# **Supporting Information**

# Water as an Oxygen Source in I<sub>2</sub>-Mediated Construction of Highly Functionalized Maleimide-fused Phenols

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## **1.** General information

The reaction via general procedure was carried out under an atmosphere of air unless otherwise noted. Column chromatography was performed using silica gel (200-300 mesh) or thin layer chromatography was performed using silica gel (GF254). <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on Bruker-AV (400 and 100 MHz, respectively) instrument using CDCl<sub>3</sub> as solvents. Mass spectra were measured on Agilent 5975 GC-MS instrument (EI). High-resolution mass spectra (ESI) were obtained with the Thermo Scientific LTQ Orbitrap XL mass spectrometer. The structures of known compounds were further corroborated by comparing their <sup>1</sup>H NMR, <sup>13</sup>C NMR data and HRMS data with those in literatures. Melting points were measured with a YUHUA X-5 melting point instrument and were uncorrected. All reagents were directly used without purification as received from commercial supplier.

### 2. Optimization of reaction conditions<sup>a</sup>

	) N— + <sup>2</sup> P	additive	acid	N-
	)	solvent, 140	$P^{*}C, 24 \text{ n} \text{ Ph}^{-1}$	
1a		2a	C	3a
			~ .	
Entry	Additive	Acid	Solvent	Yield $(\%)^{b}$
1	$I_2$	$BF_3 \cdot Et_2O$	toluene	60
2	IBr	$BF_3 \cdot Et_2O$	toluene	45
3	$I_2O_5$	$BF_3 \cdot Et_2O$	toluene	16
4	NIS	$BF_3 \cdot Et_2O$	toluene	24
5	NH <sub>4</sub> I	$BF_3 \cdot Et_2O$	toluene	trace
6	$I_2$	AcOH	toluene	trace
7	$I_2$	TsOH·H <sub>2</sub> O	toluene	40
8	$I_2$	FeCl <sub>3</sub>	toluene	30
9	$I_2$	Mg(OTf) <sub>2</sub>	toluene	23
10	$I_2$	$BF_3 \cdot Et_2O$	<i>p</i> -xylene	66
11	$I_2$	$BF_3 \cdot Et_2O$	DMSO	trace
12	$I_2$	$BF_3 \cdot Et_2O$	<i>i</i> -PrOH	trace
13	$I_2$	$BF_3 \cdot Et_2O$	PhCl	62
14	$I_2$	$BF_3 \cdot Et_2O$	p-xylene/PhCl	70
15 <sup>c</sup>	$I_2$	$BF_3 \cdot Et_2O$	<i>p</i> -xylene/PhCl	63
$16^d$	$I_2$	$BF_3 \cdot Et_2O$	<i>p</i> -xylene/PhCl	72
<b>17</b> <sup>e</sup>	$\mathbf{I}_2$	BF <sub>3</sub> ·Et <sub>2</sub> O	<i>p</i> -xylene/PhCl	80

18 <sup>f</sup>	$I_2$	$BF_3 \cdot Et_2O$	p-xylene/PhCl	trace
19 <sup>g</sup>	$I_2$	$BF_3 \cdot Et_2O$	p-xylene/PhCl	77
$20^{h}$	$I_2$	$BF_3 \cdot Et_2O$	p-xylene/PhCl	75
$21^{i}$	$I_2$	$BF_3 \cdot Et_2O$	p-xylene/PhCl	73
$22^j$	$I_2$	$BF_3 \cdot Et_2O$	p-xylene/PhCl	78
23	-	$BF_3 \cdot Et_2O$	toluene	trace
24	$I_2$	-	toluene	13

<sup>*a*</sup> Reaction conditions: **1a** (0.2 mmol), **2a** (0.5 mmol, 2.5 equiv), additive (0.4 mmol, 2.0 equiv), acid (0.2 mmol. 1.0 equiv), solvent (1.0 mL) was reacted under air at 140 °C for 24 h. <sup>*b*</sup> Isolated yield. <sup>*c*</sup> Under Ar. <sup>*d*</sup> Under O<sub>2</sub>. <sup>*e*</sup> H<sub>2</sub>O (0.4 mmol, 2.0 equiv). <sup>*f*</sup> 4 Å molecular sieves (100 mg). <sup>*g*</sup> 150 °C. <sup>*h*</sup> 130 °C. <sup>*i*</sup> H<sub>2</sub>O (0.2 mmol, 1.0 equiv). <sup>*j*</sup> H<sub>2</sub>O (0.6 mmol, 3.0 equiv).

#### **3. Experiment section**

General procedure for synthesis of various maleimides. <sup>[1][2]</sup>



**a)** Maleic anhydride (2.0 equiv) and primary amine (1.0 equiv) were stirred in acetic acid (1.5 ml per mmol of amine) until maleic anhydride dissolved completely. The reaction mixture was refluxed for 8 h at 130 °C (oil bath temperature). After completion of the reaction, the reaction mixture was then allowed to cool down to room temperature and the whole reaction mixture was transferred to a 500 mL beaker. **b)** Saturated sodium bicarbonate aqueous solution was added to the beaker containing reaction mixture until effervescence stop. The aqueous mixture was extracted with ethyl acetate (3x20 mL). **c)** The organic layer was further washed with 1 mol/L HCl (2x50 mL) and brine solution (30 mL) respectively. The excess solvent was removed under reduced pressure and the residue was purified by flash column chromatography using ethyl acetate/petroleum ether to get highly pure maleimide. All maleimides are known compounds.

#### General procedure for 4-hydroxy-isoindoline-1,3-dione derivatives



To a Schlenk tube were added **1** (0.2 mmol, 22.2 mg), **2** (0.5 mmol, 2.5 equiv),  $I_2$  (0.4 mmol, 2.0 equiv),  $BF_3 \cdot Et_2O$  (0.2 mmol. 1.0 equiv),  $H_2O$  (0.4 mmol, 2.0 equiv), *p*-xylene/PhCl (1:1) (1.0 mL). The mixture was stirred at 140 °C in air atmosphere for 24 h until complete consumption of starting material as monitored by TLC and GC-MS analysis. The crude reaction mixture was diluted by ethyl acetate and washed by saturated sodium thiosulfate pentahydrate aqueous solution. The organic layer was dried with MgSO<sub>4</sub>, filtered and the volatiles was removed under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 15:1 to 5:1) to afford the desired products **3**.

Substrates with low reactivity:



#### 4. Gram-scale reaction and further synthetic elaboration of products



To a Schlenk tube were added **1ab** (5.0 mmol, 865.0 mg), **2a** (12.5 mmol, 2.5 equiv),  $I_2$  (10.0 mmol, 2.0 equiv),  $BF_3 \cdot Et_2O$  (5.0 mmol. 1.0 equiv),  $H_2O$  (10.0 mmol, 2.0 equiv), *p*-xylene/PhCl (1:1) (15 mL). The mixture was stirred at 140 °C in air atmosphere for 24 h until complete consumption of starting material as monitored by TLC and GC-MS analysis. The crude reaction mixture was diluted by ethyl acetate and washed by saturated sodium thiosulfate pentahydrate aqueous solution. The organic layer was dried with MgSO<sub>4</sub>, filtered and the volatiles was removed under reduced pressure. The residue was purified by silica gel flash column chromatography

(hexane/ethyl acetate = 15:1 to 10:1) to afford the desired products **3ab** in 52% yield.

#### Procedure for the reduction reaction of 3ab



DIBAL-H (1 M solution in DCM, 0.45 mL, 0.45 mmol, 4.5 equiv) was slowly added to a solution of **3ab** (0.1 mmol, 39.1 mg) in DCM (1 mL) at 0 °C. After being stirred for 12 h, the mixture was diluted by ethyl acetate and washed by saturated NH<sub>4</sub>Cl aqueous solution. The organic layer was dried with MgSO<sub>4</sub>, and filtered, concentrated under the vacuum and purified through flash column chromatography to give the desired product **4** in 60% yield.

#### Procedure for the transamidation reaction of 3ab<sup>[3]</sup>



To a Schlenk tube were added **3ab** (0.1 mmol, 39.1 mg), DMAP (1.1 mmol, 1.1 equiv), 4-methoxy phenethylamine (1 mL) at room temperature. The mixture was heated at 110 °C, and then stirred for 12 h. After cooling to ambient temperature, silica gel column chromatography of the crude mixture provided the transamidation product **5** in 71% yield.

#### Procedure for the Williamson reaction of 3ab and 3w



To a Schlenk tube were added **3ab** (0.1 mmol, 39.1 mg), benzyl bromide (0.15 mmol, 1.5 equiv),  $K_2CO_3$  (0.25 mmol, 2.5 equiv), acetone (1.0 mL). The mixture was stirred at room temperature for

3 h until complete consumption of starting material as monitored by TLC, then the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 15:1 to 10:1) to give **6** in 53% yield.



То 3w Schlenk tube were added (0.1 mmol, 37.1 4-(bromomethyl) a mg), benzeneboronicacidpinacolester (0.15 mmol, 1.5 equiv), K<sub>2</sub>CO<sub>3</sub> (0.25 mmol, 2.5 equiv), acetone (1.0 mL). The mixture was stirred at room temperature for 3 h until complete consumption of starting material as monitored by TLC, then the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 to 10:1) to give 7 in 76% yield.

## Procedure for the triflation of 3ab



Trifluoromathanesulfonyl chloride (0.2 mmol, 2.0 equiv) was slowly added to a solution of **3ab** (0.1 mmol, 39.1 mg),  $Et_3N$  (0.2 mmol, 2.0 equiv) in DCM (1 mL) at 0 °C. After being stirred for 2 h, then the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 to 10:1) to give **8** in 76% yield.

### Procedure for Suzuki-Miyaura coupling reaction of 8



To a Schlenk tube were added **8** (0.1 mmol, 52.3 mg), 4-Methoxyphenylboronic acid (0.13 mmol, 1.3 equiv), Pd(PPh<sub>3</sub>)<sub>4</sub> (0.005 mmol, 5 mol%), K<sub>2</sub>CO<sub>3</sub> (0.4 ml, 2 M), THF (0.6 mL). Then the reaction vessel was purged with argon for three times and stirred at 80 °C for 16 h. After cooling to room temperature, the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 15:1 to 10:1) to give **9** in 70% yield.

**Procedure for Buchwald-Hartwig reaction of 8** 



To a Schlenk tube were added **8** (0.1 mmol, 52.3 mg), 4-methoxyaniline (0.15 mmol, 1.5 equiv),  $Pd_2(dba)_3$  (0.002 mmol, 2.0 mol%), DPPF (0.006 mmol, 6.0 mol%), *t*-BuONa (0.15 mmol, 1.5 equiv) and toluene (1.0 mL). Then the reaction vessel was purged with argon for three times and stirred at 80 °C for 16 h. After cooling to room temperature, the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 15:1 to 5:1) to give **10** in 56% yield.

# 5. Mechanistic experiments

# Monitoring the reaction



Product	(A) Yield	( <b>3a</b> ) Yield	( <b>3a'</b> ) Yield
Time	(%) ª	(%) <sup>a</sup>	(%) <sup>a</sup>
3 h	12	32	2
6 h	19	41	3
9 h	7	50	3
12 h	5	63	5
16 h	5	76	8
24 h	6	78	9

<sup>a</sup> Isolated yield based on **1a**.



#### <sup>18</sup>O-labeling experiments



To a schlenk tube were added **1a** (0.2 mmol), **2a** (0.5 mmol, 2.5 eqiuv),  $I_2$  (0.4 mmol, 2.0 equiv), BF<sub>3</sub>•Et<sub>2</sub>O (0.2 mmol. 1.0 equiv),  $H_2^{18}O$  (0.4 mmol, 2.0 equiv), *p*-xylene/PhCl (1:1) (1.0 mL). The mixture was stirred at 140 °C in air atmosphere for 24 h until complete consumption of starting material as monitored by TLC analysis. The crude reaction mixture was diluted using 10 mL EtOAc, dried over sodium sulfate, filtered and the volatiles were removed under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 15:1 to 5:1) to afford the products **3a** in 78% yield. Two products were detected on the base of GC-MS analysis: <sup>18</sup>O-**3a** and **3a** with the ratio 1:0.30.



To a schlenk tube were added **1a** (0.2 mmol), **2a** (0.5 mmol, 2.5 equiv),  $I_2$  (0.4 mmol, 2.0 equiv),  $BF_3 \cdot Et_2O$  (0.2 mmol. 1.0 equiv),  $H_2O$  (0.4 mmol, 2.0 equiv), *p*-xylene/PhCl (1:1) (1.0 mL). The mixture was stirred at 140 °C in <sup>18</sup>O<sub>2</sub> atmosphere for 24 h until complete consumption of starting material as monitored by TLC analysis. The crude reaction mixture was diluted using 10 mL EtOAc, dried over sodium sulfate, filtered and the volatiles were removed under reduced pressure.

The residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 15:1 to 5:1) to afford the products **3a** in 76% yield. Two products were detected on the base of GC-MS analysis: <sup>18</sup>O-**3a** and **3a** with the ratio 0.08:1.



To a schlenk tube were added **1a** (0.2 mmol), **A** (0.3 mmol, 1.5 eqiuv),  $I_2$  (0.4 mmol, 2.0 equiv), BF<sub>3</sub>•Et<sub>2</sub>O (0.2 mmol. 1.0 equiv),  $H_2^{18}O$  (0.4 mmol, 2.0 equiv), *p*-xylene/PhCl (1:1) (1.0 mL). The mixture was stirred at 140 °C in air atmosphere for 24 h until complete consumption of starting material as monitored by TLC analysis. The crude reaction mixture was diluted using 10 mL EtOAc, dried over sodium sulfate, filtered and the volatiles were removed under reduced pressure. The residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 15:1 to 5:1) to afford the products **3a** in 68% yield. Two products were detected on the base of GC-MS analysis: <sup>18</sup>O-**3a** and **3a** with the ratio 1:0.55.



## **Control experiments**



# 6. Characterization data of products

4-hydroxy-2-methyl-5,7-diphenylisoindoline-1,3-dione (3a)



Yield: 52.6 mg, 80 %; yellow solid; mp 141-142 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.51 (s, 1H), 7.67 (d, *J* = 6.8 Hz, 2H), 7.64 (s, 1H), 7.59 (dd, *J* = 7.9, 1.7 Hz, 2H), 7.52 – 7.40 (m, 6H), 3.15 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.0, 167.3, 151.8, 138.9, 136.0, 135.9, 135.1, 133.9, 129.3, 129.2, 128.63, 128.56, 128.5, 128.2, 125.6, 115.7, 23.8. HRMS (ESI) m/z: calcd. for C<sub>21</sub>H<sub>15</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 352.0944, found 352.0949. IR (neat) v (cm<sup>-1</sup>): 3350, 3048, 2920, 2850,





Yield: 57.8 mg, 81 %; yellow solid; mp 121-122 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.48 (s, 1H), 7.61 (s, 1H), 7.57 (d, *J* = 8.1 Hz, 2H), 7.48 (d, *J* = 8.1 Hz, 2H), 7.28 (t, *J* = 6.4 Hz, 4H), 3.14 (s, 3H), 2.42 (s, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 167.4, 151.7, 138.64, 138.61, 138.5, 136.1, 134.0, 133.1, 132.3, 129.3, 129.1, 129.0, 128.9, 125.1, 115.6, 23.8, 21.36, 21.35. HRMS (ESI) m/z: calcd. for C<sub>23</sub>H<sub>19</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 380.1257, found 380.1256. IR (neat) v (cm<sup>-1</sup>): 3372, 3017, 2995, 2943, 2859, 1757, 1684, 1442, 1375, 1238, 1072, 1000, 823, 730, 591, 577.

#### 5,7-bis(4-(tert-butyl)phenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3c)



Yield: 50.3 mg, 57 %; yellow solid; mp 174-175 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.53 (s, 1H), 7.66 (s, 1H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.56 – 7.47 (m, 6H), 3.15 (s, 3H), 1.38 (s, 9H), 1.38 (s, 9H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 167.5, 151.8, 151.7, 151.5, 138.8, 136.0, 133.9, 133.0, 132.3, 129.0, 128.9, 125.5, 125.2, 125.1, 115.6, 34.8, 34.7, 31.4, 31.3, 23.7. HRMS (ESI) m/z: calcd. for C<sub>29</sub>H<sub>31</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 464.2196, found 464.2183. IR (neat) v (cm<sup>-1</sup>): 3364, 2956, 2868, 1724, 1697, 1440, 1379, 1246, 1177, 1119, 1077, 1005, 873, 835, 763, 572. 4-hydroxy-5,7-bis(4-isobutylphenyl)-2-methylisoindoline-1,3-dione (3d)



Yield: 52.0 mg, 59 %; yellow solid; mp 50-51 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.52 (s, 1H), 7.64 (s, 1H), 7.60 (d, *J* = 8.1 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 7.24 (t, *J* = 6.4 Hz, 4H), 3.15 (s, 3H), 2.54 (d, *J* = 7.2 Hz, 4H), 1.96 – 1.88 (m, 2H), 0.96 (s, 6H), 0.94 (s, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 167.4, 151.7, 142.4, 142.2, 138.8, 136.1, 134.0, 133.3, 132.5, 129.3, 129.0, 128.9, 128.8, 125.0, 115.6, 45.3, 45.2, 30.3, 30.2, 23.7, 22.5, 22.4. HRMS (ESI) m/z: calcd. for C<sub>29</sub>H<sub>31</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 464.2196, found 464.2204. IR (neat) v (cm<sup>-1</sup>): 3374, 2953, 2921, 2866, 1758, 1691, 1486, 1439, 1382, 1244, 1172, 1087, 1002, 845, 797, 760, 624.

#### 4-hydroxy-5,7-bis(4-methoxyphenyl)-2-methylisoindoline-1,3-dione (3e)



Yield: 40.5 mg, 52 %; yellow solid; mp 165-1666 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.50 (s, 1H), 7.63 (d, *J* = 8.8 Hz, 2H), 7.57 (s, 1H), 7.53 (d, *J* = 8.7 Hz, 2H), 7.03 – 6.96 (m, 4H), 3.86 (s, 6H), 3.14 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 167.5, 159.9, 159.8, 151.5, 138.2, 135.7, 133.7, 130.5, 130.4, 128.3, 127.5, 124.5, 115.6, 114.0, 113.7, 55.4, 55.3, 23.7. HRMS (ESI) m/z: calcd. for C<sub>23</sub>H<sub>19</sub>NNaO<sub>5</sub><sup>+</sup> (M+Na)<sup>+</sup> 412.1155, found 412.1145. IR (neat) v (cm<sup>-1</sup>): 3370, 3001, 2922, 2984, 2832, 1758, 1703, 1607, 1515, 1484, 1435, 1376, 1240, 1181, 1085, 1003, 822, 760, 656, 622. 5,7-di([1,1'-biphenyl]-4-yl)-4-hydroxy-2-methylisoindoline-1,3-dione (3f)



Yield: 79.8 mg, 83 %; yellow solid; mp 122-123 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.60 (s, 1H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.75 – 7.63 (m, 11H), 7.48 (t, *J* = 7.3 Hz, 4H), 7.44 – 7.35 (m, 2H), 3.18 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.0, 167.4, 151.9, 141.5, 141.4, 140.6, 140.5, 138.7, 135.7, 134.8, 134.0, 133.5, 129.7, 129.6, 128.9, 128.8, 127.7, 127.6, 127.3, 127.2, 127.1, 127.0, 125.5, 115.8, 23.8. HRMS (ESI) m/z: calcd. for C<sub>33</sub>H<sub>23</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 504.1570, found 504.1576. IR (neat) v (cm<sup>-1</sup>): 3351, 3054, 3028, 2941, 1756, 1690, 1479, 1440, 1383, 1242, 1087, 1004, 837, 757, 733, 693, 575.

4-hydroxy-2-methyl-5,7-bis(4-(trifluoromethoxy)phenyl)isoindoline-1,3-dione (3g)



Yield: 46.7 mg, 47 %; yellow solid; mp 119-120 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.55 (s, 1H), 7.70 (d, *J* = 8.8 Hz, 2H), 7.62 (s, 1H), 7.59 (d, *J* = 6.4 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 4H), 3.16 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.7, 167.1, 151.9, 149.5, 138.5, 134.7, 134.2, 133.5, 132.3, 130.8, 130.7, 129.6, 126.0, 122.1 (q, *J* = 254.3 Hz), 121.8 (q, *J* = 257.9 Hz), 121.0, 120.6, 115.9, 23.9. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -57.69, -57.73. HRMS (ESI) m/z: calcd. for C<sub>23</sub>H<sub>13</sub>F<sub>6</sub>NNaO<sub>5</sub><sup>+</sup> (M+Na)<sup>+</sup> 520.0590, found 520.0589. IR (neat) v (cm<sup>-1</sup>): 3395, 3075, 2923, 2855, 1756, 1688, 1512, 1486, 1449, 1377, 1242, 1146, 1081, 1005, 924, 833, 760, 563.

#### 5,7-bis(4-fluorophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3h)



Yield: 58.4 mg, 80 %; yellow solid; mp 206-207 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.51 (s, 1H), 7.69 – 7.61 (m, 2H), 7.59 – 7.51 (m, 3H), 7.19 – 7.12 (m, 4H), 3.15 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.8, 167.2, 163.0 (d, J = 248.4 Hz), 162.9 (d, J = 248.9 Hz), 151.7, 138.5, 135.1, 132.8, 131.8, 131.7, 131.0 (d, J = 8.4 Hz), 130.9 (d, J = 8.3 Hz), 125.6, 115.6 (d, J = 21.8 Hz), 115.3 (d, J = 21.8 Hz), 23.8. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -112.58, -113.11. HRMS (ESI) m/z: calcd. for C<sub>21</sub>H<sub>13</sub>F<sub>2</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 388.0756, found 388.0759. IR (neat) v (cm<sup>-1</sup>): 3355, 3044, 2992, 2952, 1759, 1698, 1602, 1512, 1486, 1440, 1380, 1302, 1228, 1156, 1082, 1003, 828, 759, 672.

### 5,7-bis(4-chlorophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3i)



Yield: 61.9 mg, 78 %; yellow solid; mp 199-200 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.51 (s, 1H), 7.60 (t, *J* = 7.9 Hz, 4H), 7.53 (d, *J* = 8.8 Hz, 3H), 7.44 (d, *J* = 8.4 Hz, 2H), 3.15 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.7, 167.1, 151.8, 138.2, 135.0, 134.6, 133.8, 132.6, 131.8, 131.5, 130.8, 130.7, 125.9, 123.2, 123.1, 115.9, 23.9. HRMS (ESI) m/z: calcd. for C<sub>21</sub>H<sub>12</sub>Cl<sub>2</sub>NO<sub>3</sub><sup>-</sup> (M-H)<sup>-</sup> 396.0200, found 396.0200. IR (neat) v (cm<sup>-1</sup>): 3368, 3054, 2987, 2918, 1757, 1703, 1586, 1475, 1442, 1375, 1268, 1245, 1175, 1073, 1005, 912, 827, 757, 729, 610, 574, 545. 5,7-bis(4-bromophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3j)



Yield: 64.0 mg, 66 %; yellow solid; mp 196-197 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.52 (s, 1H), 7.60 (d, *J* = 8.5 Hz, 2H), 7.55 (s, 1H), 7.51 (d, *J* = 8.5 Hz, 2H), 7.47 – 7.41 (m, 4H), 3.15 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.8, 167.1, 151.9, 138.3, 134.9, 134.8, 134.7, 134.1, 133.3, 132.6, 130.6, 130.5, 128.8, 128.5, 125.9, 115.9, 23.9. HRMS (ESI) m/z: calcd. for C<sub>21</sub>H<sub>12</sub>Br<sub>2</sub>NO<sub>3</sub><sup>-</sup> (M-H)<sup>-</sup> 483.9189, found 483.9190. IR (neat) v (cm<sup>-1</sup>): 3354, 3059, 2993, 2922, 2847, 1759, 1704, 1477, 1444, 1379, 1271, 1245, 1177, 1092, 1016, 911, 829, 757, 730, 649, 569.

#### 4-hydroxy-2-methyl-5,7-bis(4-(trifluoromethyl)phenyl)isoindoline-1,3-dione (3k)



Yield: 57.7 mg, 62 %; yellow solid; mp 179-180 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.57 (s, 1H), 7.82 – 7.66 (m, 8H), 7.62 (s, 1H), 3.17 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.6, 166.9, 152.2, 139.1, 138.6, 138.4, 134.7, 132.2, 130.8 (q, *J* = 32.6 Hz), 130.7 (q, *J* = 32.8 Hz), 129.7, 129.6, 126.8, 125.5 (q, *J* = 3.8 Hz), 125.3 (q, *J* = 3.7 Hz), 124.1 (q, *J* = 272.2 Hz), 124.0 (q, *J* = 270.1 Hz), 116.1, 23.9. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -62.61, -62.69. HRMS (ESI) m/z: calcd. for C<sub>23</sub>H<sub>14</sub>F<sub>6</sub>NO<sub>3</sub><sup>+</sup> (M+H)<sup>+</sup> 466.0872, found 466.0871. IR (neat) v (cm<sup>-1</sup>): 3369, 3078, 2991, 2924, 1761, 1697, 1446, 1384, 1321, 1245, 1160, 1113, 1067, 1007, 840, 762, 692.

#### dimethyl 4,4'-(7-hydroxy-2-methyl-1,3-dioxoisoindoline-4,6-diyl)dibenzoate (3l)



Yield: 56.1 mg, 63 %; yellow solid; mp 140-141 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.57 (s, 1H), 8.14 (dd, *J* = 8.2, 3.8 Hz, 4H), 7.75 (d, *J* = 8.1 Hz, 2H), 7.68 – 7.61 (m, 3H), 3.95 (s, 6H), 3.16 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.7, 166.9, 166.7, 166.6, 152.2, 140.2, 139.4, 138.6, 135.0, 132.6, 130.2, 130.1, 129.8, 129.5, 129.3, 129.2, 126.6, 116.0, 52.3, 52.2, 23.9. HRMS (ESI) m/z: calcd. for C<sub>25</sub>H<sub>19</sub>NNaO<sub>7</sub><sup>+</sup> (M+Na)<sup>+</sup> 468.1054, found 468.1058. IR (neat) v (cm<sup>-1</sup>): 3345, 2993, 2950, 1758, 1693, 1609, 1433, 1380, 1274, 1242, 1179, 1104, 1003, 966, 851, 752, 722, 698.

#### 4-hydroxy-2-methyl-5,7-di-m-tolylisoindoline-1,3-dione (3m)



Yield: 39.2 mg, 55 %; yellow solid; mp 69-70 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.48 (s, 1H), 7.61 (s, 1H), 7.47 (d, *J* = 7.1 Hz, 2H), 7.42 – 7.31 (m, 4H), 7.25 (d, *J* = 7.8 Hz, 2H), 3.15 (s, 3H), 2.43 (s, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.0, 167.3, 151.8, 138.9, 138.2, 137.9, 136.2, 135.9, 135.1, 134.0, 129.9, 129.8, 129.4, 129.2, 128.4, 128.1, 126.4, 126.3, 125.5, 115.6, 23.8, 21.54, 21.50. HRMS (ESI) m/z: calcd. for C<sub>23</sub>H<sub>19</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 380.1257, found 380.1265. IR (neat) v (cm<sup>-1</sup>): 3374, 3028, 2978, 2922, 1759, 1692, 1441, 1384, 1276, 1245, 1084, 1016, 787, 759, 699, 579, 542.

#### 5,7-bis(3-chlorophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3n)



Yield: 42.8 mg, 53 %; yellow solid; mp 207-208 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.53 (s, 1H), 7.65 (s, 1H), 7.56 (d, *J* = 8.1 Hz, 3H), 7.49 – 7.37 (m, 5H), 3.16 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.7, 166.9, 152.0, 138.5, 137.3, 136.6, 134.6, 134.5, 134.2, 132.2, 129.8, 129.5, 129.2, 128.8, 128.7, 127.6, 127.4, 126.3, 115.9, 23.9. HRMS (ESI) m/z: calcd. for C<sub>21</sub>H<sub>12</sub>Cl<sub>2</sub>NO<sub>3</sub><sup>-</sup> (M-H)<sup>-</sup> 396.0200, found 396.0201. IR (neat) v (cm<sup>-1</sup>): 3367, 3068, 1754, 1701, 1594, 1564, 1443, 1379, 1239, 1077, 1011, 863, 780, 740, 685, 578, 551.

#### 5,7-bis(3-bromophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (30)



Yield: 40.7 mg, 42 %; yellow solid; mp 204-205 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.53 (s, 1H), 7.80 (s, 1H), 7.70 (s, 1H), 7.63 – 7.54 (m, 4H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.34 (q, *J* = 7.6 Hz, 2H), 3.16 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.7, 166.9, 152.0, 138.5, 137.6, 136.9, 134.5, 132.0, 131.7, 131.6, 130.1, 129.7, 128.1, 127.8, 126.3, 122.6, 122.3, 115.9, 23.9. HRMS (ESI) m/z: calcd. for C<sub>21</sub>H<sub>12</sub>Br<sub>2</sub>NO<sub>3</sub><sup>-</sup> (M-H)<sup>-</sup> 483.9189, found 483.9193. IR (neat) v (cm<sup>-1</sup>): 3374, 3064, 1756, 1701, 1561, 1465, 1442, 1377, 1238, 1174, 1085, 1009, 863, 780, 756, 718, 676, 561. **4-hydroxy-2-methyl-5,7-di-o-tolylisoindoline-1,3-dione (3p**)



Yield: 25.3 mg, 33 %; yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.10 (s, 1H), 7.34 – 7.19 (m, 9H), 3.12 (s, 3H), 2.27 (s, 3H), 2.21 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.9, 167.2, 151.5, 139.9, 136.6, 136.5, 135.9, 135.0, 132.5, 130.2, 130.1, 129.8, 129.6, 128.7, 128.4, 127.2, 125.8, 125.6, 114.8, 23.7, 20.1, 20.0. HRMS (ESI) m/z: calcd. for C<sub>23</sub>H<sub>19</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 380.1257, found 380.1264. IR (neat) v (cm<sup>-1</sup>): 3418, 3021, 2925, 2861, 1764, 1694, 1441, 1382, 1242, 1003, 752, 556.

#### 5,7-bis(3,4-dichlorophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3q)



Yield: 39.2 mg, 55 %; yellow solid; mp 230-231 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.55 (s, 1H), 7.76 (d, *J* = 2.0 Hz, 1H), 7.65 (d, *J* = 2.1 Hz, 1H), 7.58 – 7.43 (m, 4H), 7.41 (dd, *J* = 8.3, 2.2 Hz, 1H), 3.16 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.5, 166.8, 152.0, 137.9, 135.3, 134.6, 133.7, 133.1, 133.0, 132.9, 132.6, 131.1, 131.0, 130.9, 130.6, 130.2, 128.7, 128.4, 126.5, 116.0, 24.0. HRMS (ESI) m/z: calcd. for C<sub>21</sub>H<sub>10</sub>Cl<sub>4</sub>NO<sub>3</sub><sup>-</sup> (M-H)<sup>-</sup> 463.9420, found 463.9418. IR (neat) v (cm<sup>-1</sup>): 3417, 3376, 2923, 1742, 1693, 1464, 1443, 1382, 1353, 1243, 1131, 1031, 1006, 879, 824, 757, 692, 623, 582.

#### 5,7-bis(2,3-dihydro-1H-inden-5-yl)-4-hydroxy-2-methylisoindoline-1,3-dione (3r)



Yield: 35.2 mg, 43 %; yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.47 (s, 1H), 7.61 (s, 1H), 7.51 (s,

1H), 7.46 – 7.41 (m, 2H), 7.41 – 7.27 (m, 3H), 3.15 (s, 3H), 3.02 - 2.93 (m, 8H), 2.16 - 2.09 (m, 4H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  167.3, 166.6, 152.7, 142.8, 139.2, 138.9, 136.6, 136.3, 136.1, 134.7, 130.8, 129.6, 129.5, 128.5, 128.4, 128.0, 127.7, 123.6, 115.1, 83.8, 37.9, 30.6, 24.9, 24.6, 20.2, 13.7, 13.6. HRMS (ESI) m/z: calcd. for C<sub>27</sub>H<sub>22</sub>NO<sub>3</sub><sup>-</sup> (M-H)<sup>-</sup> 408.1605, found 408.1601. IR (neat) v (cm<sup>-1</sup>): 3363, 2932, 2843, 1758, 1691, 1477, 1441, 1384, 1247, 1071, 1010, 820, 760, 738, 727, 678,659.

4-hydroxy-2-methyl-5,7-di(naphthalen-2-yl)isoindoline-1,3-dione (3s)



Yield: 52.3 mg, 61 %; yellow solid; mp 138-139 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.60 (s, 1H), 8.18 (s, 1H), 8.07 (s, 1H), 7.98 – 7.87 (m, 6H), 7.86 (s, 1H), 7.82 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.73 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.57 – 7.49 (m, 4H), 3.18 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.0, 167.3, 152.1, 139.3, 136.1, 133.9, 133.5, 133.3, 133.2, 133.1, 132.6, 128.7, 128.4, 128.3, 128.1, 127.8, 127.72, 127.69, 127.2, 126.8, 126.7, 126.6, 126.5, 126.4, 125.8, 115.8, 23.8. HRMS (ESI) m/z: calcd. for C<sub>29</sub>H<sub>19</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 452.1257, found 452.1260. IR (neat) v (cm<sup>-1</sup>): 3376, 3054, 2922, 2852, 1757, 1690, 1441, 1382, 1246, 1201, 1073, 1006, 886, 856, 815, 750, 677, 668, 642.





Yield: 55.0 mg, 52 %; yellow solid; mp 105-106 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.02 (s, 1H),

8.94 (s,1H), 8.74 (d, J = 8.1 Hz, 2H), 8.64 (s, 1H), 8.00 – 7.72 (m, 10H), 7.70 – 7.51 (m, 4H), 7.18 – 7.06 (m, 1H), 3.14 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.9, 167.3, 152.0, 139.3, 136.2, 134.1, 133.9, 133.2, 132.3, 131.92, 131.90, 130.4, 130.3, 130.2, 130.1, 129.2, 128.7, 128.6, 128.3, 127.9, 127.72, 127.70, 127.4, 126.92, 126.88, 126.8, 126.7, 126.5, 126.4, 125.7, 123.7, 122.7, 115.9, 23.8. HRMS (ESI) m/z: calcd. for C<sub>37</sub>H<sub>23</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 552.1570, found 552.1570. IR (neat) v (cm<sup>-1</sup>): 3361, 3053, 2921, 2851, 1753, 1691, 1440, 1379, 1239, 1004, 884, 838, 800, 740, 677, 665.

(7-hydroxy-2-methyl-1,3-dioxoisoindoline-4,6-diyl)bis(4,1-phenylene)bis(2-(4-isobutylphenyl) propanoate) (3u)



Yield: 75.2 mg, 51 %; yellow solid; mp 58-59 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.48 (s, 1H), 7.63 (d, *J* = 8.6 Hz, 2H), 7.56 (s, 1H), 7.53 (d, *J* = 8.6 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 4H), 7.16 (d, *J* = 7.8 Hz, 4H), 7.10 (dd, *J* = 8.5, 5.6 Hz, 4H), 3.97 (q, *J* = 7.0 Hz, 2H), 3.13 (s, 3H), 2.48 (d, *J* = 7.2 Hz, 4H), 1.91 – 1.84 (m, 2H), 1.63 (s, 3H), 1.62 (s, 3H), 0.92 (s, 6H), 0.91 (s, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  173.2, 170.9, 167.2, 151.8, 151.2, 151.1, 140.94, 140.91, 138.6, 137.14, 137.11, 135.2, 133.3, 132.9, 132.5, 130.3, 130.2, 129.6, 129.5, 127.7, 127.3, 127.2, 125.6, 121.9, 121.6, 121.3, 115.7, 45.3, 45.1, 30.2, 23.8, 22.4, 18.5, 18.4. HRMS (ESI) m/z: calcd. for C<sub>47</sub>H<sub>46</sub>NO<sub>7</sub> (M-H)<sup>-</sup> 736.3280, found 736.3283. IR (neat) v (cm<sup>-1</sup>): 3367, 2952, 2925, 1757, 1695, 1510, 1485, 1446, 1383, 1332, 1277, 1244, 1204, 1166, 1135, 1068, 1004, 895, 846, 763, 669.

(7-hydroxy-2-methyl-1,3-dioxoisoindoline-4,6-diyl)bis(4,1-phenylene)bis(2-(4-(2,2-dichlorocy clopropyl)phenoxy)-2-methylpropanoate) (3v)



Yield: 75.7 mg, 42 %; yellow solid; mp 83-84 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.50 (s, 1H), 7.65 (d, *J* = 8.7 Hz, 2H), 7.56 (d, *J* = 8.1 Hz, 3H), 7.18 (d, *J* = 8.6 Hz, 4H), 7.08 (dd, *J* = 8.8, 2.5 Hz, 4H), 6.96 (d, *J* = 8.6 Hz, 4H), 3.14 (s, 3H), 2.91 – 2.82 (m, 2H), 2.00 – 1.92 (m, 2H), 1.82 (d, *J* = 8.0 Hz, 2H), 1.78 (s, 12H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  172.8, 172.7, 170.8, 167.1, 155.0, 154.9, 151.8, 150.8, 150.7, 138.6, 135.0, 133.7, 132.9, 132.7, 130.9, 130.5, 130.4, 129.9, 128.6, 128.5, 125.8, 121.5, 121.2, 118.6, 115.8, 115.3, 79.4, 60.9, 34.8, 29.7, 25.9, 25.5, 23.8. HRMS (ESI) m/z: calcd. for C<sub>47</sub>H<sub>38</sub>Cl<sub>4</sub>NO<sub>9</sub><sup>-</sup> (M-H)<sup>-</sup> 900.1306, found 900.1304. IR (neat) v (cm<sup>-1</sup>): 3379, 2991, 2923, 2851, 1754, 1693, 1509, 1383, 1240, 1205, 1165, 1104, 1004, 869, 830, 759, 734.

#### 2-butyl-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3w)



Yield: 49.7 mg, 67 %; yellow solid; mp 73-74 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.59 (s, 1H), 7.69 (d, *J* = 7.5 Hz, 2H), 7.65 (s, 1H), 7.62 (d, *J* = 7.4 Hz, 2H), 7.53 – 7.42 (m, 6H), 3.67 (t, *J* = 7.2 Hz, 2H), 1.69 (p, *J* = 7.2 Hz, 2H), 1.40 (q, *J* = 7.5 Hz, 2H), 0.97 (t, *J* = 7.4 Hz, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 167.3, 151.9, 138.9, 136.0, 135.9, 135.2, 133.8, 129.3, 129.2, 128.60, 128.56, 128.5, 128.2, 127.4, 125.5, 115.6, 37.8, 30.6, 20.2, 13.7. HRMS (ESI) m/z: calcd. for C<sub>24</sub>H<sub>21</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup>

394.1414, found 394.1419. IR (neat) ν (cm<sup>-1</sup>): 3377, 2956, 2931, 2870, 2859, 1757, 1682, 1478, 1439, 1410, 1378, 1330, 1249, 1167, 1053, 948, 897, 873, 760, 747, 695, 638, 592.

### 4-hydroxy-2-octyl-5,7-diphenylisoindoline-1,3-dione (3x)



Yield: 63.3 mg, 74 %; yellow solid; mp 53-54 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.56 (s, 1H), 7.67 (d, *J* = 7.0 Hz, 2H), 7.64 (s, 1H), 7.60 (d, *J* = 6.8 Hz, 2H), 7.55 – 7.39 (m, 6H), 3.64 (t, *J* = 7.3 Hz, 2H), 1.67 (p, *J* = 7.1 Hz, 2H), 1.35 – 1.23 (m, 10H), 0.87 (t, *J* = 6.6 Hz, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 167.3, 151.9, 138.9, 136.0, 135.9, 135.2, 133.8, 129.3, 128.6, 128.5, 128.4, 128.2, 127.4, 125.5, 115.6, 38.1, 31.8, 29.2, 29.1, 28.6, 26.9, 22.6, 14.1. HRMS (ESI) m/z: calcd. for C<sub>28</sub>H<sub>29</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 450.2040, found 450.2032. IR (neat) v (cm<sup>-1</sup>): 3380, 3061, 3030, 2925, 2854, 1757, 1690, 1446, 1409, 1090, 1001, 862, 758, 696, 641, 594.

#### 2-cyclopentyl-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3y)



Yield: 32.2 mg, 42 %; yellow solid; mp 123-124 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.67 (s, 1H), 7.67 (d, *J* = 7.4 Hz, 2H), 7.62 (s, 1H), 7.59 (d, *J* = 7.5 Hz, 2H), 7.53 – 7.38 (m, 6H), 4.69 – 4.54 (m, 1H), 2.21 – 2.06 (m, 2H), 2.00 – 1.88 (m, 4H), 1.69 – 1.60 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$ 171.3, 167.3, 151.9, 138.8, 136.0, 135.9, 135.3, 133.6, 129.3, 129.2, 128.5, 128.4, 128.2, 127.4, 125.5, 115.6, 51.0, 29.6, 25.1. HRMS (ESI) m/z: calcd. for C<sub>25</sub>H<sub>22</sub>NO<sub>3</sub><sup>+</sup> (M+H)<sup>+</sup> 384.1594, found 384.1607. IR (neat) v (cm<sup>-1</sup>): 3246, 3064, 2957, 2864, 1751, 1674, 1416, 1403, 1371, 1281, 1258, 1199, 1088, 1065, 959, 900, 809, 772, 748, 619. 2-cyclohexyl-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3z)



Yield: 49.2 mg, 62 %; yellow solid; mp 114-115 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.70 (s, 1H), 7.68 (d, *J* = 7.4 Hz, 2H), 7.63 (s, 1H), 7.60 (d, *J* = 7.4 Hz, 2H), 7.52 – 7.39 (m, 6H), 4.08 (t, *J* = 12.1 Hz, 1H), 2.20 (q, *J* = 11.7 Hz, 2H), 1.82 (dd, *J* = 43.0, 11.7 Hz, 4H), 1.70 (d, *J* = 12.0 Hz, 1H), 1.38 – 1.24 (m, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.4, 167.2, 152.0, 138.8, 136.0, 135.9, 135.3, 133.6, 129.3, 129.2, 129.1, 128.6, 128.4, 128.2, 125.4, 115.5, 51.0, 29.9, 26.1, 25.2. HRMS (ESI) m/z: calcd. for C<sub>26</sub>H<sub>23</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 420.1570, found 420.1567. IR (neat) v (cm<sup>-1</sup>): 3373, 2924, 2850, 1752, 1686, 1479, 1444, 1402, 1375, 1250, 1171, 1098, 1071, 1036, 988, 947, 891, 811, 760, 695, 636.

#### 2-benzyl-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3aa)



Yield: 68.9 mg, 85 %; yellow solid; mp 189-190 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.53 (s, 1H), 7.66 (d, *J* = 7.2 Hz, 2H), 7.64 (s, 1H), 7.59 (d, *J* = 7.2 Hz, 2H), 7.51 – 7.42 (m, 8H), 7.36 – 7.29 (m, 3H), 4.82 (s, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.7, 166.8, 151.9, 139.1, 136.2, 136.1, 135.9, 135.1, 134.0, 129.3, 129.3, 129.2, 128.8, 128.7, 128.6, 128.3, 127.9, 127.4, 125.4, 115.6, 41.6. HRMS (ESI) m/z: calcd. for C<sub>27</sub>H<sub>19</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 428.1257, found 428.1237. IR (neat) v (cm<sup>-1</sup>): 3341, 3065, 3032, 2943, 1754, 1682, 1453, 1436, 1399, 1339, 1293, 1281, 1088, 1066, 1032, 959, 760, 701, 650.

### 4-hydroxy-2,5,7-triphenylisoindoline-1,3-dione (3ab)



Yield: 64.9 mg, 83%; yellow solid; mp 191-193 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.72 (s, 1H), 7.73 (s, 1H), 7.71 (d, *J* = 7.1 Hz, 2H), 7.63 (dd, *J* = 7.7, 1.9 Hz, 2H), 7.53 – 7.40 (m, 11H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.1, 166.1, 152.4, 139.5, 136.5, 135.8, 135.1, 134.4, 131.2, 129.4, 129.2, 129.1, 128.8, 128.6, 128.3, 128.2, 126.4, 124.9, 115.2. HRMS (ESI) m/z: calcd. for C<sub>26</sub>H<sub>17</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 414.1101, found 414.1095. IR (neat) v (cm<sup>-1</sup>): 3385, 3062, 3026, 1760, 1702, 1503, 1478, 1399, 1383, 1277, 1195, 1143, 1112, 1065, 1027, 983, 920, 893, 829, 769, 739, 685, 638, 630.

4-hydroxy-5,7-diphenyl-2-(p-tolyl)isoindoline-1,3-dione (3ac)



Yield: 57.6 mg, 76 %; yellow solid; mp 205-206 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.71 (s, 1H), 7.71 (d, *J* = 7.7 Hz, 3H), 7.62 (d, *J* = 7.1 Hz, 2H), 7.52 – 7.41 (m, 6H), 7.33 – 7.28 (m, 4H), 2.40 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.2, 166.2, 152.4, 139.4, 138.3, 136.4, 135.8, 135.1, 134.3, 129.8, 129.4, 129.2, 128.7, 128.6, 128.5, 128.2, 126.3, 125.0, 115.3, 21.2. HRMS (ESI) m/z: calcd. for C<sub>27</sub>H<sub>19</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 428.1257, found 428.1255. IR (neat) v (cm<sup>-1</sup>): 3383, 3029, 2920, 1754, 1698, 1515, 1478, 1443, 1401, 1384, 1277, 1196, 1178, 1144, 1065, 1028, 919, 895, 781, 688, 588.

4-hydroxy-2-(4-methoxyphenyl)-5,7-diphenylisoindoline-1,3-dione (3ad)



Yield: 67.4 mg, 80 %; yellow solid; mp 185-186 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.70 (s, 1H), 7.71 (d, *J* = 7.4 Hz, 3H), 7.63 (d, *J* = 7.1 Hz, 2H), 7.54 – 7.41 (m, 6H), 7.34 (d, *J* = 9.0 Hz, 2H), 7.00 (d, *J* = 8.9 Hz, 2H), 3.84 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.3, 166.4, 159.3, 152.4, 139.4, 136.4, 135.8, 135.1, 134.3, 129.4, 129.2, 128.7, 128.6, 128.2, 127.8, 127.4, 124.9, 123.8, 115.3, 114.5, 55.6. HRMS (ESI) m/z: calcd. for C<sub>27</sub>H<sub>19</sub>NNaO<sub>4</sub><sup>+</sup> (M+Na)<sup>+</sup> 444.1206, found 444.1205. IR (neat) v (cm<sup>-1</sup>): 3400, 3059, 3026, 1760, 1702, 1508, 1478, 1383, 1277, 1196, 1143, 1112, 1065, 920, 893, 829, 739, 685, 658.

2-(4-chlorophenyl)-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3ae)



Yield: 57.8 mg, 68 %; yellow solid; mp 245-247 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.65 (s, 1H), 7.72 (s, 1H), 7.70 (d, *J* = 7.8 Hz, 2H), 7.61 (dd, *J* = 7.6, 1.8 Hz, 2H), 7.53 – 7.39 (m, 10H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  169.7, 165.8, 152.5, 139.6, 136.7, 135.7, 135.0, 134.6, 133.9, 129.8, 129.3, 129.2, 128.8, 128.7, 128.6, 128.3, 127.5, 124.7, 115.1. HRMS (ESI) m/z: calcd. for C<sub>26</sub>H<sub>16</sub>ClNNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 448.0711, found 448.0711. IR (neat) v (cm<sup>-1</sup>): 3399, 3032, 2922, 1754, 1698, 1493, 1446, 1383, 1278, 1198, 1146, 1088, 1063, 982, 919, 819, 772, 689, 617.

2-(4-bromophenyl)-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3af)



Yield: 65.7 mg, 70 %; yellow solid; mp 240-241 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.65 (s, 1H), 7.73 (s, 1H), 7.70 (d, *J* = 7.8 Hz, 2H), 7.65 – 7.58 (m, 4H), 7.54 – 7.42 (m, 6H), 7.36 (d, *J* = 8.9 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  169.7, 165.7, 152.5, 139.6, 136.7, 135.7, 134.9, 134.6, 132.3, 130.3, 129.3, 129.2, 128.8, 128.7, 128.6, 128.3, 127.8, 124.7, 121.9, 115.0. HRMS (ESI) m/z: calcd. for C<sub>26</sub>H<sub>15</sub>BrNO<sub>3</sub><sup>-</sup> (M-H)<sup>-</sup> 468.0241, found 468.0238. IR (neat) v (cm<sup>-1</sup>): 3392, 3030, 2923, 1754, 1698, 1489, 1473, 1381, 1278, 1195, 1146, 1116, 1062, 1012, 919, 834, 817, 744, 691, 613. 4-hydroxy-2-(naphthalen-1-yl)-5,7-diphenylisoindoline-1,3-dione (3ag)



Yield: 71.5 mg, 81 %; yellow solid; mp 231-232 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.71 (s, 1H), 8.04 – 7.92 (m, 2H), 7.80 (s, 1H), 7.77 – 7.67 (m, 5H), 7.60 (t, *J* = 7.8 Hz, 1H), 7.56 – 7.42 (m, 9H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.5, 166.6, 152.5, 139.6, 136.7, 135.7, 135.1, 134.6, 134.5, 130.3, 130.1, 129.6, 129.4, 129.3, 129.2, 128.8, 128.7, 128.6, 128.3, 127.7, 127.5, 127.3, 127.1, 126.6, 125.4, 125.2, 122.5, 115.5. HRMS (ESI) m/z: calcd. for C<sub>30</sub>H<sub>19</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 464.1257, found 464.1262. IR (neat) v (cm<sup>-1</sup>): 3365, 2956, 2867, 1759, 1696, 1440, 1379, 1246, 1177, 1119, 1076, 1005, 835, 763, 632, 555.

4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3ah)



Yield: 49.1 mg, 78 %; yellow solid; mp 169-170 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.49 (s, 1H), 7.88 (s, 1H), 7.72 – 7.64 (m, 3H), 7.58 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.53 – 7.39 (m, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.7, 166.7, 152.3, 139.6, 136.4, 135.7, 135.0, 134.3, 129.3, 129.2, 128.7, 128.6, 128.5, 128.3, 125.9, 116.1. HRMS (ESI) m/z: calcd. for C<sub>20</sub>H<sub>13</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 338.0788, found 338.0796. IR (neat) v (cm<sup>-1</sup>): 3397, 3200, 3034, 2920, 1760, 1703, 1478, 1446, 1400, 1374, 1273, 1231, 1177, 1070, 1054, 965, 759, 697, 637.

3,7-dihydroxy-2,4,6-triphenylisoindolin-1-one (4)



Yield: 47.2 mg, 60 %; white solid; mp 185-186 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.31 (s, 1H), 7.74 (d, *J* = 8.1 Hz, 2H), 7.67 (d, *J* = 7.9 Hz, 2H), 7.63 (s, 1H), 7.60 (d, *J* = 7.8 Hz, 2H), 7.52 – 7.35 (m, 8H), 7.23 (d, *J* = 7.2 Hz, 1H), 6.59 (d, *J* = 9.8 Hz, 1H), 2.86 (d, *J* = 9.9 Hz, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  168.7, 152.9, 138.2, 137.8, 137.0, 136.4, 135.9, 131.0, 130.3, 129.3, 129.2, 128.9, 128.5, 128.4, 127.9, 127.8, 125.8, 121.9, 115.9, 83.2. HRMS (ESI) m/z: calcd. for C<sub>26</sub>H<sub>20</sub>NO<sub>3</sub><sup>+</sup> (M+H)<sup>+</sup> 394.1438, found 394.1440. IR (neat) v (cm<sup>-1</sup>): 3381, 3262, 3054, 2921, 1655, 1596, 1500, 1408, 1280, 1186, 1144, 1118, 1029, 989, 913, 793, 775, 683, 630, 558.

#### 4-hydroxy-2-(4-methoxyphenethyl)-5,7-diphenylisoindoline-1,3-dione (5)



Yield: 63.8 mg, 71 %; yellow solid; mp 136-137 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.50 (s, 1H), 7.67 (d, *J* = 7.4 Hz, 2H), 7.63 (s, 1H), 7.57 (dd, *J* = 7.9, 1.7 Hz, 2H), 7.51 – 7.40 (m, 6H), 7.17 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 3.84 (t, *J* = 7.6 Hz, 2H), 3.79 (s, 3H), 2.92 (t, *J* = 7.6 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.8, 167.1, 158.4, 151.9, 138.9, 136.1, 135.9, 135.2, 133.9, 129.9, 129.8, 129.3, 129.2, 128.6, 128.5, 128.5, 128.2, 125.4, 115.5, 114.1, 55.3, 39.5, 33.8. HRMS (ESI) m/z: calcd. for C<sub>29</sub>H<sub>24</sub>NO<sub>4</sub><sup>+</sup> (M+H)<sup>+</sup> 450.1700, found 450.1711. IR (neat) v (cm<sup>-1</sup>): 3388, 2925, 2853, 2834, 1753, 1686, 1610, 1510, 1438, 1408, 1378, 1327, 1236, 1170, 1083, 1028, 997, 817, 754, 697, 638, 586.

#### 4-(benzyloxy)-2,5,7-triphenylisoindoline-1,3-dione (6)



Yield: 51.0 mg, 53 %; white solid; mp 192-193 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.70 (s, 1H), 7.64 – 7.57 (m, 4H), 7.51 – 7.37 (m, 11H), 7.26 – 7.18 (m, 5H), 5.02 (s, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.2, 165.5, 153.3, 143.4, 139.5, 137.4, 136.2, 136.0, 135.9, 131.7, 129.6, 129.5, 129.1, 128.9, 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 128.0, 127.5, 126.9, 126.7, 123.5. HRMS (ESI) m/z: calcd. for C<sub>33</sub>H<sub>24</sub>NO<sub>3<sup>+</sup></sub> (M+H)<sup>+</sup> 482.1751, found 482.1763. IR (neat) v (cm<sup>-1</sup>): 3057, 3031, 2339, 2314, 1767, 1714, 1495, 1470, 1441, 1360, 1198, 1113, 990, 911, 850, 742, 696.

# 2-butyl-5,7-diphenyl-4-((4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)oxy)isoindolin e-1,3-dione (7)



Yield: 89.2 mg, 76 %; brown oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.68 (d, *J* = 8.0 Hz, 2H), 7.60 (s, 1H), 7.60 – 7.50 (m, 4H), 7.49 – 7.39 (m, 6H), 7.15 (d, *J* = 7.8 Hz, 2H), 5.08 (s, 2H), 3.67 (t, *J* = 7.3 Hz, 2H), 1.69 – 1.63 (m, 2H), 1.42 – 1.35 (m, 2H), 1.34 (s, 12H), 0.94 (t, *J* = 7.4 Hz, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 167.5, 151.6, 145.0, 144.8, 144.7, 144.3, 139.0, 136.6, 134.5, 133.9, 133.1, 127.3, 127.2, 125.14, 125.11, 125.0, 124.4, 124.1, 115.5, 32.9, 32.8, 32.7, 25.5, 23.7. HRMS (ESI) m/z: calcd. for C<sub>37</sub>H<sub>38</sub>BNNaO<sub>5</sub><sup>+</sup> (M+Na)<sup>+</sup> 610.2735, found 610.2721. IR (neat) v (cm<sup>-1</sup>): 3060, 2974, 2932, 2872, 1762, 1706, 1468, 1441, 1397, 1357, 1273, 1236, 1144, 1088, 960, 859, 761, 698, 657.

#### 1,3-dioxo-2,5,7-triphenylisoindolin-4-yl trifluoromethanesulfonate (8)



Yield: 79.5 mg, 76 %; grayish brown solid; mp 185-186 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.83 (s, 1H), 7.64 – 7.37 (m, 15H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  164.9, 163.9, 142.0, 141.5, 140.7, 140.3, 134.7, 133.8, 131.1, 129.7, 129.5, 129.4, 129.1, 128.9, 128.4, 128.3, 127.3, 126.6, 125.6 (q, *J* = 146.2 Hz), 125.4, 119.6, 116.4. HRMS (ESI) m/z: calcd. for C<sub>27</sub>H<sub>17</sub>F<sub>3</sub>NO<sub>5</sub>S<sup>+</sup> (M+H)<sup>+</sup> 524.0774, found 524.0772. IR (neat) v (cm<sup>-1</sup>): 2984, 2942, 2908, 1778, 1724, 1492, 1423, 1369, 1237, 1210, 1158, 1044, 973, 908, 850, 700, 623, 586.

4-(4-methoxyphenyl)-2,5,7-triphenylisoindoline-1,3-dione (9)



Yield: 67.3 mg, 70 %; White solid; mp 202-203 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 (s, 1H), 7.66 (dd, J = 7.5, 2.0 Hz, 2H), 7.51 – 7.44 (m, 3H), 7.43 – 7.37 (m, 4H), 7.35 – 7.30 (m, 1H), 7.25 – 7.22 (m, 3H), 7.18 – 7.10 (m, 4H), 6.81 (d, J = 8.7 Hz, 2H), 3.79 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.5, 166.4, 159.2, 148.7, 140.2, 139.3, 138.6, 138.3, 136.2, 131.7, 129.7, 129.6, 128.8, 128.7, 128.1, 127.9, 127.5, 126.8, 126.7, 113.1, 55.1. HRMS (ESI) m/z: calcd. for C<sub>33</sub>H<sub>23</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup> 504.1570, found 504.1575. IR (neat) v (cm<sup>-1</sup>): 3062, 3003, 2922, 2837, 1768, 1715, 1605, 1514, 1500, 1461, 1439, 1379, 1246, 1118, 1025, 913, 733, 645, 578.

4-((4-methoxyphenyl)amino)-2,5,7-triphenylisoindoline-1,3-dione (10)



Yield: 55.6 mg, 56 %; yellow solid; mp 179-181 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.54 (s, 1H), 7.62 (d, *J* = 6.6 Hz, 2H), 7.54 (s, 1H), 7.49 – 7.35 (m, 8H), 7.27-7.25 (m, 2H), 7.09 (d, *J* = 5.8 Hz, 3H), 6.66 (d, *J* = 8.8 Hz, 2H), 6.46 (d, *J* = 8.9 Hz, 2H), 3.65 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  169.5, 166.5, 155.7, 141.7, 141.2, 138.4, 136.7, 136.3, 133.8, 132.6, 131.7, 129.4, 129.0, 128.3, 128.2, 128.1, 128.0, 127.9, 127.5, 126.7, 126.1, 123.1, 116.1, 113.6, 55.5. HRMS (ESI) m/z: calcd. for C<sub>33</sub>H<sub>23</sub>N<sub>2</sub>O<sub>3</sub><sup>-</sup> (M-H)<sup>-</sup> 495.1714, found 495.1716. IR (neat) v (cm<sup>-1</sup>): 3306, 3056, 2920, 2851, 1763, 1692, 1617, 1510, 1468, 1442, 1387, 1238, 1159, 1114, 1038, 924, 819, 755, 737, 695, 624.

(E)-1,3-diphenylbut-2-en-1-one (A) [4]

Yield: 20.4 mg, 46 %; yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.01 (d, J = 7.2 Hz, 2H), 7.62 – 7.52 (m, 3H), 7.52 – 7.35 (m, 5H), 7.18 (s, 1H), 2.61 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  191.9, 155.2, 142.8, 139.4, 132.6, 129.2, 128.7, 128.6, 128.3, 126.5, 122.1, 18.9.

#### 2-methyl-4,6-diphenylisoindoline-1,3-dione (3a')



Yield: 5.6 mg, 9%; yellow solid; mp 146-147 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.08 (s, 1H), 7.85 (s, 1H), 7.69 (d, *J* = 7.3 Hz, 2H), 7.62 (d, *J* = 7.6 Hz, 2H), 7.53 – 7.45 (m, 6H), 3.18 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  168.2, 167.9, 147.0, 141.4, 138.9, 136.3, 134.4, 134.3, 129.4, 129.2, 129.0, 128.9, 128.2, 127.4, 126.1, 120.7, 24.0. HRMS (ESI) m/z: calcd. for C<sub>21</sub>H<sub>15</sub>NNaO<sub>2</sub><sup>+</sup> (M+Na)<sup>+</sup>336.0995, found 336.1008. IR (neat) v (cm<sup>-1</sup>): 3060, 2924, 2353, 1770, 1710, 1433, 1374, 1255, 1175, 1031, 890, 765, 688, 600.

# 7. X-ray diffraction data of compound 3a.



Table 1 Crystal data and structure refinement for 3a (CCDC 2194562).		
Identification code	1357	
Empirical formula	$C_{21}H_{15}NO_3$	

Formula weight	329.34
Temperature/K	219.99(10)
Crystal system	orthorhombic
Space group	Pca2 <sub>1</sub>
a/Å	15.55070(17)
b/Å	14.54247(18)
c/Å	7.17802(9)
α/°	90
β/°	90
γ/°	90
Volume/Å <sup>3</sup>	1623.28(3)
Z	4
$\rho_{calc}g/cm^3$	1.348
µ/mm <sup>-1</sup>	0.735
F(000)	688.0
Crystal size/mm <sup>3</sup>	0.12 imes 0.11 imes 0.09
Radiation	Cu Ka ( $\lambda = 1.54184$ )
$2\Theta$ range for data collection/°	6.078 to 172.726
Index ranges	$-17 \le h \le 17, -16 \le k \le 18, -8 \le l \le 5$
Reflections collected	7012
Independent reflections	2091 [ $R_{int} = 0.0214$ , $R_{sigma} = 0.0211$ ]
Data/restraints/parameters	2091/9/229
Goodness-of-fit on F <sup>2</sup>	1.058
Final R indexes [I>=2σ (I)]	$R_1 = 0.0298, wR_2 = 0.0847$
Final R indexes [all data]	$R_1 = 0.0309, wR_2 = 0.0858$
Largest diff. peak/hole / e Å-3	0.18/-0.15
Flack parameter	0.22(14)

# 8. H-H NOESY spectra of 4



# 9. References

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- Li, X.; Zhang, X.; Zhang, F.; Luo, X.; Luo, H., Construction of Pyridine Ring Systems by Mn(OAc)<sub>2</sub>-Promoted Formal Dehydrative Dehydroaromatizing [4+2] Cycloaddition of Enamides with Maleimides. *Adv. Synth. Catal.* 2022, **364**, 1683-1688.
- 3. Hwang, J. Y.; Ji, A. Y.; Lee, S. H.; Kang, E. J., Redox-Selective Iron Catalysis for α-Amino C–H Bond Functionalization via Aerobic Oxidation. *Org. Lett.* 2020, **22**, 16-21.
- Na, F.; Lopez, S. S.; Beauseigneur, A.; Hernandez, L. W.; Sun, Z.; Antilla, J. C., Catalytic Asymmetric Transfer Hydrogenation of trans-Chalcone Derivatives Using BINOL-derived Boro-phosphates. *Org. Lett.* 2020, 22, 5953-5957.

# 10. Copies of NMR spectra of all products



4-hydroxy-2-methyl-5,7-diphenylisoindoline-1,3-dione (3a)

# 4-hydroxy-2-methyl-5,7-di-p-tolylisoindoline-1,3-dione (3b)



S35

# 5,7-bis(4-(tert-butyl)phenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3c)


# 4-hydroxy-5,7-bis(4-isobutylphenyl)-2-methylisoindoline-1,3-dione (3d)



## 4-hydroxy-5,7-bis(4-methoxyphenyl)-2-methylisoindoline-1,3-dione (3e)



5,7-di([1,1'-biphenyl]-4-yl)-4-hydroxy-2-methylisoindoline-1,3-dione (3f)



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S40



5,7-bis(4-fluorophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3h)





10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210

## 5,7-bis(4-chlorophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3i)



# 5,7-bis(4-bromophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3j)



# 4-hydroxy-2-methyl-5,7-bis(4-(trifluoromethyl)phenyl)isoindoline-1,3-dione (3k)





dimethyl 4,4'-(7-hydroxy-2-methyl-1,3-dioxoisoindoline-4,6-diyl)dibenzoate (3l)





4-hydroxy-2-methyl-5,7-di-m-tolylisoindoline-1,3-dione (3m)





5,7-bis(3-chlorophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3n)





5,7-bis(3-bromophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (30)





4-hydroxy-2-methyl-5,7-di-o-tolylisoindoline-1,3-dione (3p)





5,7-bis(3,4-dichlorophenyl)-4-hydroxy-2-methylisoindoline-1,3-dione (3q)





5,7-bis(2,3-dihydro-1H-inden-5-yl)-4-hydroxy-2-methylisoindoline-1,3-dione (3r)







4-hydroxy-2-methyl-5,7-di(phenanthren-2-yl)isoindoline-1,3-dione (3t)





(7-hydroxy-2-methyl-1, 3-dioxoisoindoline-4, 6-diyl) bis (4, 1-phenylene) bis (2-(4-isobutyl phenyl)) bis (4, 1-phenylene) bis (2-(4-isobutyl phenylene)) bis (4, 1-phenylene) bis (2-(4-isobutyl phenylene)) bis (4, 1-phenylene) bis (2-(4-isobutyl phenylene)) bis (4, 1-phenylene) bis (4,





(7-hydroxy-2-methyl-1, 3-dioxoisoindoline-4, 6-diyl) bis (4, 1-phenylene) bis (2-(4-(2, 2-dichlorocynthetal)) bis (2-(4-(2, 2-dichlorocy

clopropyl)phenoxy)-2-methylpropanoate) (3v)





2-butyl-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3w)



9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5









2-cyclopentyl-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3y)





2-cyclohexyl-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3z)





2-benzyl-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3aa)





4-hydroxy-2,5,7-triphenylisoindoline-1,3-dione (3ab)





4-hydroxy-5,7-diphenyl-2-(p-tolyl)isoindoline-1,3-dione (3ac)





4-hydroxy-2-(4-methoxyphenyl)-5,7-diphenylisoindoline-1,3-dione (3ad)





2-(4-chlorophenyl)-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3ae)





2-(4-bromophenyl)-4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3af)





4-hydroxy-2-(naphthalen-1-yl)-5,7-diphenylisoindoline-1,3-dione (3ag)





4-hydroxy-5,7-diphenylisoindoline-1,3-dione (3ah)





3,7-dihydroxy-2,4,6-triphenylisoindolin-1-one (4)







4-(benzyloxy)-2,5,7-triphenylisoindoline-1,3-dione (6)

77,7703 77,6105 77,61015 77,5101 77,5101 77,5101 77,510 77,520 77,520 77,520 77,520 77,520 77,520 77,420 77,7200 77,7200 77,7200 77,7200 77,7200 77,7200 77,7200 77,7200 77,72000



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





2-butyl-5,7-diphenyl-4-((4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)oxy)isoindolin e-1,3-dione (7)




1,3-dioxo-2,5,7-triphenylisoindolin-4-yl trifluoromethanesulfonate (8)

- 0.015

## 7,833 7,563 7,563 7,564 7,564 7,559









<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





4-((4-methoxyphenyl)amino)-2,5,7-triphenylisoindoline-1,3-dione (10)









170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0