol), tetrahydrofuran (100 mol%), H<sub>2</sub>O (3.0 mL), 400-405 nm LED, rt, O<sub>2</sub> (balloon). <sup>b</sup> Isolated yields. <sup>c</sup> Additive (50 mol%). <sup>d</sup> Additive (30 mol%). <sup>e</sup> Additive (150 mol%). <sup>f</sup> Under air atmosphere. <sup>g</sup> Under N<sub>2</sub> atmosphere.

0	THF (50           400-405 r           H <sub>2</sub> O, C	$\frac{\text{mol}\%)}{\text{m LED}} \longrightarrow 0$	*	52	
Entry	try Light source	Additivo	Time	Yield (%)	
		Additive		3	52
1	360–365 nm	Tetrahydrofuran	12	43	50
2	360–365 nm	Tetrahydrofuran	18	25	65
3	360–365 nm	Tetrahydrofuran	24	11	77
4	360–365 nm	Tetrahydrofuran	36	trace	85
5	360–365 nm	Tetrahydrofuran	48	trace	83

Table S4. Optimization of reaction time for the synthesis of carboxylic acid under 360–365 nm LED<sup>a</sup>

<sup>a</sup> Reaction conditions: **1a** (0.5 mmol), tetrahydrofuran (50 mol%), H<sub>2</sub>O (3.0 mL), 360-365 nm LED, rt, O<sub>2</sub> (balloon). <sup>b</sup> Isolated yields.

#### 1.2 General procedure for the synthesis of aldehydes (3-19)



To a 15 mL tube was added olefins (1) (0.5 mmol), THF (50 mol%) and H<sub>2</sub>O (3.0 mL). The above mixture was vigorous stirred under O<sub>2</sub> (balloon) with the irradiation of light (LEDs, 400-405 nm, 10 W) for 18 hours. After then, the resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 10:1) to afford the target product.

#### **1.3** General procedure for the synthesis of ketones (20-51)

$$\begin{array}{c|c} & THF (50 \text{ mol}\%) \\ \hline R & 400-405 \text{ nm LED} \\ 1 & H_2O, O_2, \text{ rt, 18 h} \end{array} \xrightarrow{P} R' \\ \hline 20-51 \end{array}$$

To a 15 mL tube was added olefins (1) (0.5 mmol), THF (50 mol%) and H<sub>2</sub>O (3.0 mL). The above mixture was vigorous stirred under O<sub>2</sub> (balloon) with the irradiation of light (LEDs, 400-405 nm, 10 W) for 18 hours. After then, the resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 10:1) to afford the target product.

#### 1.4 General procedure for the synthesis of carboxylic acids (52-64)



To a 15 mL tube was added olefins (1) (0.5 mmol), THF (50 mol%) and  $H_2O$  (3.0 mL). The above mixture was vigorous stirred under  $O_2$  (balloon) with the irradiation of light (LEDs, 360-365 nm, 10 W) for 36 hours. After then, the resulting aqueous phase was extracted with ethyl acetate and the

collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 3:1) to afford the target product.

# 1.5 General procedure for the large-scale synthesis of aldehyde (3)



To a 100 mL flask was added olefin (1a) (5.0 mmol), THF (50 mol%) and H<sub>2</sub>O (30 mL). The above mixture was vigorous stirred under O<sub>2</sub> (balloon) with the irradiation of light (LEDs, 400-405 nm, 10 W) for 18 hours. After then, the resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 10:1) to afford the target product.

# 1.6 General procedure for the large-scale synthesis of ketone (40)



To a 100 mL flask was added olefins (1x) (5.0 mmol), THF (50 mol%) and H<sub>2</sub>O (30 mL). The above mixture was vigorous stirred under O<sub>2</sub> (balloon) with the irradiation of light (LEDs, 400-405 nm, 10 W) for 18 hours. After then, the resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 10:1) to afford the target product.

# **1.7 General procedure for the large-scale synthesis of carboxylic acid (52)**



To a 100 mL flask was added olefin (1a) (5.0 mmol), THF (50 mol%) and H<sub>2</sub>O (30 mL). The above mixture was vigorous stirred under O<sub>2</sub> (balloon) with the irradiation of light (LEDs, 360-365 nm, 10 W) for 36 hours. After then, the resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 3:1) to afford the target product.

# 1.8 General procedure for the synthesis of 2-phenyl-1*H*-benzo[*d*]imidazole (67)



To a dried 15 mL tube was added acetophenone (**20**) (0.5 mmol), *o*-phenylenediamine (OPD, 0.5 mmol), SeO<sub>2</sub> (0.75 mmol) and toluene (2.0 mL). The mixture was stirred at 100 °C for 12 h. After completion, the reaction mixture was poured into water. The resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 8:1) to afford the target product **67**.

#### 1.9 General procedure for the synthesis of chalcone (68)



To a dried 15 mL tube was added acetophenone (**20**) (0.5 mmol), benzaldehyde (**4**) (0.5 mmol), NaOH (10 mol%) and EtOH (3 mL). The mixture was stirred at room temperature for 6 h. After completion, the reaction mixture was poured into water. The resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 8:1) to afford the target product **68**.

#### 1.10 General procedure for the synthesis of acetophenone oxime (69)



To a dried 15 mL tube was added acetophenone (**20**) (0.5 mmol), hydroxylamine hydrochloride (1.0 mmol), NaOAc (1.0 mmol) and EtOH (10 mL). The mixture was stirred at room temperature for 1 h. After completion, the reaction mixture was poured into water. The resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 8:1) to afford the target product **69**.

#### 1.11 General procedure for the synthesis of N-phenylacetamide (70)



To a dried 15 mL tube was added acetophenone (**20**) (0.5 mmol), hydroxylamine-*O*-sulfonic acid (0.8 mmol),  $ZnCl_2$  (10 mol%) and  $H_2O$  (2 mL). The mixture was stirred at room temperature for 8 h. After completion, the reaction mixture was poured into water. The resulting aqueous phase was extracted with ethyl acetate and the collected organic layer was washed with brine, dried with MgSO<sub>4</sub>. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EA = 5:1) to afford the target product **70**.



Scheme S1. Investigation of specific alkenes for selective oxidation



Scheme S2. Mechanistic studies of selective oxidation of alkenes to carbonyls

# 2. Characterization of Products

## 4-Methoxybenzaldehyde (3)<sup>1</sup>



Obtained as a colourless liquid (56 mg, 82% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  9.89 (s, 1H), 7.84 (d, J = 8.9 Hz, 2H), 7.03 – 6.98 (m, 2H), 3.89 (s, 3H).

## Benzaldehyde (4)<sup>2</sup>



Obtained as a colourless liquid (41 mg, 77% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.04 (s, 1H), 7.91 (d, J = 6.9 Hz, 2H), 7.65 (t, J = 7.4 Hz, 1H), 7.55 (t, J = 7.6 Hz, 2H).

# [1,1'-Biphenyl]-4-carbaldehyde (5)<sup>2</sup>



Obtained as a white solid (72 mg, 79% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.09 (s, 1H), 7.99 (d, J = 8.3 Hz, 2H), 7.79 (d, J = 8.3 Hz, 2H), 7.67 (d, J = 7.1 Hz, 2H), 7.52 (t, J = 7.5 Hz, 2H), 7.45 (t, J = 7.3 Hz, 1H).

# 4-Fluorobenzaldehyde (6)<sup>2</sup>



Obtained as a faint yellow liquid (51 mg, 82% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  9.99 (s, 1H), 7.93 (dd, J = 8.6, 5.4 Hz, 2H), 7.23 (t, J = 8.6 Hz, 2H).

## 4-Chlorobenzaldehyde (7)<sup>2</sup>



Obtained as a white solid (56 mg, 80% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.01 (s, 1H), 7.85 (d, J = 8.4 Hz, 2H), 7.54 (d, J = 8.3 Hz, 2H).

## 4-(Trifluoromethyl)benzaldehyde (8)<sup>2</sup>



Obtained as a colourless liquid (64 mg, 74% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.11 (s, 1H), 8.02 (d, J = 8.0 Hz, 2H), 7.81 (d, J = 8.2 Hz, 2H).

# 3-Methylbenzaldehyde (9)<sup>2</sup>



Obtained as a colourless liquid (48 mg, 80% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.01 (s, 1H), 7.70 (d, J = 8.1 Hz, 2H), 7.48 – 7.42 (m, 2H), 2.45 (s, 3H).

## 3-Methoxybenzaldehyde (10)<sup>2</sup>



Obtained as a colourless liquid (56 mg, 82% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  9.99 (s, 1H), 7.49 – 7.44 (m, 2H), 7.42 – 7.39 (m, 1H), 7.19 (dt, *J* = 6.3, 2.6 Hz, 1H), 3.88 (s, 3H).

## 3-Fluorobenzaldehyde (11)<sup>2</sup>



Obtained as a faint yellow liquid (48 mg, 77% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.01 (s, 1H), 7.70 (d, J = 7.6 Hz, 1H), 7.61 – 7.51 (m, 2H), 7.35 (td, J = 8.3, 2.7 Hz, 1H).

# 3-Chlorobenzaldehyde (12)<sup>2</sup>



Obtained as a colourless liquid (52 mg, 74% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.00 (s, 1H), 7.87 (t, J = 1.8 Hz, 1H), 7.79 (d, J = 7.6 Hz, 1H), 7.61 (ddd, J = 7.8, 4.5, 1.0 Hz, 1H), 7.51 (t, J = 7.8 Hz, 1H).

# 3-Bromobenzaldehyde (13)<sup>3</sup>



Obtained as a colourless liquid (65 mg, 70% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 9.97 (s, 1H), 8.02 (d, *J* = 1.6 Hz, 1H), 7.86 – 7.81 (m, 1H), 7.79 – 7.72 (m, 1H), 7.47 – 7.41 (m, 1H).

# 2-Methylbenzaldehyde (14)<sup>5</sup>



Obtained as a colourless liquid (46 mg, 77% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 10.29 (s, 1H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 1H), 2.70 (s, 3H).

# 2-Fluorobenzaldehyde (15)<sup>2</sup>



Obtained as a colourless liquid (45 mg, 73% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.39 (s, 1H), 7.89 (t, *J* = 7.4 Hz, 1H), 7.65 - 7.59 (m, 1H), 7.31 - 7.26 (m, 1H), 7.22 - 7.16 (m, 1H).

# 2-Chlorobenzaldehyde (16)<sup>2</sup>



Obtained as a colourless liquid (49 mg, 70% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 10.51 (s, 1H), 7.94 (dt, *J* = 7.7, 1.7 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.47 (d, *J* = 8.1 Hz, 1H), 7.40 (dd, *J* = 11.1, 3.9 Hz, 1H).

## 2-Bromobenzaldehyde (17)<sup>2</sup>



Obtained as a faint yellow liquid (55 mg, 59% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.39 (s, 1H), 7.97 – 7.89 (m, 1H), 7.71 – 7.64 (m, 1H), 7.46 (ddd, J = 4.9, 4.2, 2.1 Hz, 2H).

# 2-Naphthaldehyde (18)<sup>2</sup>



Obtained as a white solid (51 mg, 65% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.15 (s, 1H), 8.33 (s, 1H), 7.99 (d, J = 8.1 Hz, 1H), 7.96 – 7.88 (m, 3H), 7.63 (dd, J = 12.2, 4.0 Hz, 1H), 7.58 (t, J = 7.5 Hz, 1H).

## Picolinaldehyde (19)6



Obtained as a faint yellow liquid (28 mg, 52% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  10.09 (s, 1H), 8.81 (dd, J = 6.0, 2.4 Hz, 1H), 8.02 – 7.94 (m, 1H), 7.89 (dd, J = 2.3, 1.6 Hz, 1H), 7.58 – 7.50 (m, 1H).

# Acetophenone (20)<sup>2</sup>



Obtained as a colourless liquid (49 mg, 82% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.02 – 7.95 (m, 2H), 7.58 (dd, J = 11.5, 4.1 Hz, 1H), 7.48 (t, J = 7.7 Hz, 2H), 2.63 (s, 3H).

# 1-(4-Methylphenyl)ethan-1-one (21)9



Obtained as a colourless liquid (58 mg, 87% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.91 – 7.84 (m, 2H), 7.30 – 7.24 (m, 2H), 2.59 (s, 3H), 2.42 (s, 3H).

# 1-(4-Methoxyphenyl)ethan-1-one (22)<sup>8</sup>



Obtained as a white solid (66 mg, 88% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.95 (d, *J* = 8.9 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 3.88 (s, 3H), 2.57 (s, 3H).

## 1-(4-Fluorophenyl)ethan-1-one (23)<sup>2</sup>



Obtained as a faint yellow liquid (57 mg, 83% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.99 (dd, J = 8.7, 5.5 Hz, 2H), 7.13 (t, J = 8.6 Hz, 2H), 2.59 (s, 3H).

# 1-(4-Chlorophenyl)ethan-1-one (24)<sup>2</sup>



Obtained as a colourless liquid (66 mg, 85% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.89 (dd, J = 8.6, 1.0 Hz, 2H), 7.42 (dd, J = 8.5, 1.2 Hz, 2H), 2.58 (s, 3H).

# 1-(4-Bromophenyl)ethan-1-one (25)<sup>2</sup>



Obtained as a white solid (80 mg, 80% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.84 (d, *J* = 8.6 Hz, 2H), 7.62 (d, *J* = 8.6 Hz, 2H), 2.60 (s, 3H).

## 1-(4-Nitrophenyl)ethan-1-one (26)<sup>2</sup>



Obtained as a faint yellow solid (32 mg, 39% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.30 (d, J = 6.9 Hz, 2H), 8.10 (d, J = 8.9 Hz, 2H), 2.67 (s, 3H).

#### 1-(3-Methylphenyl)ethan-1-one (27)<sup>9</sup>



Obtained as a colourless liquid (54 mg, 81% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.82 – 7.75 (m, 2H), 7.38 (dt, J = 15.0, 7.3 Hz, 2H), 2.61 (s, 3H), 2.43 (s, 3H).

#### 1-(3-Chlorophenyl)ethan-1-one (28)<sup>10</sup>



Obtained as a colourless liquid (59 mg, 76% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.93 (d, J = 1.4 Hz, 1H), 7.84 (dd, J = 7.8, 1.0 Hz, 1H), 7.57 – 7.52 (m, 1H), 7.45 – 7.39 (m, 1H), 2.61 (s, 3H).

#### 1-(3-(Trifluoromethyl)phenyl)ethan-1-one (29)<sup>2</sup>



Obtained as a colourless liquid (61 mg, 65% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.15 (s, 1H), 8.10 – 8.05 (m, 1H), 7.75 (d, J = 5.4 Hz, 1H), 7.60 – 7.51 (m, 1H), 2.59 (s, 3H).

# 1-(2-Methylphenyl)ethan-1-one (30)<sup>10</sup>



Obtained as a colourless liquid (49 mg, 73% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.72 (d, J = 7.7 Hz, 1H), 7.40 (t, J = 7.5 Hz, 1H), 7.28 (dd, J = 13.9, 7.5 Hz, 2H), 2.60 (s, 3H), 2.56 (s, 3H).

## 1-(2-Chlorophenyl)ethan-1-one (31)<sup>10</sup>



Obtained as a colourless liquid (58 mg, 75% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.55 (dd, J = 7.6, 1.6 Hz, 1H), 7.40 (dtd, J = 9.7, 8.0, 1.6 Hz, 2H), 7.35 – 7.30 (m, 1H), 2.65 (s, 3H).

## Propiophenone (32)<sup>2</sup>



Obtained as a colourless liquid (57 mg, 85% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.98 (dd, J = 8.3, 1.1 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 3.02 (q, J = 7.2 Hz, 2H), 1.25 (t, J = 7.3 Hz, 3H).

## 1-Phenylbutan-1-one (33)<sup>8</sup>



Obtained as a colourless liquid (58 mg, 78% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.98 (dd, J = 8.2, 1.0 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 2.97 (t, J = 7.3 Hz, 2H), 1.85 – 1.73 (m, 2H), 1.03 (t, J = 7.4 Hz, 3H).

## 3,4-Dihydronaphthalen-1(2H)-one (34)<sup>11</sup>



Obtained as a colourless liquid (45 mg, 62% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.05 (d, J = 7.8 Hz, 1H), 7.48 (t, J = 7.4 Hz, 1H), 7.32 (t, J = 7.5 Hz, 1H), 7.27 (d, J = 7.6 Hz, 1H), 2.98 (t, J = 6.1 Hz, 2H), 2.70 – 2.65 (m, 2H), 2.18 – 2.12 (m, 2H).

# 1-(Naphthalen-2-yl)ethan-1-one (35)<sup>8</sup>



Obtained as a white solid (58 mg, 68% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  8.69 (s, 1H), 8.15 (d, J = 8.0 Hz, 1H), 8.01 (d, J = 8.1 Hz, 3H), 7.66 (dtd, J = 16.2, 6.9, 1.3 Hz, 2H), 2.73 (s, 3H).

# 1-(Furan-2-yl)ethan-1-one (36)8



Obtained as a colourless liquid (29 mg, 53% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.61 – 7.57 (m, 1H), 7.18 (d, J = 3.6 Hz, 1H), 6.55 – 6.52 (m, 1H), 2.48 (s, 3H).

# 1-(Pyridin-2-yl)ethan-1-one (37)9



Obtained as a faint yellow liquid (34 mg, 56% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.72 – 8.66 (m, 1H), 8.04 (dd, J = 7.9, 1.0 Hz, 1H), 7.84 (dt, J = 9.4, 4.7 Hz, 1H), 7.50 – 7.43 (m, 1H), 2.73 (s, 3H).

## 1-(Pyridin-4-yl)ethan-1-one (38)<sup>10</sup>



Obtained as a faint yellow liquid (36 mg, 60% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.82 – 8.74 (m, 2H), 7.70 (dd, J = 4.5, 1.6 Hz, 2H), 2.60 (s, 3H).

# 1-(Thiophen-2-yl)ethan-1-one (39)<sup>10</sup>



Obtained as a faint yellow liquid (40 mg, 64% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.70 (d, J = 3.7 Hz, 1H), 7.63 (d, J = 4.9 Hz, 1H), 7.16 – 7.10 (m, 1H), 2.56 (s, 3H).

# Benzophenone (40)<sup>2</sup>



Obtained as a yellow liquid (76 mg, 84% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.83 – 7.78 (m, 4H), 7.59 (t, J = 7.4 Hz, 2H), 7.48 (t, J = 7.7 Hz, 4H).

# (4-Methoxyphenyl)(phenyl)methanone (41)<sup>12</sup>



Obtained as a yellow liquid (91 mg, 86% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.82 (d, J = 8.9 Hz, 2H), 7.75 (d, J = 7.0 Hz, 2H), 7.57 – 7.53 (m, 1H), 7.46 (t, J = 7.6 Hz, 2H), 6.96 (d, J = 8.9 Hz, 2H), 3.87 (s, 3H).

# (4-Chlorophenyl)(phenyl)methanone (42)<sup>13</sup>



Obtained as a white solid (87 mg, 80% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (t, J = 8.4 Hz, 4H), 7.60 (t, J = 7.4 Hz, 1H), 7.53 – 7.41 (m, 4H).

# (4-Iodophenyl)(phenyl)methanone (43)<sup>14</sup>



Obtained as a white solid (112 mg, 73% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (d, J = 8.5 Hz, 2H), 7.68 (dd, J = 8.2, 1.2 Hz, 2H), 7.51 (t, J = 7.4 Hz, 1H), 7.44 – 7.37 (m, 4H).

# Phenyl(4-(trifluoromethyl)phenyl)methanone (44)<sup>13</sup>



Obtained as a yellow liquid (92.5 mg, 74% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.89 (d, J = 8.0 Hz, 2H), 7.81 (dd, J = 8.3, 1.2 Hz, 2H), 7.75 (d, J = 8.1 Hz, 2H), 7.63 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.7 Hz, 2H).

# (3,4-Dichlorophenyl)(phenyl)methanone (45)<sup>15</sup>



Obtained as a white solid (88 mg, 70% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.81 (d, J = 1.9 Hz, 1H), 7.71 – 7.67 (m, 2H), 7.54 (dt, J = 8.9, 4.6 Hz, 2H), 7.48 (d, J = 8.3 Hz, 1H), 7.42 (t, J = 7.8 Hz, 2H).

## (4-Methoxyphenyl)(p-tolyl)methanone (46)<sup>16</sup>



Obtained as a white solid (95 mg, 84% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.81 (d, J = 8.7 Hz, 2H), 7.67 (d, J = 8.1 Hz, 2H), 7.27 (d, J = 8.2 Hz, 2H), 6.95 (d, J = 8.8 Hz, 2H), 3.87 (s, 3H), 2.43 (s, 3H).

# (4-Fluorophenyl)(p-tolyl)methanone (47)<sup>16</sup>



Obtained as a white solid (87 mg, 81% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.82 (dd, J = 8.8, 5.5 Hz, 2H), 7.68 (d, J = 8.1 Hz, 2H), 7.28 (d, J = 7.9 Hz, 2H), 7.14 (t, J = 8.7 Hz, 2H), 2.44 (s, 3H).

## (4-Chlorophenyl)(p-tolyl)methanone (48)<sup>13</sup>



Obtained as a white solid (90 mg, 78% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.72 – 7.65 (m, 3H), 7.65 – 7.60 (m, 3H), 7.29 (d, J = 7.9 Hz, 2H), 2.44 (s, 3H).

## (4-Bromophenyl)(p-tolyl)methanone (49)<sup>13</sup>



Obtained as a white solid (99 mg, 72% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.73 (d, J = 8.6 Hz, 2H), 7.69 (d, J = 8.1 Hz, 2H), 7.44 (d, J = 8.6 Hz, 2H), 7.28 (d, J = 7.9 Hz, 2H), 2.44 (s, 3H).

# (4-Iodophenyl)(p-tolyl)methanone (50)<sup>16</sup>



Obtained as a white solid (111 mg, 69% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.84 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.0 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 7.28 (d, J = 7.9 Hz, 2H), 2.44 (s, 3H).

## Thiophen-2-yl(p-tolyl)methanone (51)<sup>13</sup>



Obtained as a yellow liquid (73 mg, 72% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.79 (d, J = 8.1 Hz, 2H), 7.70 (dd, J = 4.9, 1.0 Hz, 1H), 7.65 (dd, J = 3.8, 1.0 Hz, 1H), 7.30 (d, J = 8.0 Hz, 2H), 7.16 (dd, J = 4.9, 3.8 Hz, 1H), 2.45 (s, 3H).

#### 4-Methoxybenzoic acid (52)<sup>7</sup>



Obtained as a white solid (65 mg, 85% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  12.62 (s, 1H), 7.90 (d, J = 8.9 Hz, 2H), 7.02 (d, J = 8.9 Hz, 2H), 3.83 (s, 3H).

#### Benzoic acid (53)<sup>7</sup>



Obtained as a white solid (49 mg, 80% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  13.01 (s, 1H), 8.02 (dd, J = 8.3, 1.3 Hz, 2H), 7.64 (t, J = 7.4 Hz, 1H), 7.53 (t, J = 7.7 Hz, 2H).

## [1,1'-Biphenyl]-4-carboxylic acid (54)<sup>7</sup>



Obtained as a white solid (75 mg, 76% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  13.00 (s, 1H), 8.03 (d, J = 8.4 Hz, 2H), 7.80 (d, J = 8.4 Hz, 2H), 7.77 – 7.72 (m, 2H), 7.51 (t, J = 7.6 Hz, 2H), 7.43 (t, J = 7.3 Hz, 1H).

#### 4-Chlorobenzoic acid (55)<sup>7</sup>



Obtained as a white solid (56 mg, 72% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  13.21 (s, 1H), 7.98 (d, J = 8.5 Hz, 2H), 7.58 (d, J = 8.5 Hz, 2H).

# 3-Methylbenzoic acid (56)<sup>7</sup>



Obtained as a white solid (56 mg, 82% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.93 (d, J = 8.7 Hz, 2H), 7.42 (d, J = 7.5 Hz, 1H), 7.36 (t, J = 7.5 Hz, 1H), 2.42 (s, 3H).

## 3-Fluorobenzoic acid (57)<sup>8</sup>



Obtained as a white solid (52 mg, 74% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  13.32 (s, 1H), 7.83 (d, J = 7.7 Hz, 1H), 7.69 (ddd, J = 9.6, 2.4, 1.4 Hz, 1H), 7.58 (td, J = 8.0, 5.8 Hz, 1H), 7.50 (ddd, J = 8.3, 2.6, 1.9 Hz, 1H).

# 3-Chlorobenzoic acid (58)<sup>7</sup>



Obtained as a white solid (62 mg, 79% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  13.35 (s, 1H), 7.93 – 7.90 (m, 2H), 7.73 – 7.68 (m, 1H), 7.56 (t, J = 8.1 Hz, 1H).

## 3-Bromobenzoic acid (59)<sup>8</sup>



Obtained as a white solid (73 mg, 73% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  13.33 (s, 1H), 8.05 (t, J = 1.7 Hz, 1H), 7.96 - 7.93 (m, 1H), 7.84 (ddd, J = 8.0, 2.1, 1.0 Hz, 1H), 7.49 (t, J = 7.9 Hz, 1H).

# 2-Fluorobenzoic acid (60)<sup>8</sup>



Obtained as a white solid (49 mg, 70% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.05 (td, J = 7.6, 1.8 Hz, 1H), 7.64 – 7.54 (m, 1H), 7.26 – 7.23 (m, 1H), 7.18 (ddd, J = 10.8, 8.4, 0.8 Hz, 1H).

# 2-Chlorobenzoic acid (61)<sup>7</sup>



Obtained as a white solid (56 mg, 72% yield); <sup>1</sup>H NMR (500 MHz, DMSO) δ 13.40 (s, 1H), 7.85 – 7.78 (m, 1H), 7.57 – 7.52 (m, 2H), 7.44 (ddd, *J* = 7.9, 6.2, 2.4 Hz, 1H).

#### 2-Bromobenzoic acid (62)<sup>8</sup>



Obtained as a white solid (65 mg, 65% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.01 (dd, *J* = 7.4, 2.1 Hz, 1H), 7.72 (dd, *J* = 7.1, 2.1 Hz, 1H), 7.43 – 7.36 (m, 2H).

#### 2-Naphthoic acid (63)<sup>7</sup>



Obtained as a white solid (49 mg, 57% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  13.20 (s, 1H), 8.72 (s, 1H), 8.17 (d, J = 7.9 Hz, 1H), 8.12 – 8.02 (m, 3H), 7.67 (dt, J = 14.6, 6.9 Hz, 2H).

#### Picolinic acid (64)<sup>8</sup>



Obtained as a white solid (18 mg, 29% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  13.13 (s, 1H), 8.72 (ddd, J = 4.7, 1.5, 0.8 Hz, 1H), 8.10 – 8.04 (m, 1H), 8.00 (td, J = 7.7, 1.7 Hz, 1H), 7.64 (ddd, J = 7.5, 4.7, 1.2 Hz, 1H).

#### Isopropyl-2-(4-(4-chlorobenzoyl)phenoxy)-2-methylpropanoate (65)<sup>17</sup>



Obtained as a white solid (135 mg, 75% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.73 (d, *J* = 8.9 Hz, 2H), 7.70 (d, *J* = 8.6 Hz, 2H), 7.45 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.9 Hz, 2H), 5.13 – 5.05 (m, 1H), 1.66 (s, 6H), 1.21 (d, *J* = 6.3 Hz, 6H).

#### 3-Oxo-3-phenylpropyl-2-(4-isobutylphenyl)propanoate (66)



Obtained as a yellow liquid (123 mg, 73% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.90 (d, J = 7.5 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.7 Hz, 2H), 7.15 (d, J = 8.0 Hz, 2H), 7.04 (d, J = 8.0 Hz, 2H), 4.55 – 4.48 (m, 2H), 3.66 (q, J = 7.2 Hz, 1H), 3.26 – 3.21 (m, 2H), 2.42 (d, J = 7.2 Hz, 2H), 1.83 (dd, J = 13.5, 6.7 Hz, 1H), 1.46 (d, J = 7.2 Hz, 3H), 0.88 (d, J = 6.6 Hz, 6H); <sup>13</sup>C NMR (126 MHz,

CDCl<sub>3</sub>)  $\delta$  197.1, 174.7, 140.5, 137.6, 136.7, 133.4, 129.3, 128.7, 128.1, 127.1, 60.1, 45.1, 45.0, 37.3, 30.2, 22.4, 18.5; HRMS (ESI+): Calculated for C<sub>22</sub>H<sub>26</sub>O<sub>3</sub>Na: [M + Na]<sup>+</sup> 361.1774, Found 361.1777.

# 2-Phenyl-1*H*-benzo[*d*]imidazole (67)<sup>18</sup>



Obtained as a white solid (78 mg, 80% yield); <sup>1</sup>H NMR (500 MHz, DMSO)  $\delta$  12.93 (s, 1H), 8.20 (d, J = 7.2 Hz, 2H), 7.62 (dd, J = 5.4, 3.2 Hz, 2H), 7.56 (t, J = 7.5 Hz, 2H), 7.50 (t, J = 7.3 Hz, 1H), 7.22 (dd, J = 6.0, 3.1 Hz, 2H).

## Chalcone (68)<sup>19</sup>



Obtained as a faint yellow solid (88 mg, 85% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.02 (d, J = 7.1 Hz, 2H), 7.81 (d, J = 15.7 Hz, 1H), 7.64 (dd, J = 6.7, 2.8 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.51 (dd, J = 18.5, 11.5 Hz, 3H), 7.42 (dd, J = 5.0, 1.8 Hz, 3H).

# 1-Phenylethan-1-one oxime (69)<sup>20</sup>



Obtained as a white solid (61 mg, 90% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  9.49 (s, 1H), 7.62 (dd, J = 6.6, 2.7 Hz, 2H), 7.38 (dd, J = 4.8, 1.5 Hz, 3H), 2.31 (s, 3H).

# N-Phenylacetamide (70)<sup>21</sup>



Obtained as a white solid (62 mg, 92% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.19 (s, 1H), 7.51 (d, J = 7.7 Hz, 2H), 7.27 (t, J = 7.9 Hz, 2H), 7.08 (t, J = 7.4 Hz, 1H), 2.13 (s, 3H).

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# 4. Copies of <sup>1</sup>H and <sup>13</sup>C NMR Spectra





# <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



# <sup>1</sup>H NMR Spectrum of Compound 3





<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



# <sup>1</sup>H NMR Spectrum of Compound 4









# <sup>1</sup>H NMR Spectrum of Compound 5





<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



<sup>1</sup>H NMR Spectrum of Compound 6









<sup>1</sup>H NMR Spectrum of Compound 7



<sup>1</sup>H NMR Spectrum of Compound 8



<sup>1</sup>H NMR Spectrum of Compound 10









<sup>1</sup>H NMR Spectrum of Compound 11



<sup>1</sup>H NMR Spectrum of Compound 12







<sup>1</sup>H NMR Spectrum of Compound 13













<sup>1</sup>H NMR Spectrum of Compound 15



<sup>1</sup>H NMR Spectrum of Compound 16









<sup>1</sup>H NMR Spectrum of Compound 17



<sup>1</sup>H NMR Spectrum of Compound 18

#### -10.09















<sup>1</sup>H NMR Spectrum of Compound 21



<sup>1</sup>H NMR Spectrum of Compound 22





3.08-

2.0

1.0

0.0

3.0

4.0



7.0 6.0 5.0 Chemical Shift (ppm)

2.06<del>-</del> 2.00-=

8.0

12.0

11.0

10.0

9.0



<sup>1</sup>H NMR Spectrum of Compound 25



<sup>1</sup>H NMR Spectrum of Compound 26



<sup>1</sup>H NMR Spectrum of Compound 27



<sup>1</sup>H NMR Spectrum of Compound 28









<sup>1</sup>H NMR Spectrum of Compound 30



















<sup>1</sup>H NMR Spectrum of Compound 34







<sup>1</sup>H NMR Spectrum of Compound 36







<sup>1</sup>H NMR Spectrum of Compound 38



<sup>1</sup>H NMR Spectrum of Compound 39



<sup>1</sup>H NMR Spectrum of Compound 40



<sup>1</sup>H NMR Spectrum of Compound 41



<sup>1</sup>H NMR Spectrum of Compound 42

7.77 7.75 7.75 7.75 7.69 7.60 7.49 7.40 7.40 7.40 7.40 7.40 7.38



<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



# <sup>1</sup>H NMR Spectrum of Compound 43





<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



<sup>1</sup>H NMR Spectrum of Compound 44

# 



# <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



# <sup>1</sup>H NMR Spectrum of Compound 45



-2.43



<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



<sup>1</sup>H NMR Spectrum of Compound 46





<sup>1</sup>H NMR Spectrum of Compound 48



<sup>1</sup>H NMR Spectrum of Compound 50

0.0

10.0





# <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



# <sup>1</sup>H NMR Spectrum of Compound 51















<sup>1</sup>H NMR Spectrum of Compound 58



<sup>1</sup>H NMR Spectrum of Compound 60





<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



<sup>1</sup>H NMR Spectrum of Compound 62



<sup>1</sup>H NMR Spectrum of Compound 64



<sup>1</sup>H NMR Spectrum of Compound 65

#### 0.0000 0.0000 0.0000 0.000 0.000 0.000 0.000 0.000 0.000 0.000



<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)



<sup>1</sup>H NMR Spectrum of Compound 66





<sup>1</sup>H NMR Spectrum of Compound 67















