Organic & Biomolecular Chemistry

PAPER

View Article Online
View Journal | View Issue

Cite this: *Org. Biomol. Chem.*, 2019, **17**, 7517

Ruthenium(II)-catalysed selective C(sp^2)–H bond benzoxylation of biologically appealing *N*-arylisoindolinones†

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Received 14th May 2019,
Accepted 22nd July 2019

DOI: 10.1039/c9ob01122f

rsc.li/obc

Site- and regio-selective aromatic C–H bond benzoxylations were found to take place using biologically appealing *N*-arylisoindolinones under ruthenium(II) catalysis in the presence of (hetero)aromatic carboxylic acid derivatives as coupling partners. Besides the presence of two potential C(sp^2)–H sites available for functionalization in the substrates, exclusive *ortho* selectivity was achieved in the phenyl ring attached to the nitrogen atom. Notably, the reactions occurred in a selective manner as only mono-functionalized products were formed and they tolerated a large number of functional chemical groups. The ability of the cyclic tertiary amide within the isoindolinone skeleton to act as a weak directing group in order to accommodate six-membered ring ruthenacycle intermediates appears to be the key to reach such high levels of selectivity. In contrast, the more sterically demanding cyclic imides were unreactive under identical reaction conditions.

Introduction

Since the pioneering contributions from Crabtree and Sanford on palladium-catalysed C–H bond acyloxylation reactions,[1] metal-catalysed C–H bond oxidation has represented an important entry to form new C–O bonds in valuable organic molecules.[2] Indeed, it enables one to rapidly access molecular diversity using different transition metal catalysts derived from Pd, Rh, Ir, Ru, Fe, Cu, Co, *etc*.[3] The main advantage of the transition metal-catalysed C–H bond acyloxylation reactions is that they traditionally operate under mild reaction conditions, thus avoiding the use of harsh oxidizing reagents which are not always compatible with many sensitive functional groups.[4] So far, most of the research in this area has been devoted to the use of nitrogen-containing directing groups that are capable of strongly coordinating to the metal catalyst leading to the formation of key metallacycle species.[4] Making these methodologies compatible with weak directing groups is rather attractive[5] as they can be further implemented in the late stage functionalization of chemicals relevant for medicine, pharmacology, agrochemistry and materials sciences.[6]

A particular class of useful weak directing groups are amides. They have been used in many transition metal-catalysed C–H bond functionalization reactions,[7] although their use in C–O bond forming reactions is rare.[8] In addition, *N*-substituted phenylbenzamides (Ar^1-CONR-Ar^2) are a subfamily of amides that present two aromatic fragments with two types of C–H bonds that can *a priori* react in a similar manner.[9–11] In the case of C–O bond forming reactions *via* transition metal-catalysed C–H bond functionalizations, Rao *et al.* showed that ruthenium catalysts enabled the hydroxylation to take place in a C–H bond from the benzamide ring **A** (Fig. 1a) while palladium catalysts hydroxylated the C–H bond from the acetanilide ring **B** (Fig. 1b) in TFA/TFAA medium (TFA = trifluoroacetic acid, TFAA = trifluoroacetic acid anhydride).[9] Additionally, Jeganmohan *et al.* described the difficulty in performing selective C–H bond benzoxylation reactions with *N*-substituted phenylbenzamides as both possible products are formed in a 1 : 1 ratio (Fig. 1c).[10] We recently applied a modified Rao's methodology to one example of *N*-arylisoindolinone,[11] which is the simplest cyclic version of *N*-substituted phenylbenzamides and the skeleton of which is found in many biologically relevant compounds.[12] For *N*-phenylisoindolinone, the C–H bond hydroxylation exclusively occurred in the aromatic ring **B** either with palladium or ruthenium catalysts (Fig. 1d).[11] These examples show the current limitations and challenges encountered in C–H bond acyloxylation reactions in order to discriminate between two aromatic C–H sites for tertiary amides as weak directing

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† Electronic supplementary information (ESI) available: ^{1}H and ^{13}C NMR spectra for all new compounds. CCDC 1910244 (**2a**), 1910245 (**2b**), 1910246 (**2c**), 1910247 (**2o**), and 1910248 (**2t**). For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/c9ob01122f

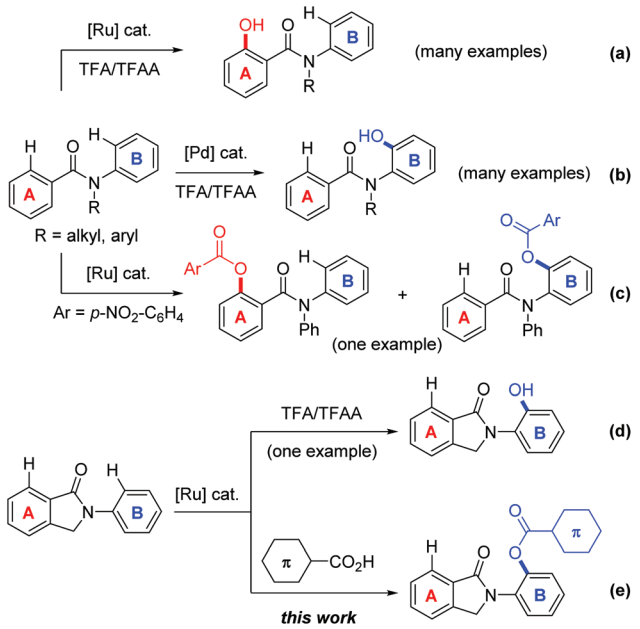


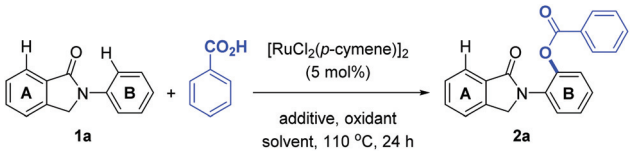
Fig. 1 State-of-the-art for the transition metal-catalysed C–H bond hydro/benzoxylations of *N*-substituted phenylbenzamides as weak directing groups (a–d) and the present work (e).

groups. It is relevant to note that ruthenium-catalysed C(sp^2)–H bond benzoxylation reactions using strong directing groups have been studied recently.[13] Herein, we report the development and scope of ruthenium catalysis enabling C–H bond benzoxylation reactions exclusively in the acetanilide ring **B** from the biologically relevant *N*-arylisoindolinone (Fig. 1e).

Results and discussion

Initially, we focused on the ruthenium-catalysed benzoxylation of isoindolinone **1a** in the presence of benchmark benzoic acid (1.5 equiv.) as the coupling partner (Table 1). A systematic study was performed using the air and moisture stable $[RuCl_2(p\text{-cymene})]_2$ complex as the pre-catalyst (5 mol%) in combination with different additives and oxidants (Table 1). First, we tested the reaction conditions employed by Jeganmohan and co-workers for the benzoxylation of acyclic tertiary amides (Fig. 1c),[10] namely, $AgSbF_6$ (20 mol%) as a chloride scavenger and $(NH_4)_2S_2O_8$ (2 equiv.) as an oxidant in 1,2-dichloroethane (DCE) as a solvent at 100 °C. After 24 h, only trace amounts of product **2a** were detected (entry 1). Interestingly, increasing the temperature of the reaction to 110 °C afforded **2a** in 66% yield (entry 2), with no functionalization in the benzene ring **A**. A similar result (69% yield) was observed when the reaction was conducted with Ag_2CO_3 as the oxidant (entry 3). However, the use of $K_2S_2O_8$ or $PhI(OAc)_2$ as the oxidant, respectively, was detrimental to the formation of **2a** (entries 4 and 5) as was the use of KPF_6 or AgOTf as additives, respectively (entries 6 and 7). The use of 2-MeTHF as the solvent provided **2a** in 66% yield (entry 8) and other solvents

Table 1 Optimization of the reaction conditions for the ruthenium-catalysed benzoxylation[a]



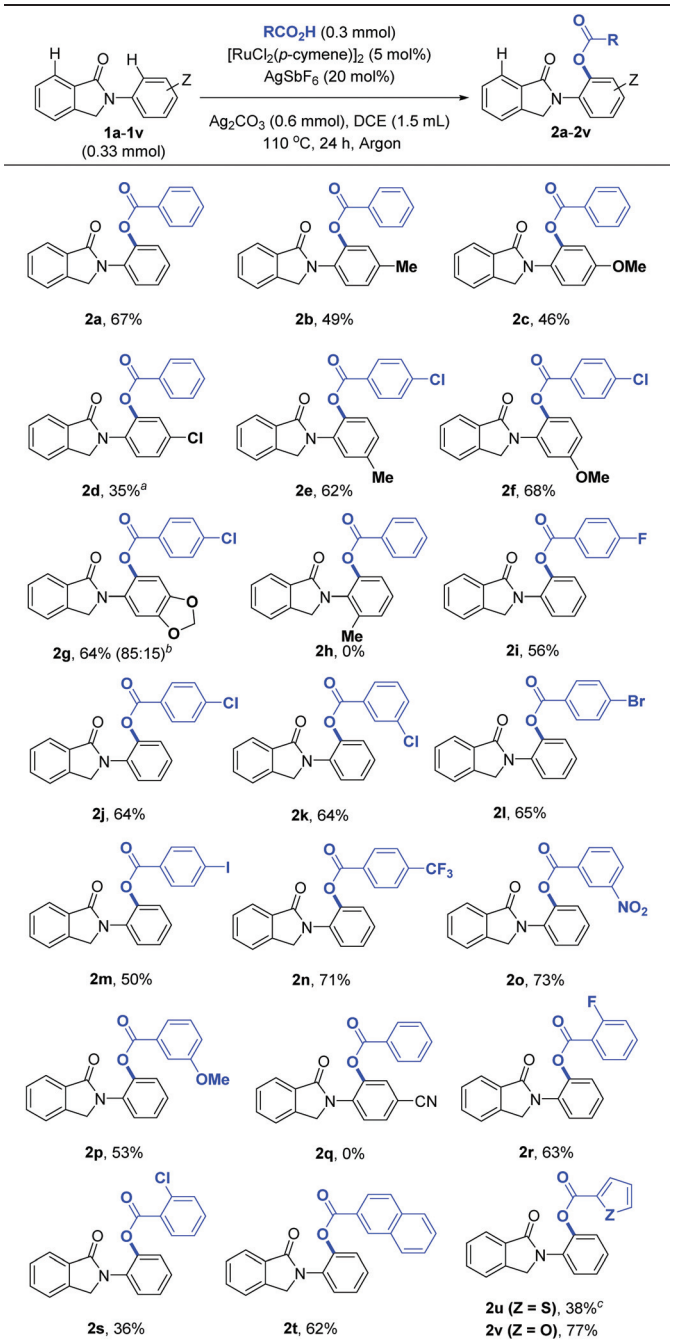
Entry	Additive	Oxidant	Solvent	Yield[b] (%)
1[c]	$AgSbF_6$	$(NH_4)_2S_2O_8$	DCE	Traces
2	$AgSbF_6$	$(NH_4)_2S_2O_8$	DCE	66
3	$AgSbF_6$	Ag_2CO_3	DCE	69 (67)
4	$AgSbF_6$	$K_2S_2O_8$	DCE	Traces
5	$AgSbF_6$	$PhI(OAc)_2$	DCE	Traces
6	KPF_6	Ag_2CO_3	DCE	Traces
7	AgOTf	Ag_2CO_3	DCE	Traces
8	$AgSbF_6$	Ag_2CO_3	2-MeTHF	66
9	$AgSbF_6$	Ag_2CO_3	1,4-Dioxane	36
10	$AgSbF_6$	Ag_2CO_3	DMF	0
11	$AgSbF_6$	Ag_2CO_3	Toluene	49
12[d]	$AgSbF_6$	Ag_2CO_3	DCE	56
13[e]	$AgSbF_6$	Ag_2CO_3	DCE	77 (74)
14[f]	$AgSbF_6$	Ag_2CO_3	DCE	0
15	—	Ag_2CO_3	DCE	0
16	$AgSbF_6$	—	DCE	0

[a] Reaction conditions: **1a** (0.1 mmol, 1 equiv.), benzoic acid (0.15 mmol, 1.5 equiv.), $[RuCl_2(p\text{-cymene})]_2$ (5 mol%), additive (20 mol%), oxidant (0.2 mmol, 2 equiv.), solvent (0.5 mL), 110 °C, 24 h, argon. [b] Determined by ^1H NMR spectroscopy analysis using dibromomethane as the internal standard; isolated yields obtained after purification by column chromatography are given in parentheses. [c] 100 °C. [d] Benzoic acid (0.12 mmol, 1.2 equiv.). [e] **1a** (0.11 mmol, 1.1 equiv.), benzoic acid (0.1 mmol, 1 equiv.). [f] Without $[RuCl_2(p\text{-cymene})]_2$.

such as 1,4-dioxane, *N*,*N*-dimethylformamide (DMF) and toluene were not as high yielding as DCE (entries 9–11). Decreasing the amount of benzoic acid to 1.2 equiv. afforded **2a** in 56% yield (entry 12). Conversely, using a slight excess of substrate **1a** (1.1 equiv.) with respect to benzoic acid (1 equiv.) gave rise to **2a** in 74% yield of isolated product (entry 13), which constituted the highest yielding reaction conditions so far obtained. Furthermore, no bis-functionalization was observed. The starting substrate **1a** was recovered unreacted when the reaction was carried out in the absence of $[RuCl_2(p\text{-cymene})]_2$, $AgSbF_6$ and Ag_2CO_3, respectively (entries 14–16), thus, supporting the need of all reagents for this transformation.

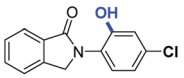
With the developed reaction conditions in hand (Table 1, entry 13), we evaluated the scope for this transformation (Table 2). The catalysis was compatible with different *para*- and *meta*-substitution patterns in the phenyl ring **B** of the *N*-arylisoindolinone backbone. As such, benzoxylated products **2b–2g** that contain methyl, methoxy, chloro and dioxolane functional groups were obtained in 35%–68% yields. During the purification of **2d**, the debenzoylated alcohol product **2dd** was isolated in 19%, and in the case of **2g**, small amounts of the other regioisomer were obtained as well. The formation of the debenzoylated product was also observed in other cases

Table 2 Scope and limitations of the ruthenium-catalysed C–H bond benzoxylation of *N*-arylisoindolinones **1**



[a] 19% of debenzoylated product **2dd** was isolated. [b] The minor *ortho*-regioisomer is not represented. [c] 21% of debenzoylated product was isolated.

(*vide infra* for discussion). Isoindolinone bearing an *ortho*-tolyl substituent attached to the nitrogen atom was unreactive under catalytic conditions (**2h**), indicating that the sterically hindered substrates are significantly less reactive than the unhindered substrates. Benzoic acids containing different *para*- and *meta*-substituted functional groups as useful as fluoride, chloride, bromide, iodide, trifluoromethyl, nitro and methoxy were reactive, and they provided the corresponding products **2i–2p** in 50%–73% yields. Nitrile-substituted isoindolinones, however, did not afford the corresponding product **2q**. On the other hand, *ortho*-substituted benzoic acids afforded the corresponding products with yields depending on the steric bulk of the substituent as shown in the synthesis of the fluoro derivative **2r** in 63% yield and the chloro derivative **2s** in 36% yield. Polycyclic aromatic hydrocarbon fragments such as naphthalene were also compatible with the catalysis leading to **2t** in 62% yield. Gratifyingly, heteroaromatic-containing carboxylic acids were also compatible with the reaction. Thiophene-containing **2u** and furan-containing **2v** were obtained in 38% and 77% yields, respectively. In the latter case, 21% of debenzoylated alcohol product was also isolated. We also noted that the catalysis was not compatible with oxidation-sensitive formyl groups.



Additionally, the molecular structures of **2a**, **2b**, **2c**, **2o**, and **2t** were unambiguously confirmed by single-crystal X-ray diffraction (SCXRD) studies which further supported the regio- and site-selectivity of the reaction (Fig. 2). Unfortunately, carboxylic acids featuring nitrile, dimethylamino, pyridine and pyrrole functional groups were not viable for this reaction, in analogy to the reactions reported before for substrates with strong nitrogen-containing directing groups.[13] Neither acrylic acid nor trifluoroacetic acid formed any product in our case.

In general, trace amounts of the starting material remained once the reaction was stopped. However, during the purification of **2d** and **2u**, small amounts of debenzoylated product were observed. This feature was observed in another set of carboxylic acids as coupling partners in which no acyloxylated products were detected so far and debenzoylated phenol **3** was the only product that was formed (eqn (1)). For example, *N*-phenylisoindolinone **1a** reacted under the standard reaction conditions with *para*-toluic acid, *para*-methoxybenzoic acid, *ortho*-toluic acid, phenylacetic acid and acetic acid, respect-

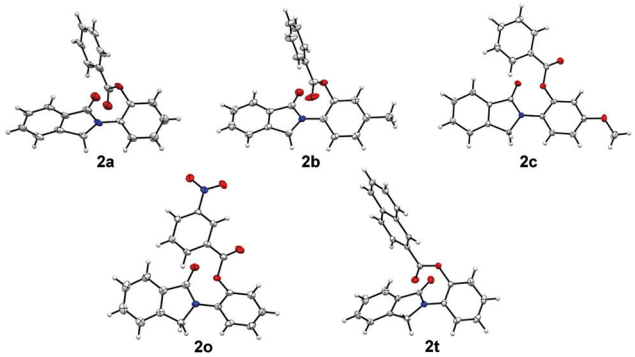
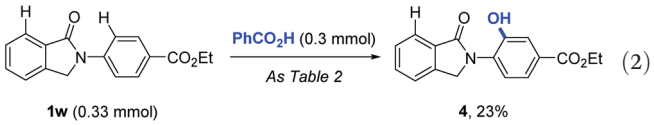


Fig. 2 SCXRD molecular structures of **2a**, **2b**, **2c**, **2o** and **2t**.

ively, leading exclusively to phenol **3** in variable yields (eqn (1)). The same behaviour was identified when using ester substituted isoindolinone **1w**, which afforded **4** in 23% yield besides unreacted starting material **1w** (eqn (2)). Although we do not have any reason to explain such findings, it can be suggested that the corresponding benzoxylated products that transiently form are rather unstable under the conditions used in the catalysis and they are readily hydrolysed.

(1)



(2)

Next, we evaluated whether the carbonyl group of the isoindolinone fragment **1** can behave as a weak coordinating group to ruthenium during the reaction. For that, we performed a control experiment with a substrate lacking any carbonyl group, isoindoline **5**. With this substrate no benzoxylated product was observed under the reaction conditions employed in the catalysis (eqn (3)). The same observations were made using cyclic imides **6** and **7** as substrates, respectively (eqn (4) and (5)).[14] The lack of reactivity in these cases likely arises from the rigidity and/or bulkiness of the directing groups that prevent the accommodation of catalytically productive ruthenacycle intermediates.

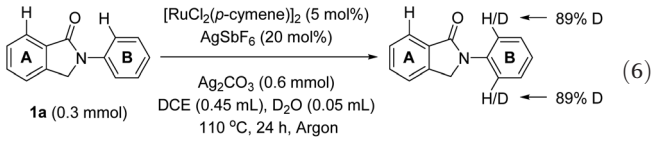
(3)

(4)

(5)

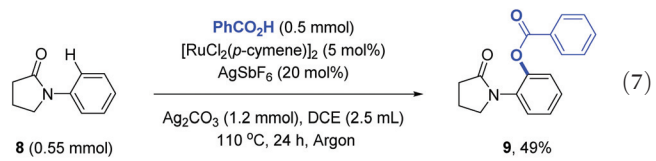
To further verify that the carbonyl group in **1** plays a key role in the C–H bond activation step, we performed the ruthenium-based catalysis in the absence of benzoic acid using a mixture of solvents DCE : D_2O (eqn (6)). Under these reaction conditions, 89% deuterium was incorporated into both the *ortho* C–H bonds of the phenyl ring **B**. This indicates the ease and reversibility of the C–H bond activation step in the presence of the ruthenium catalyst.

Based on the above-described data and previous reports,[10,13,15] a catalytic cycle is proposed in Scheme 1. First, chloride-free carboxylate-containing ruthenium species are formed, which coordinate to the carbonyl group of the isoindolinone core (**I**). Then, base-assisted C–H bond metalation gives rise to a six-membered ruthenacycle intermediate **II**. Due to the large amount of carboxylic acid in the reaction media, intermediate **III** is formed which undergoes reductive elimination towards product formation and oxidation of the metal centre to regenerate ruthenium(II) species.

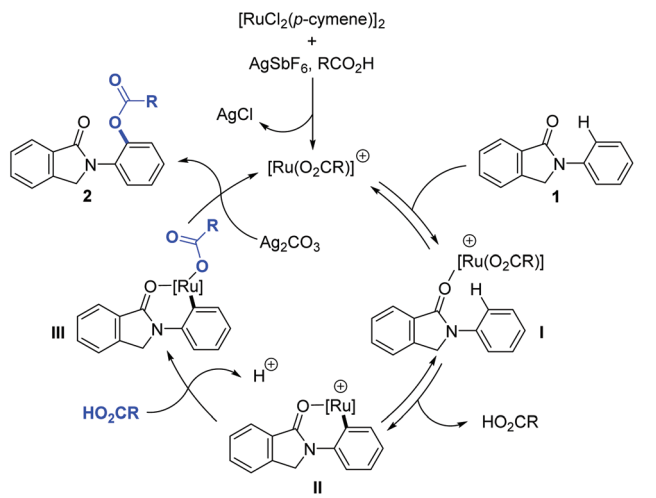


(6)

Finally, to investigate whether other cyclic amides can act as weak directing groups for this transformation, we performed the catalysis with *N*-phenylpyrrolidinone (**8**) as the substrate (eqn (7)). Under our standard reaction conditions, the ruthenium-catalyzed benzoxylation gave rise to the *ortho*-substituted product **9** in 49% isolated yield (56% conversion of **8**) with no evidence of bis-functionalization. For this type of transformation, the pyrrolidinone motif seems not to be an appropriate directing group as the isoindolinone motif. Probably, subtle stereoelectronic properties associated with each directing group are responsible for these observations.



(7)



Scheme 1 Proposed catalytic cycle for the ruthenium-catalysed C–H bond benzoxylation of *N*-arylisoindolinones **1**.

Conclusions

In summary, we have developed an efficient benzoxylation reaction to selectively form unprecedented C–O bonds within the isoindolinone core. The catalysis tolerates a large number of functional groups at different positions within both coupling partners. This ruthenium(II) catalysis constitutes an example of site-selective transformation where the combination of the weak directing group (cyclic amide) with the ruthenium catalyst enables the discrimination between two aromatic C–H bonds having comparable bond dissociation energies. Moreover, mono-functionalized products are exclusively obtained for this C–O bond-forming reaction. Overall, this methodology highlights the uniqueness of ruthenium catalysts enabling the formation of six-membered ring intermediates throughout the catalysis even with very weak directing groups.

Experimental

All reagents were obtained from commercial sources and used as supplied. All reactions were carried out in flame-dried glassware under an argon atmosphere unless otherwise noted. Catalytic experiments were performed in Schlenk-type flasks under an argon atmosphere unless otherwise noted. Organic solutions were concentrated under reduced pressure using a rotary evaporator. Thin-layer chromatography (TLC) was carried out on 0.25 mm Merck silica gel (60-F254). Flash column chromatography was performed using silica gel Silica 60 M, 0.04–0.063 mm. Technical grade petroleum ether (40–60), *n*-heptane and ethyl acetate were used for column chromatography. $CDCl_3$ was stored under nitrogen over molecular sieves. NMR spectra were recorded on an AVANCE III 400 spectrometer. ^{1}H NMR spectra were referenced to the residual protiated solvent (δ = 7.26 ppm for $CDCl_3$, δ = 2.50 ppm for DMSO-d_6 and δ = 2.05 ppm for acetone-d_6) and ^{13}C chemical shifts are reported relative to deuterated solvents (δ = 77.0 ppm for $CDCl_3$, δ = 39.5 ppm for DMSO-d_6 and δ = 29.8 ppm for acetone-d_6) [Note: acetone-d_6 contains traces of water at *ca.* 3 ppm]. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet, and br. for broad. GC-MS analyses were performed with a GCMS-QP2010S (Shimadzu) instrument with a GC-2010 equipped with a 30 m capillary column (Supelco, SLBTM-5 ms, fused silica capillary column, 30 m × 0.25 mm × 0.25 mm film thickness), which was used with helium as the vector gas. The following GC conditions were used: initial temperature 80 °C for 2 minutes, then rate 20 °C min^{-1} until 280 °C and 280 °C for 28 minutes. HRMS were recorded on a Waters Q-Tof 2 mass spectrometer at the corresponding facilities of the CRMPO, Centre Régional de Mesures Physiques de l'Ouest, Université de Rennes 1.

Synthesis and characterization of *N*-arylisoindolinone substrates (1)

A mixture of 2-formylbenzoic acid (5.0 mmol, 1 equiv.), aniline derivative (6.0 mmol, 1.2 equiv.), DABCO (10.0 mmol, 2 equiv.), HCOOH (1.25 mL), and $Pd(OAc)_2$ (0.25 mmol, 5 mol%) in 1,4-dioxane (5 mL) was heated at 80 °C for 3 h. After completion of the reaction, the mixture was cooled to room temperature, and diluted with DCM (50 mL). The solid was removed by filtration, and the filtrate was washed with water (50 mL) and brine (50 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/acetone = 5/1, v/v) to afford the desired product **1**.

2-Phenylisoindolin-1-one (1a). Starting from aniline in 98% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93 (d, J = 7.6 Hz, 1H), 7.89–7.86 (m, 2H), 7.62–7.58 (m, 1H), 7.53–7.49 (m, 2H), 7.43 (dd, J = 8.4, 7.2 Hz, 2H), 7.18 (dd, J = 7.2, 7.2 Hz, 1H), 4.87 (s, 2H) ppm. The spectral data match those previously reported.[16]

2-(*p*-Tolyl)isoindolin-1-one (1b). Starting from *p*-toluidine in 80% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92 (d, J = 7.2 Hz, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.59 (dd, J = 7.6, 7.6 Hz, 1H), 7.50 (dd, J = 6.8, 6.8 Hz, 2H), 7.24 (d, J = 8.4 Hz, 2H), 4.84 (s, 2H), 2.36 (s, 3H) ppm. The spectral data match those previously reported.[16]

2-(*p*-Methoxyphenyl)isoindolin-1-one (1c). Starting from *p*-anisidine in 62% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92 (d, J = 7.6 Hz, 1H), 7.74 (d, J = 9.2 Hz, 2H), 7.58 (dd, J = 7.6, 7.6 Hz, 1H), 7.52–7.48 (m, 2H), 6.97 (d, J = 9.2 Hz, 2H), 4.83 (s, 2H), 3.83 (s, 3H) ppm. The spectral data match those previously reported.[16]

2-(*p*-Chlorophenyl)isoindolin-1-one (1d). Starting from *p*-chloroaniline in 61% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92 (d, J = 7.2 Hz, 1H), 7.74 (dd, J = 9.2, 2.4 Hz, 2H), 7.61 (ddd, J = 7.6, 7.6, 1.2 Hz, 1H), 7.54–7.50 (m, 2H), 7.39 (dd, J = 9.2, 2.4 Hz, 2H), 4.84 (s, 2H) ppm. The spectral data match those previously reported.[16]

2-(*m*-Tolyl)isoindolin-1-one (1e). Starting from *m*-toluidine in 65% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93 (d, J = 6.8 Hz, 1H), 7.73 (s, 1H), 7.65–7.58 (m, 2H), 7.51 (dd, J = 7.2, 6.8 Hz, 2H), 7.32 (dd, J = 8.0, 8.0 Hz, 1H), 7.01 (d, J = 7.6 Hz, 1H), 4.86 (s, 2H), 2.41 (s, 3H) ppm. The spectral data match those previously reported.[16]

2-(*m*-Methoxyphenyl)isoindolin-1-one (1f). Starting from *m*-anisidine in 80% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93 (d, J = 7.2 Hz, 1H), 7.69 (dd, J = 2.0, 1.6 Hz, 1H), 7.62–7.58 (m, 1H), 7.51 (dd, J = 7.6, 7.6 Hz, 2H), 7.34–7.32 (m, 2H), 6.76–6.73 (m, 1H), 4.86 (s, 2H), 3.87 (s, 3H) ppm. The spectral data match those previously reported.[16]

2-(Benzo[*d*][1,3]dioxol-5-yl)isoindolin-1-one (1g). Starting from 3,4-(methylenedioxy)aniline in 58% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92 (d, J = 8.0 Hz, 1H), 7.61–7.57 (m, 2H), 7.51 (dd, J = 6.4, 5.6 Hz, 2H), 7.11 (dd, J = 8.4, 2.0 Hz, 1H), 6.85 (d, J = 8.4 Hz, 1H), 5.99 (s, 2H), 4.81 (s, 2H) ppm. The spectral data match those previously reported.[16]

2-(*o*-Tolyl)isoindolin-1-one (1h). Starting from *o*-toluidine in 68% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96 (d, J = 7.6 Hz, 1H), 7.61 (ddd, J = 7.2, 7.2, 1.2 Hz, 1H), 7.55–7.50 (m, 2H), 7.35–7.32 (m, 1H), 7.30–7.24 (m, 3H), 4.74 (s, 2H), 2.27

(s, 3H) ppm. The spectral data match those previously reported.[16]

4-(1-Oxoisoindolin-2-yl)benzonitrile (1q). Starting from 4-aminobenzonitrile in 64% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.06 (d, J = 9.2 Hz, 2H), 7.94 (d, J = 7.2 Hz, 1H), 7.71 (d, J = 8.8 Hz, 2H), 7.65 (dd, J = 7.6, 7.2 Hz, 1H), 7.54 (dd, J = 7.6, 7.2 Hz, 2H), 4.89 (s, 2H) ppm. The spectral data match those previously reported.[16]

Ethyl 4-(1-oxoisoindolin-2-yl)benzoate (1w). Starting from ethyl 4-aminobenzoate in 96% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.11 (d, J = 8.4 Hz, 2H), 7.99 (d, J = 8.8 Hz, 2H), 7.94 (d, J = 7.6 Hz, 1H), 7.63 (dd, J = 7.6, 7.6 Hz, 1H), 7.53 (dd, J = 7.6, 7.6 Hz, 2H), 4.90 (s, 2H), 4.39 (q, J = 7.2 Hz, 2H), 1.41 (t, J = 7.2 Hz, 3H) ppm. The spectral data match those previously reported.[16]

Synthesis and characterization of isoindoline (5)

1,2-Bis(bromomethyl)benzene (5.0 mmol, 1 equiv.), DIPEA (12.5 mmol, 2.5 eq.), and aniline (7.50 mmol, 1.5 equiv.) were dissolved in toluene (25 mL) and added to a sealed tube before vigorously stirring at 110 °C under an argon atmosphere. The resulting mixture was cooled down to room temperature and extracted with ethyl acetate (3 × 10 mL). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The crude product was purified by column chromatography (petroleum ether) to obtain the desired product **5** (89% isolated yield) as a light yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38–7.31 (m, 6H), 6.78 (dd, J = 7.6, 7.6 Hz, 1H), 6.71 (d, J = 7.6 Hz, 2H), 4.68 (s, 4H) ppm. The spectral data match those previously reported.[17]

Synthesis and characterization of *N*-phenylphthalimide (6)

Phthalic anhydride (5 mmol, 0.74 g, 1 equiv.) and aniline (5 mmol, 1 equiv.) were refluxed in acetic acid (30 mL) for 2–5 h. Once at room temperature, water was added and the solid was recovered by filtration. After drying under vacuum the desired phthalimide **6** (80% isolated yield) was obtained. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96 (dd, J = 5.6, 3.2 Hz, 2H), 7.80 (dd, J = 5.2, 3.2 Hz, 2H), 7.52 (dd, J = 7.6, 7.6 Hz, 2H), 7.34–7.27 (m, 3H) ppm. The spectral data match those previously reported.[18]

Synthesis and characterization of 2-phenylhexahydro-1*H*-isoindole-1,3(2*H*)-dione (7)

Hexahydrophthalic anhydride (10 mmol, 1.54 g, 1 equiv.), aniline (10 mmol, 1 equiv.) and THF (15 mL) were added to a 100 mL round bottom flask. The solution was stirred for 30 minutes at 40 °C. Removal of the solvent using a rotary evaporator gave the corresponding carboxylic acid-amide as a white solid. The white solid was then heated at 190 °C under argon for 4 h. The desired phthalimide **7** (86% isolated yield) was purified by silica gel column chromatography with a mixture of petroleum ether and ethyl acetate as the eluent. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48–7.44 (m, 2H), 7.39–7.35 (m, 1H), 7.30–7.27 (m, 2H), 3.06–3.00 (m, 2H), 1.95–1.85 (m, 4H), 1.53–1.50 (m, 4H) ppm. The spectral data match those previously reported.[19]

General procedure for the ruthenium-catalysed C–H bond benzoxylation reaction, and synthesis and characterization of products 2

$[RuCl_2(p\text{-cymene})]_2$ (5 mol%), $AgSbF_6$ (20 mol%), Ag_2CO_3 (0.2 mmol, 2.0 equiv.), *N*-arylisoindolinone **1** (0.33 mmol, 1.1 equiv.) and the corresponding benzoic acid derivative (0.3 mmol, 1.0 equiv.) were taken in a 15 mL Schlenk tube, which was equipped with a magnetic stirrer bar. 1,2-Dichloroethane (1.5 mL) was added to the Schlenk tube *via* a syringe, and the reaction mixture was degassed with argon three times. The reaction mixture was allowed to stir at 110 °C for 24 h. After being cooled to ambient temperature, the reaction mixture was diluted with DCM and then filtered through Celite. After evaporation of the solvent *in vacuo*, the crude product was purified by column chromatography on silica gel (*n*-heptane/EtOAc: 10 : 1 to 5 : 1, v/v) to give the desired product **2**.

2-(1-Oxoisoindolin-2-yl)phenyl benzoate (2a). 73.1 mg (74% yield), white solid, mp: 139–141 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.08 (d, J = 8.0 Hz, 2H), 7.85 (d, J = 7.6 Hz, 1H), 7.56–7.49 (m, 3H), 7.46–7.36 (m, 7H), 4.76 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.7, 164.9, 146.8, 141.7, 133.8, 132.0, 131.9, 131.0, 130.3, 129.0, 128.73, 128.66, 128.33, 128.27, 126.9, 124.3, 124.0, 122.9, 52.5 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{21}H_{15}NO_3Na$ 352.0944, found 352.0948 (1 ppm).

5-Methyl-2-(1-oxoisoindolin-2-yl)phenyl benzoate (2b). 50.7 mg (49% yield), white solid, mp: 158–160 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.07 (d, J = 7.2 Hz, 2H), 7.84 (d, J = 7.6 Hz, 1H), 7.55–7.49 (m, 2H), 7.43–7.35 (m, 5H), 7.18 (d, J = 7.6 Hz, 2H), 4.72 (s, 2H), 2.42 (s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.8, 165.0, 146.6, 141.7, 139.2, 133.7, 132.0, 131.8, 130.3, 129.0, 128.6, 128.2, 128.0, 127.6, 124.3, 124.2, 122.8, 52.5, 21.3 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{22}H_{17}NO_3Na$ 366.1101, found 366.1104 (1 ppm).

5-Methoxy-2-(1-oxoisoindolin-2-yl)phenyl benzoate (2c). 60.4 mg (56% yield), white solid, mp: 111–113 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.06 (d, J = 8.0 Hz, 2H), 7.83 (d, J = 7.6 Hz, 1H), 7.54–7.48 (m, 2H), 7.43–7.34 (m, 5H), 6.91 (d, J = 7.6 Hz, 2H), 4.70 (s, 2H), 3.84 (s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.9, 164.8, 159.8, 147.8, 141.7, 133.8, 132.0, 131.7, 130.3, 129.0, 128.9, 128.6, 128.2, 124.2, 123.6, 122.8, 112.8, 109.2, 55.8, 52.7 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{22}H_{17}NO_4Na$ 382.1050, found 382.1054 (1 ppm).

5-Chloro-2-(1-oxoisoindolin-2-yl)phenyl benzoate (2d). 38.1 mg (35% yield), yellow solid, mp: <50 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.06 (d, J = 7.6 Hz, 2H), 7.84 (d, J = 7.6 Hz, 1H), 7.58–7.52 (m, 2H), 7.46–7.35 (m, 7H), 4.74 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.8, 164.5, 147.1, 141.6, 134.1, 133.7, 132.1, 131.7, 130.4, 129.8, 129.0, 128.8, 128.6, 128.4, 127.1, 124.5, 124.4, 122.9, 52.4 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{21}H_{14}NO_3{}^{35}ClNa$ 386.0554, found 386.0555 (0 ppm).

2-(4-Chloro-2-hydroxyphenyl)isoindolin-1-one (2dd). 14.8 mg (19% yield), white solid, mp: 243–245 °C. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.31 (br, 1H), 7.77 (d, J = 7.6 Hz, 1H), 7.69–7.63 (m, 2H), 7.54 (ddd, J = 7.6, 7.6, 1.6 Hz, 1H), 7.36 (d, J = 8.4 Hz, 1H), 7.01 (d, J = 2.4 Hz, 1H), 6.95 (dd, J = 8.4, 2.4 Hz, 1H), 4.82 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6): δ = 167.1, 154.0, 142.4, 132.0, 131.8, 130.2, 127.9, 124.6, 123.4, 123.2, 119.1, 116.4, 51.5 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{14}H_{10}NO_2{}^{35}$ClNa 282.0292, found 282.0291 (0 ppm).

4-Methyl-2-(1-oxoisoindolin-2-yl)phenyl 4-chlorobenzoate (2e). 70.4 mg (62% yield), yellow solid, mp: <50 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.99 (d, J = 8.0 Hz, 2H), 7.82 (d, J = 7.6 Hz, 1H), 7.52 (dd, J = 7.6, 7.2 Hz, 1H), 7.44–7.40 (m, 2H), 7.34 (d, J = 8.0 Hz, 2H), 7.27 (s, 1H), 7.25–7.20 (m, 2H), 4.73 (s, 2H), 2.40 (s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.5, 164.2, 144.3, 141.6, 140.2, 136.9, 131.9, 131.6, 130.4, 129.3, 128.9, 128.6, 128.2, 127.5, 124.2, 123.3, 122.8, 52.4, 21.0 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{22}H_{16}NO_3{}^{35}$ClNa 400.0711, found 400.0708 (1 ppm).

4-Methoxy-2-(1-oxoisoindolin-2-yl)phenyl 4-chlorobenzoate (2f). 79.8 mg (68% yield), yellow solid, mp: <50 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.00 (d, J = 8.4 Hz, 2H), 7.83 (d, J = 7.6 Hz, 1H), 7.54 (dd, J = 7.6, 7.2 Hz, 1H), 7.44 (dd, J = 8.8, 8.0 Hz, 2H), 7.35 (d, J = 8.4 Hz, 2H), 7.27 (d, J = 8.8 Hz, 1H), 7.02 (s, 1H), 6.96 (d, J = 8.4 Hz, 1H), 4.75 (s, 2H), 3.83 (s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.5, 164.3, 157.9, 141.6, 140.1, 139.9, 132.0, 131.8, 131.6, 131.4, 128.9, 128.3, 127.5, 124.22, 124.19, 122.9, 114.0, 113.2, 55.8, 52.4 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{22}H_{16}NO_4{}^{35}$ClNa 416.0660, found 416.0659 (0 ppm).

6-(1-Oxoisoindolin-2-yl)benzo[*d*][1,3]dioxol-5-yl 4-chlorobenzoate (2g). 77.8 mg (64% yield), yellow solid, mp: <50 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95 (d, J = 8.4 Hz, 2H), 7.82 (d, J = 7.2 Hz, 1H), 7.51 (dd, J = 7.6, 7.2 Hz, 1H), 7.44–7.38 (m, 2H), 7.32 (d, J = 8.4 Hz, 2H), 6.88 (s, 1H), 6.84 (s, 1H), 6.04 (s, 2H), 4.65 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.8, 164.2, 147.6, 146.0, 141.6, 141.2, 140.3, 131.9, 131.6, 129.0, 128.3, 127.3, 124.2, 123.7, 122.9, 107.8, 104.6, 102.4, 52.6 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{22}H_{14}NO_5{}^{35}$ClNa 430.0453, found 430.0458 (1 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 4-fluorobenzoate (2i). 58.7 mg (56% yield), white solid, mp: 125–127 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.09 (m, 2H), 7.82 (d, J = 7.6 Hz, 1H), 7.54–7.34 (m, 7H), 7.04 (dd, J = 8.4, 8.4 Hz, 2H), 4.74 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.5, 166.2 (d, J_{C-F} = 253.6 Hz), 163.8, 146.7, 141.7, 132.9 (d, J_{C-F} = 9.5 Hz), 131.9, 131.8, 130.9, 128.7, 128.3, 128.1, 126.9, 125.2 (d, J_{C-F} = 3.0 Hz), 124.2, 123.8, 122.9, 115.8 (d, J_{C-F} = 22.0 Hz), 52.4 ppm. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$): δ = −104.1 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{21}H_{14}NO_3FNa$ 370.0850, found 370.0850 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 4-chlorobenzoate (2j). 69.8 mg (64% yield), yellow solid, mp: 58–60 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.00 (d, J = 8.8 Hz, 2H), 7.83 (d, J = 7.6 Hz, 1H), 7.54 (dd, J = 7.6, 7.6 Hz, 1H), 7.49–7.35 (m, 8H), 4.75 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.6, 164.0, 146.7, 141.7, 140.3, 132.0, 131.9, 131.7, 130.9, 129.0, 128.8, 128.4, 128.2, 127.5, 127.0, 124.3, 123.8, 122.9, 52.5 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{21}H_{14}NO_3{}^{35}$ClNa 386.0554, found 386.0553 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 3-chlorobenzoate (2k). 70.3 mg (64% yield), yellow solid, mp: <50 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.02 (s, 1H), 7.96 (d, J = 7.6 Hz, 1H), 7.83 (d, J = 7.2 Hz, 1H), 7.55–7.35 (m, 8H), 7.32 (dd, J = 7.6, 7.6 Hz, 1H), 4.76 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.5, 163.6, 146.5, 141.6, 134.7, 133.7, 131.9, 131.8, 130.9, 130.8, 130.2, 129.9, 128.6, 128.33, 128.28, 128.1, 127.0, 124.2, 123.7, 122.9, 52.4 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{21}H_{14}NO_3{}^{35}$ClNa 386.0554, found 386.0555 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 4-bromobenzoate (2l). 79.9 mg (65% yield), yellow solid, mp: 54–56 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92 (d, J = 8.4 Hz, 2H), 7.83 (d, J = 7.6 Hz, 1H), 7.55–7.51 (m, 3H), 7.49–7.42 (m, 4H), 7.40–7.36 (m, 2H), 4.75 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.5, 164.2, 146.6, 141.7, 132.00, 131.98, 131.86, 131.76, 130.9, 129.1, 128.7, 128.4, 128.1, 128.0, 127.0, 124.3, 123.8, 122.9, 52.4 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{21}H_{14}NO_3{}^{79}$BrNa 430.0049, found 430.0050 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 4-iodobenzoate (2m). 68.5 mg (50% yield), yellow solid, mp: 61–63 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83 (d, J = 7.6 Hz, 1H), 7.78–7.72 (m, 4H), 7.53 (dd, J = 7.6, 7.6 Hz, 1H), 7.48–7.35 (m, 6H), 4.75 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.5, 164.4, 146.6, 141.6, 138.0, 132.0, 131.8, 131.6, 130.9, 128.7, 128.5, 128.3, 128.1, 127.0, 124.3, 123.8, 122.9, 101.9, 52.4 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{21}H_{14}NO_3INa$ 477.9911, found 477.9913 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 4-(trifluoromethyl)benzoate (2n). 84.6 mg (71% yield), yellow solid, mp: <50 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.18 (d, J = 8.0 Hz, 2H), 7.81 (d, J = 8.0 Hz, 1H), 7.65 (d, J = 8.0 Hz, 2H), 7.54 (ddd, J = 7.6, 7.6, 1.2 Hz, 1H), 7.49–7.37 (m, 6H), 4.77 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.4, 163.7, 146.5, 141.7, 135.0 (q, J_{C-F} = 32.5 Hz), 132.3, 132.0, 131.8, 130.9, 130.7, 128.7, 128.4, 128.0, 127.1, 125.6 (q, J_{C-F} = 3.7 Hz), 124.2, 123.8, 123.6 (q, J_{C-F} = 270.1 Hz), 122.9, 52.4 ppm. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$): δ = −63.2 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{22}H_{14}NO_3F_3Na$ 420.0818, found 420.0820 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 3-nitrobenzoate (2o). 82.4 mg (73% yield), yellow solid, mp: 111–113 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.82 (s, 1H), 8.40 (d, J = 8.0 Hz, 1H), 8.36 (dd, J = 8.0, 8.0 Hz, 1H), 7.76 (d, J = 7.6 Hz, 1H), 7.60 (dd, J = 8.0, 8.0 Hz, 1H), 7.53 (dd, J = 7.6, 7.6 Hz, 1H), 7.48–7.37 (m, 6H), 4.80 (s, 2H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 167.3, 162.7, 148.3, 146.2, 141.6, 135.8, 132.0, 131.7, 130.9, 130.8, 130.0, 128.6, 128.3, 128.0, 127.8, 127.2, 125.1, 124.1, 123.7, 123.0, 52.4 ppm. HRMS (ESI) calcd for [M + Na]$^+$ $C_{21}H_{14}N_2O_5Na$ 397.0795, found 397.0799 (1 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 3-methoxybenzoate (2p). 57.5 mg (53% yield), white solid, mp: 142–144 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85 (d, J = 7.6 Hz, 1H), 7.68 (d, J = 7.6 Hz, 1H), 7.58 (s, 1H), 7.55–7.49 (m, 2H), 7.45–7.35 (m, 5H), 7.29 (dd, J = 8.0, 8.0 Hz, 1H), 7.08 (dd, J = 8.0, 2.4 Hz, 1H), 4.76 (s, 2H), 3.76

(s, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 167.6, 164.7, 159.7, 146.8, 141.7, 132.0, 131.9, 130.9, 130.3, 129.6, 128.7, 128.3, 128.2, 126.8, 124.2, 123.9, 122.9, 122.7, 120.8, 114.2, 55.5, 52.5 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{22}H_{17}NO_4Na$ 382.1050, found 382.1051 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 2-fluorobenzoate (2r). 65.6 mg (63% yield), white solid, mp: 88–90 °C. ^1H NMR (400 MHz, $CDCl_3$): δ = 8.01 (dd, J = 7.6, 7.6 Hz, 1H), 7.85 (d, J = 7.6 Hz, 1H), 7.56–7.35 (m, 8H), 7.15 (dd, J = 7.6, 7.6 Hz, 1H), 7.06 (dd, J = 8.4, 8.4 Hz, 1H), 4.80 (s, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 167.6, 166.2 (d, J_{C-F} = 259.9 Hz), 162.0 (d, J_{C-F} = 3.9 Hz), 146.5, 141.8, 135.3 (d, J_{C-F} = 9.0 Hz), 132.6, 132.0, 131.9, 130.9, 128.6, 128.3, 128.2, 127.0, 124.24 (d, J_{C-F} = 4.1 Hz), 124.22, 123.9, 122.9, 117.6 (d, J_{C-F} = 9.2 Hz), 117.1 (d, J_{C-F} = 21.8 Hz), 52.4 ppm. $^{19}F\{^1H\}$ NMR (376 MHz, $CDCl_3$): δ = −108.8 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{21}H_{14}NO_3FNa$ 370.0850, found 370.0852 (1 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 2-chlorobenzoate (2s). 38.9 mg (36% yield), yellow solid, mp: 110–112 °C. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.95 (d, J = 7.6 Hz, 1H), 7.87 (d, J = 7.6 Hz, 1H), 7.55 (dd, J = 7.6, 7.2 Hz, 1H), 7.48–7.37 (m, 8H), 7.29–7.23 (m, 1H), 4.80 (s, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 167.6, 163.4, 146.7, 141.9, 134.3, 133.3, 133.2, 132.1, 132.04, 131.96, 131.2, 131.0, 128.89, 128.86, 128.3, 127.1, 126.9, 124.3, 123.8, 123.0, 52.5 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{21}H_{14}NO_3{}^{35}ClNa$ 386.0554, found 386.0555 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl 2-naphthoate (2t). 70.3 mg (62% yield), yellow solid, mp: <50 °C. ^1H NMR (400 MHz, $CDCl_3$): δ = 8.67 (s, 1H), 8.07 (d, J = 8.4 Hz, 1H), 7.88–7.82 (m, 4H), 7.60–7.37 (m, 9H), 4.80 (s, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 167.8, 165.0, 146.9, 141.7, 135.9, 132.5, 132.2, 131.94, 131.89, 131.0, 129.6, 128.8, 128.5, 128.4, 128.2, 127.8, 126.8, 126.2, 125.4, 124.3, 124.0, 122.9, 52.5 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{25}H_{17}NO_3Na$ 402.1101, found 402.1099 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl thiophene-2-carboxylate (2u). 73.8 mg (77% yield), yellow solid, mp: <50 °C. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.86 (d, J = 7.6 Hz, 1H), 7.57–7.36 (m, 8H), 7.29 (d, J = 7.6 Hz, 1H), 6.47 (dd, J = 2.8, 2.0 Hz, 1H), 4.79 (s, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 167.6, 156.3, 147.5, 145.9, 143.4, 141.8, 131.9, 130.9, 128.5, 128.24, 128.20, 126.9, 124.2, 123.7, 122.9, 120.1, 112.2, 52.4 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{19}H_{13}NO_3SNa$ 358.0508, found 358.0510 (0 ppm).

2-(1-Oxoisoindolin-2-yl)phenyl furan-2-carboxylate (2v). 73.8 mg (77% yield), yellow solid, mp: <50 °C. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.86 (d, J = 7.6 Hz, 1H), 7.57–7.36 (m, 8H), 7.29 (d, J = 7.6 Hz, 1H), 6.47 (dd, J = 2.8, 2.0 Hz, 1H), 4.79 (s, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 167.6, 156.3, 147.5, 145.9, 143.4, 141.8, 131.9, 130.9, 128.5, 128.24, 128.20, 126.9, 124.2, 123.7, 122.9, 120.1, 112.2, 52.4 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{19}H_{13}NO_4Na$ 392.0737, found 392.0740 (1 ppm).

2-(2-Hydroxyphenyl)isoindolin-1-one (3)

40.9 mg (63% yield), colourless solid, ^1H NMR (400 MHz, $CDCl_3$): δ = 8.82 (br, 1H), 7.93 (d, J = 7.6 Hz, 1H), 7.64 (dd, J = 7.6, 7.2 Hz, 1H), 7.56–7.52 (m, 2H), 7.25–7.21 (m, 2H), 7.14 (dd, J = 8.4, 1.6 Hz, 1H), 6.99 (ddd, J = 8.0, 8.0, 1.6 Hz, 1H), 4.96 (s, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 169.1, 150.9, 141.9, 132.6, 131.8, 128.8, 128.1, 127.5, 124.4, 122.8, 122.5, 121.3, 121.1, 52.7 ppm. MS (EI): m/z = 225 (M^+, 84), 196 (30), 132 (100), 120 (45). The spectral data match those previously reported.[11]

Ethyl 3-hydroxy-4-(1-oxoisoindolin-2-yl)benzoate (4)

20.1 mg (23% yield), white solid, mp: 205–207 °C. ^1H NMR (400 MHz, $CDCl_3$): δ = 9.09 (br, 1H), 7.94 (d, J = 7.6 Hz, 1H), 7.79 (d, J = 1.6 Hz, 1H), 7.68–7.64 (m, 2H), 7.58–7.54 (m, 2H), 7.27 (d, J = 8.0 Hz, 1H), 5.00 (s, 2H), 4.37 (q, J = 7.2 Hz, 2H), 1.40 (t, J = 7.2 Hz, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 169.4, 166.0, 150.5, 141.9, 133.0, 131.5, 130.0, 129.0, 124.6, 122.9, 122.6, 122.3, 122.0, 61.2, 52.8, 14.4 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{17}H_{15}NO_4Na$ 320.0893, found 320.0888 (2 ppm).

2-(2-Oxopyrrolidin-1-yl)phenyl benzoate (9)

68.8 mg (49% yield), brown oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 8.18 (d, J = 7.6 Hz, 2H), 7.62 (dd, J = 7.6, 7.2 Hz, 1H), 7.49 (dd, J = 7.6, 7.6 Hz, 2H), 7.38–7.27 (m, 4H), 3.71 (t, J = 7.2 Hz, 2H), 2.36 (t, J = 8.0 Hz, 2H), 2.05–1.97 (m, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 174.3, 164.7, 146.4, 133.8, 131.3, 130.2, 129.0, 128.7, 128.4, 127.5, 126.7, 123.7, 50.1, 31.1, 19.3 ppm. HRMS (ESI) calcd for $[M + Na]^+$ $C_{17}H_{15}NO_3Na$ 304.0944, found 309.0946 (1 ppm).

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

CNRS, Université de Rennes 1, Rennes Metropole, China Scholarship Council (PhD grant to Y.-C. Y.), Agence Nationale de la Recherche (ANR-JCJC) and the COST Action CA15106 (CHAOS) are acknowledged.

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