

Cite this: *RSC Adv.*, 2018, 8, 25292

# Palladium-catalyzed direct C(sp<sup>3</sup>)-H arylation of indole-3-ones with aryl halides: a novel and efficient method for the synthesis of nucleophilic 2-monoarylated indole-3-ones†

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Accepted 9th July 2018

DOI: 10.1039/c8ra04807j

rsc.li/rsc-advances

A novel and efficient method for the synthesis of nucleophilic 2-monoarylated indole-3-ones via palladium-catalyzed direct C(sp<sup>3</sup>)-H arylation of indole-3-ones with aryl halides has been developed. Various 2-monoarylated indole-3-ones were readily obtained with yields up to 95%. As a class of important nucleophilic intermediates, 2-monoarylated indole-3-ones can be used for the construction of C2-quaternary indolin-3-one skeletons.

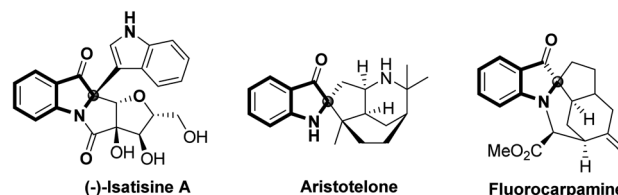
2,2-Disubstituted indole-3-ones (trivially known as pseudo-indoxyl) are privileged core heterocyclic structural motifs that occur in a great number of biologically active natural products.

(Fig. 1, (–)Isatisine A,<sup>1a-c</sup> Aristotelone,<sup>1e</sup> Fluorocarpamine<sup>1d</sup>).<sup>1</sup> In addition, they can be used as a key synthetic intermediate in the synthesis of many natural products (Fig. 1, Hinckdentine A,<sup>2a-c</sup> Lapidilectine B<sup>2d,e</sup> and (–)-Trigonoliimine C<sup>2f-j</sup>).<sup>2</sup> Owing to its interesting structure and biological activity, the pseudo-indoxyl scaffold has attracted extensive attention from both synthetic and medicinal chemists. Numerous elegant synthetic protocols have been developed for the construction of the 2,2-disubstituted indole-3-one scaffold.<sup>2c,3,4</sup> Recently, nucleophilic indole-3-ones have been demonstrated to be very reliable building blocks for the enantioselective or racemic construction of C2-quaternary indolin-3-one skeletons.<sup>3d,4</sup> Among these significant advances, one of the key substrates was nucleophilic 2-monoarylated indole-3-ones (Scheme 1a).<sup>3d,4a</sup>

So far, much of the effort has been focused on the synthesis of C2-quaternary indolin-3-one skeletons,<sup>2c,3,4</sup> however, the routes for the synthesis of nucleophilic 2-monoarylated indole-3-ones have been challenging and are rare (Scheme 1a).<sup>3d,4a,5</sup>

Although some useful methods were developed via Baeyer-Villiger oxidation of C-3 phenyl substituted indole derivatives (A and B) and direct arylation of indolin-3-one 1a with aryllead triacetate, it should be noted that the application of these methods is restricted by the limited number of available substrates and the low yields (Scheme 1b).<sup>5</sup> In 2015, a method for the potassium *tert*-butoxide mediated direct C2-arylation of indolin-3-ones 1 was reported by Liu *et al.*, however, diaryliodonium salts were required as the arylating agents and lower yields (up to 70%) were obtained.<sup>3d</sup> In addition, the “Selective Problem” arose due to the use of unsymmetric diaryliodonium salts as the arylating agents and the mixtures of two C2-

### Selected natural products containing 2,2-disubstituted indole-3-one:



### Natural products synthesized with Pseudo-indoxyl as the key intermediate:

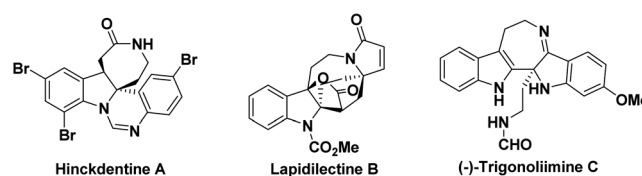


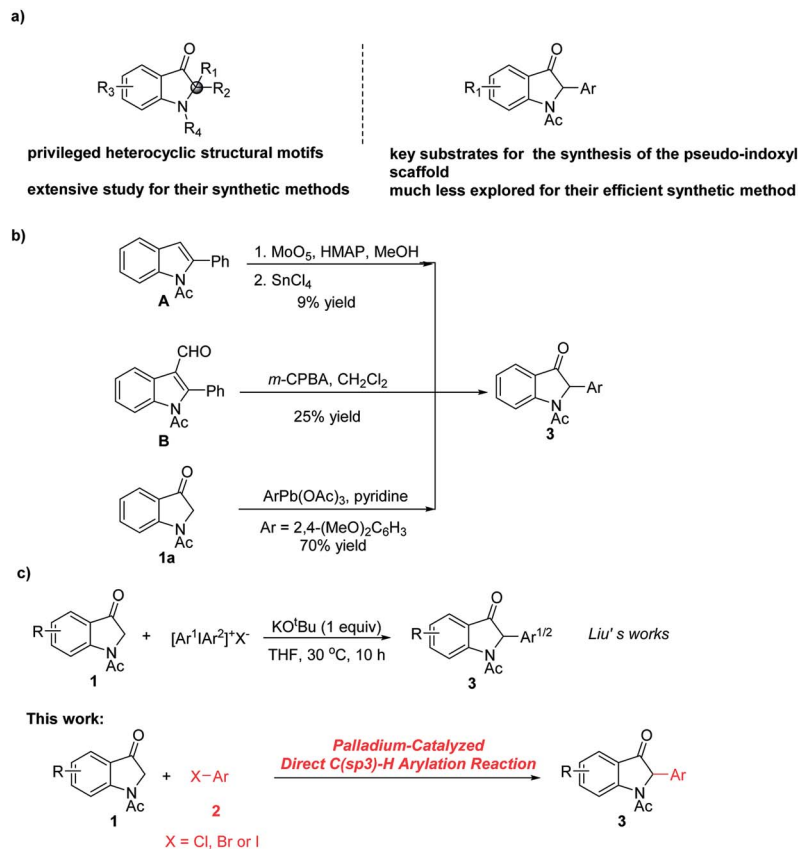
Fig. 1 Natural products containing the C2-quaternary indolin-3-ones fragment and representative natural products that were synthesized with using 2,2-disubstituted indole-3-one as the key intermediate.

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† Electronic supplementary information (ESI) available. See DOI: 10.1039/c8ra04807j





Scheme 1 Reported approaches toward the synthesis of C2-quaternary indolin-3-one skeletons and palladium-catalyzed direct  $\text{C}(\text{sp}^3)\text{-H}$  arylation reaction of indole-3-ones with aryl halides.

arylation products were produced (Scheme 1c).<sup>3d</sup> Therefore, more efficient methods for the synthesis of nucleophilic 2-monoarylated indole-3-ones are highly desired.

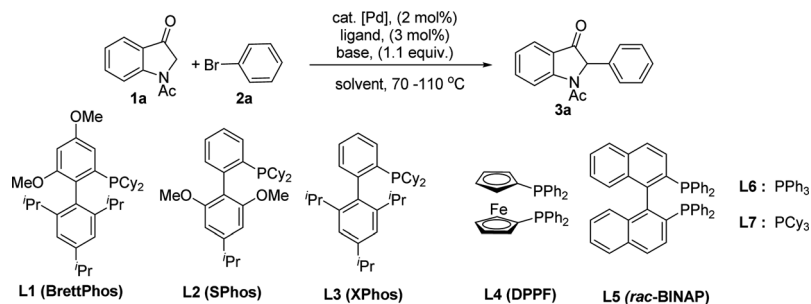
Palladium-catalyzed  $\alpha$ -arylation of carbonyl and related compounds has served as a powerful tool for the quick construction of C–C bonds.<sup>6</sup> Although palladium-catalyzed C-3 arylation of 2-oxindole has been reported by Willis *et al.*,<sup>6c</sup> palladium-catalyzed direct  $\text{C}(\text{sp}^3)\text{-H}$  arylation of indole-3-ones with aryl halides remains unexplored.<sup>3d</sup> With our ongoing interest in the study of palladium-catalyzed coupling reactions<sup>6d,7</sup> and indolin-3-one chemistry,<sup>4d,e</sup> we developed an efficient procedure for this transformation for the synthesis of nucleophilic 2-monoarylated indole-3-ones **3** (Scheme 1c).

1-Acetylindolin-3-one **1a** and bromobenzene **2a** were used as the model substrate for the initial study and the results are summarized in Table 1. It was found that 1-acetylindolin-3-one **1a** and bromobenzene **2a** reacted in the presence of the Brettphos ligand **L1** (3 mol%),  $\text{K}_2\text{CO}_3$  (1.1 equiv.) and  $\text{Pd}(\text{dba})_2$  (2 mol%) in THF under a high pure nitrogen atmosphere at 70 °C to furnish the desired product **3a** with a low yield (Table 1, entry 1). Various ligands were then screened and Xphos ligand **L3** was found to be the best ligand for this  $\alpha$ -arylation reaction (Table 1, entry 3). For ligands **L4** and **L5**, no desired product was obtained (Table 1, entries 4 and 5). For ligands **L2**, **L6** and **L7**, desired product **3a** was obtained albeit with lower yields (Table

1, entry 2, 6 and 7). Next,  $\text{Pd}(\text{OAc})_2$ ,  $\text{Pd}(\text{TFA})_2$ ,  $\text{PdCl}_2$  and  $\text{Pd}(\text{dba})_2$  were also tested for this  $\alpha$ -arylation reaction using  $\text{K}_2\text{CO}_3$  as the base in the presence of Xphos ligand **L3** (Table 1, entry 3, 8, 9 and 10), and  $\text{Pd}(\text{dba})_2$  was found to be the optimal choice (Table 1, entry 3). Finally, various bases such as  $\text{K}_2\text{CO}_3$ ,  $\text{KHMDs}$ ,  $^t\text{BuOK}$ ,  $^t\text{BuONa}$ ,  $\text{CsCO}_3$ ,  $\text{K}_3\text{PO}_4$  and  $\text{AcONa}$  were also tested (Table 1, entry 3, 12, 13, 14, 15 and 16), and  $\text{K}_2\text{CO}_3$  was found to be the most promising base (Table 1, entry 3). We then tested the effect of high boiling-point solvents on this  $\alpha$ -arylation process, some common solvents such as THF, toluene and dioxane were used, and THF was found to be the optimal choice (Table 1, entry 3). Finally, we examined the effect of different concentrations and reaction time on this  $\alpha$ -arylation process, and found this process could be finished in 14 hours in the presence of the  $\text{Pd}(\text{dba})_2$  (2 mol%) and Xphos ligand **L3** (3 mol%) under 2.0 ml THF. Accordingly,  $\text{Pd}(\text{dba})_2$  (2 mol%), Xphos ligand **L3** (3 mol%) and  $\text{K}_2\text{CO}_3$  (1.1 equiv.) with THF as the solvent under a high pure nitrogen atmosphere at 70 °C are the optimal conditions for this  $\alpha$ -arylation reaction of indole-3-ones with aryl halides.

With the reaction conditions optimized, we next investigated the substrate scopes of indole-3-ones **1** and aryl halides **2** (Table 2). In most cases, the reactions afforded the corresponding 2-monoarylated indole-3-one products **3a–f** with moderate to excellent yields (19–95%). First, we examined the reactions of 1-



Table 1 Optimization of Reaction Conditions<sup>a</sup>

Entry	Cat. [Pd]	Ligand	Base	Solvent	Yield <sup>b</sup> (%)
1	Pd(dba) <sub>2</sub>	L1	K <sub>2</sub> CO <sub>3</sub>	THF	<10
2	Pd(dba) <sub>2</sub>	L2	K <sub>2</sub> CO <sub>3</sub>	THF	26
3	Pd(dba) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	THF	87
4	Pd(dba) <sub>2</sub>	L4	K <sub>2</sub> CO <sub>3</sub>	THF	NR <sup>c</sup>
5	Pd(dba) <sub>2</sub>	L5	K <sub>2</sub> CO <sub>3</sub>	THF	NR <sup>c</sup>
6	Pd(dba) <sub>2</sub>	L6	K <sub>2</sub> CO <sub>3</sub>	THF	<10
7	Pd(dba) <sub>2</sub>	L7	K <sub>2</sub> CO <sub>3</sub>	THF	<10
8	Pd(OAc) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	THF	<10
9	PdCl <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	THF	<10
10	Pd(TFA) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	THF	24
11	Pd(dba) <sub>2</sub>	L3	KHMDS	THF	<10
12	Pd(dba) <sub>2</sub>	L3	<sup>t</sup> BuOK	THF	— <sup>d</sup>
13	Pd(dba) <sub>2</sub>	L3	CsCO <sub>3</sub>	THF	<10
14	Pd(dba) <sub>2</sub>	L3	K <sub>3</sub> PO <sub>4</sub>	THF	32
15	Pd(dba) <sub>2</sub>	L3	AcONa	THF	NR <sup>c</sup>
16	Pd(dba) <sub>2</sub>	L3	<sup>t</sup> BuONa	THF	— <sup>d</sup>
17	Pd(dba) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	Dioxane	53
18 <sup>i</sup>	Pd(dba) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	Toluene	24
19 <sup>f</sup>	Pd(dba) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	THF	80
20 <sup>g</sup>	Pd(dba) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	THF	73
21 <sup>h</sup>	Pd(dba) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	THF	66
22 <sup>j</sup>	Pd(dba) <sub>2</sub>	L3	K <sub>2</sub> CO <sub>3</sub>	THF	84

<sup>a</sup> Reactions performed on a 0.25 mmol scale using **1a** (1.0 equiv.) and **2a** (1.1 equiv.) in 2.0 ml of the solvent for 14 h, reaction performed at 70 °C under a high pure nitrogen atmosphere. <sup>b</sup> Isolated yield. <sup>c</sup> NR = no reaction. <sup>d</sup> Decomposition observed based on TLC. <sup>e</sup> 1.0 ml THF was used as the solvent. <sup>f</sup> 3.0 ml THF was used as the solvent. <sup>g</sup> Reaction time = 10 h. <sup>h</sup> Reaction performed at 100–110 °C and 1.0 ml solvent was used. <sup>i</sup> Reaction time = 18 h.

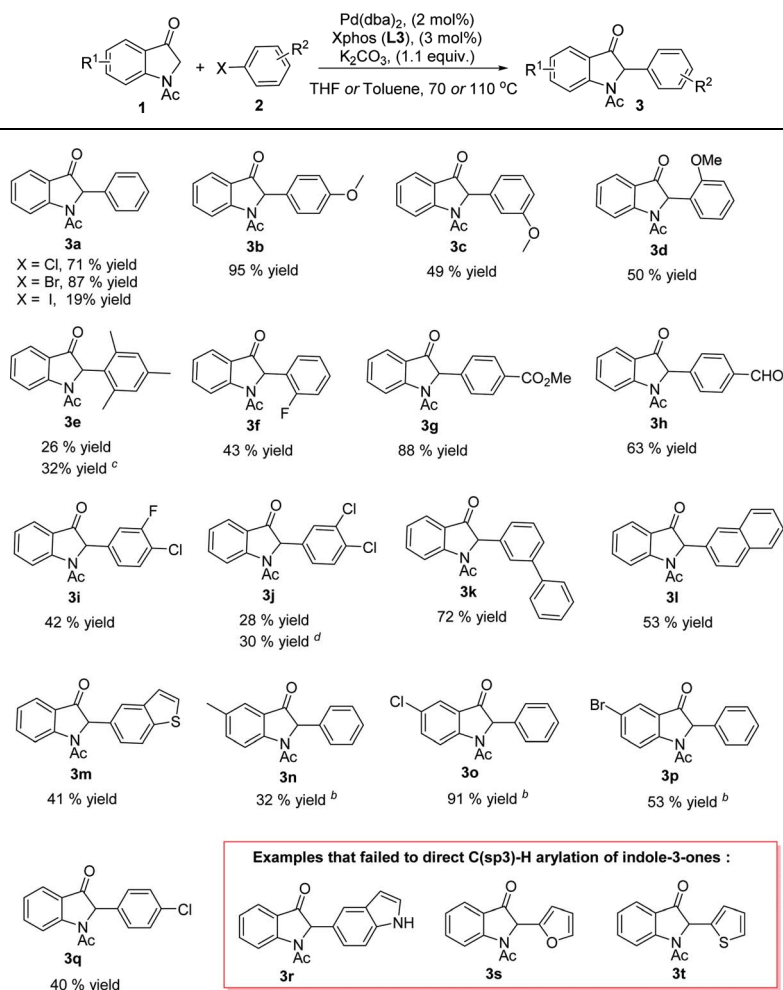
acetylidole-3-one **1a** with various substituted aryl halides **2**. Bromobenzene, chlorobenzene and iodobenzene were all effective substrates for the synthesis of **3a** and bromobenzene was the best substrate for the  $\alpha$ -arylation of indole-3-one **1a**. The structural variation of aryl bromides **2** could be well tolerated in this reaction irrespective of the electronic nature or the positions of the substituents on the aromatic ring. Compared with the electron-withdrawing aryl bromides (**3g–h**), the electron-donating aryl bromides (**3b**) gave higher yields. Generally speaking, the steric effect of aryl bromides decreased the yields of products (**3c–d** vs. **3b**) and **3e** was obtained in only 26% yield. When both bromo- and chloro-substituents were present in the arenes, selective reactions at the bromo positions were always observed (**3i–j**, **3q**). It is noteworthy that this  $\alpha$ -arylation process could also be successfully extended to others complex aryl bromides and corresponding desired products **3k–m** were obtained with 41–72% yields, however, the examples for 5-bromo-

1*H*-indole, 2-bromofuran and 2-bromothiophene are failed (**3r–t**). The variation on the indole-3-one scaffold could also be tolerated in this  $\alpha$ -arylation process and the desired products **3n–p** were afforded with 32–91% yields. Furthermore, when 5-bromo- and chloro-substituted of indole-3-ones were used, corresponding products **3o** and **3p** were also obtained.

To investigate the potential utility of this strategy, the large-scale synthesis of **3a** was also performed under the optimized conditions. The reaction proceeded smoothly to afford the corresponding 2-monoarylated indole-3-one **3a** product albeit with the actual yield decreased to 72% (Scheme 2). In addition, the 2-monoarylated indole-3-one **3** could be used as a kind of key nucleophilic substrate for the chiral or achiral synthesis of C2-quaternary indolin-3-one skeletons.<sup>3d,4a</sup>

As shown in Scheme 3, a possible reaction mechanism for this palladium-catalyzed  $\alpha$ -arylation of indole-3-ones was proposed based on the reported mechanisms of the similar



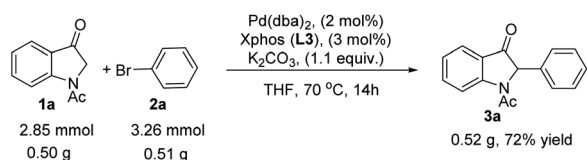
Table 2 Substrate scope of palladium-catalyzed direct C(sp<sup>3</sup>)-H arylation of indole-3-ones with aryl halides<sup>a</sup>

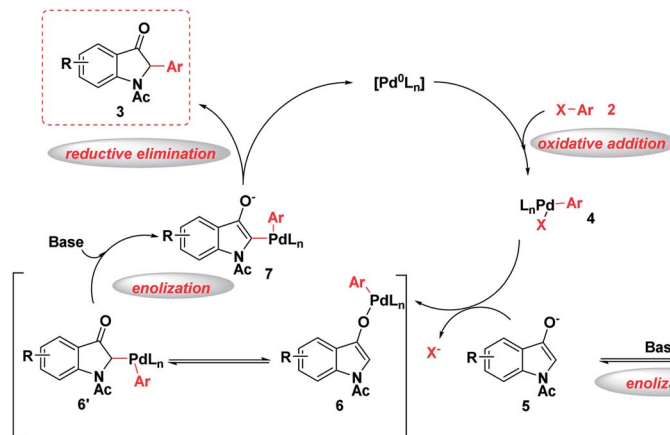
<sup>a</sup> Unless otherwise specified, all reactions were carried out with using **1** (0.25 mmol, 1.0 equiv.) and **2** (1.1 equiv.) in 2.0 mL of the THF for 14 h at 70 °C under an high pure nitrogen atmosphere and all the yields were isolated yield. <sup>b</sup> Reaction performed at 110 °C and 1.0 ml PhMe was used as the solvent. <sup>c</sup> Pd(dba)<sub>2</sub> (10 mol%) and Xphos (10 mol%) was used. <sup>d</sup> Aryl bromides (2.0 equiv.) and K<sub>2</sub>CO<sub>3</sub> (2.0 equiv.) was used.

palladium-catalyzed  $\alpha$ -arylation of carbonyl and related compounds with aryl halides.<sup>6</sup> The oxidative addition of a palladium complex into the C-X bond of aryl halides first occurs and the palladium complex intermediate **4** is formed. At the same time the enolate intermediate **5** is also produced by indole-3-one **1** in the presence of a base, which reacts with intermediate **4** to get the arylpalladium enolate complex **6** and

its isomerism intermediate **6'**. Finally, anionic palladium intermediate **7** is formed by above isomerism intermediate **6'** in the presence of a base,<sup>6e,f</sup> which then undergoes a reductive elimination to form the desired 2-monoarylated indole-3-one **3**, at the same time restores the original palladium catalyst and completes the catalytic cycle.

In conclusion, we have developed an efficient method for the synthesis of 2-monoarylated indole-3-ones *via* palladium-catalyzed direct C(sp<sup>3</sup>)-H arylation of indole-3-ones with aryl halides. The nucleophilic 2-monoarylated indole-3-ones were obtained in moderate to good yields (up to 95%). The products could be used as building blocks for the enantioselective or racemic synthesis of C2-quaternary indolin-3-one skeletons. Further investigation and application of the nucleophilic 2-monoarylated indole-3-one derivatives are ongoing in our laboratories.

Scheme 2 The large-scale synthesis of the product **3a**.



Scheme 3 A proposed mechanism for the palladium-catalyzed direct C(sp<sup>3</sup>)-H arylation of indole-3-ones with aryl halides.

## Conflicts of interest

There are no conflicts of interest to declare.

## Acknowledgements

We are grateful for financial support from the National Natural Science Foundation of China (grants. 21502030, 81660348, 21601039 and 81460523) and the PhD early development program of Guizhou Medical University (J[2014]017). Financial support from the Innovation Team of Nature Science Foundation of Department of Education of Guizhou Province (No. QJHRCTDZ[2015]57) is also acknowledged.

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