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Acknowledgements

We are grateful for financial support from the National Natural Science Foundation of China (No. 21372267, 21402150, 21572027, 21602023) and the Postdoctoral Research Grant (2016M602658) and Fundamental Research Funds for the Central Universities (10611201CDJXY460001) for financial support.

COMMUNICATION

Palladium-Catalyzed Oxidative Arylacetoxylation of Alkenes: Synthesis of Indole and Indoline Derivatives

Received 00th January 20xx, Accepted 00th January 20xx

DOI: 10.1039/x0xx00000x

www.rsc.org/

A method for oxidative arylacetoxylation of alkenes has been developed to synthesize indole and indoline derivatives from readily accessible substrates. The cinnamyl tethered anilines with picolinamide as a directing group provided 3-substituted indoles via intramolecular oxidative arylacetoxylation, and the 2-methyl substituted cinnamyl anilines furnished indoline derivatives with 3-position quaternary stereocenters in good to excellent yields via sequential intramolecular oxidative arylacetoxylation, hydrolysis and oxidation steps.

Metal catalyzed oxidative difunctionalization of alkenes is an emerging field which establishes multiple bonds in a single operation to facilitate the synthesis of organic molecules from simple starting materials without the requirement of prefunctionalizations. Intramolecular carboheterofunctionalization of unsaturated bonds of alkenes or alkynes proved to be an efficient synthetic strategy to access various heterocyclic natural products.¹ In recent years, transition-metal-catalyzed oxidative C(sp²)-H functionalization directed by a functional group has been recognized as an increasingly viable method to construct C-X (X = carbon or heteroatom) bonds.² In these reactions, directing groups are essential for acceleration and regioselective controls,³ and the intermolecular version has been under active investigation and proven to be highly fruitful.⁴ In contrast, the intramolecular oxidative arene/olefin coupling, especially with unactivated olefins, has been poorly studied.⁵ We envisioned that such an intramolecular reaction of electronrich arenes with unactivated olefins could provide an efficient synthesis of indole and indoline derivatives.

Many heterocycles including natural products possessing an indole core are important structural motifs as they display a

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diverse range of biological activities.⁶ As a result, effective methods for synthesizing indoles to access these classes of compounds have been actively investigated,⁷ and one of the most desirable strategy is via direct oxidative C-H functionalization. In particular, the construction of substituted indole derivatives via intramolecular oxidative functionalization is even more valuable.⁸ Similarly, indolines with a guaternary center at the 3-position are of considerable interest because of their remarkable prevalence in numerous natural products and biologically active compounds.⁹ To continue our research in palladium-catalyzed C-H functionalization,¹⁰ herein we report the development of a carbo-acetoxy functionalization method to efficiently synthesize indole and indoline derivatives employing a similar palladium catalytic system.

We began the investigation of the intramolecular carboacetoxy functionalization using anilide 1a as a model substrate, which contains a tethered cinnamyl moiety and a picolinamide (PA) as an easily removable directing group (Table 1). After screening various reaction conditions, we isolated the desired spiro-dihydroqunoline 2a in 68% yield employing the following catalytic system [Pd(OAc)₂ (0.10 equiv), PhI(OAc)₂ (2.50 equiv), 2-chloro-4-cyanopyridine (PyClCN, 0.40 equiv), 110 °C, 2 h, toluene], and the concentration of 1a in toluene was 0.075 mol/L (entry 1). We then investigated the role of each reactant by performing various control experiments. As expected, Pd(OAc)₂ and PhI(OAc)₂ played pivotal roles in the transformation, and no reaction was observed in the absence of them (entry 2). Various Pd(II) catalysts such as PdCl₂(PhCN)₂, PdCl₂(PPh₃)₂, or PdCl₂(dppf) were examined and found to be less effective than Pd(OAc)₂, providing the desired product in significantly lower yields (15-42%) (entries 3-5). Other common metal catalysts such as FeCl₃, RuCl₃, Sc(OTf)₃, Bi(OTf)₃, or In(OTf)₃ could not initiate this reaction (entry 6). Omitting PyCICN led to low yield, confirming its essential role in the reaction (entry 7). Use of various additives such as PhCN, 2,2- bipyridine, S-BINAP, or S-BINAL resulted in lower yields (entries 8-11), and varying the solvent or lowering the reaction temperature did not improve



DOI: 10.1039/C7CC06448A

⁺ Electronic Supplementary Information (ESI) available: CCDC 1562681, 1562684. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/x0xx00000x

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Table 1 Optimization of the arylacetoxylation reaction^a



entry	variation from the "standard" condition	yield (%)
1	none	68
2	without Pd(OAc) ₂ or PhI(OAc) ₂	0
3	$PdCl_2(PhCN)_2$ instead of $Pd(OAc)_2$	42
4	$PdCl_2(PPh_3)_2$ instead of $Pd(OAc)_2$	21
5	PdCl ₂ (dppf) instead of Pd(OAc) ₂	15
6	$FeCl_3$, RuCl_3, Sc(OTf)_3, Bi(OTf)_3 or In(OTf)_3 instead of Pd(OAc)_2	0
7	without PyCICN	48
8	PhCN instead of PyClCN	55
9	2,2-bipyridine instead of PyCICN	< 5
10	S-BINAP instead of PyCICN	< 5
11	S-BINAL instead of PyCICN	< 5
12	DCE instead of toluene	41
13	dioxane instead of toluene	25
14	CH ₃ CN instead of toluene	28
15	80 °C instead of 110 °C	45
16	PA replaced by Me	0
17	PA replaced by Ac	0
18	PA replaced by Ts	0

 $^{\mathrm{a}}\text{All}$ reactions were carried out on a 0.3 mmol scale. $^{\mathrm{b}}\text{Yield}$ was that of the isolated product

the yield (entries 12-15). The reaction did not occur if the PA group was replaced by Me, Ac or Ts group (entries 16-18), suggesting the critical role of PA.

With the optimized conditions established, the generality of the carbo-acetoxy functionalization reaction was subsequently investigated (Scheme 1), and the electronic effects of different aniline substituents (R) were examined. Introduction of an electron-donating group, such as p-Me, p-Et, p-^{*i*}Pr, p-^{*i*}Bu and p-OMe, to the anilide moiety of **1** led to the corresponding products 2b-f in good yields (66-81%). Substitutions of electron-withdrawing p-F, p-Cl, p-COMe, p-CO₂Me and *p*-CF₃ groups on N-aryl ring were well tolerated, and provided 2g-k in slightly reduced yields (51-68%), presumably due to the lower nucleophilicity of the N-aryl moiety. However, substrate bearing cyano group (11) at the para-position, turned out to be a difficult one, did not afford the desired product. Substrate with *m*-OMe substitution (1m) was chosen to test the regioselectivity of the reaction, which selectively cyclized to produce the 6-methoxy indole (2m) as the predominate product (2m : 2m' = 5:1) as confirmed by 1H-NMR analysis. Substrates bearing o-Me also afforded the corresponding products 2n in 66% yield. Interestingly,

Scheme 1 Synthesis of indole derivatives^a



^aReaction conditions: **1** (0.3 mmol), Pd(OAc)₂ (0.03 mmol), PhI(OAc)₂ (0.75 mmol), PyCICN (0.12 mmol), toluene (4 mL), N₂, 2 h. ^bYield was that of the isolated product. ^CYield in parentheses on gram scale. ^dPd(OAc)₂ (0.03 mmol), PhI(OAc)₂ (0.75 mmol), Ac₂O (1.5 mmol), AcOH (1.5 mmol), 2 h.

withdrawing aryl groups at the tethered chain were welltolerated and afforded the corresponding substituted indoles (**2o-u**) in good yields. However, attempts to utilize a terminal alkene devoid of an aryl moiety for the cascade reaction were unsuccessful (see ESI).

After establishing an efficient route to a variety of indole derivatives, we were interested in extending the method to prepare indoline derivatives by exploring the reaction with substrates bearing a methyl substitution at double bond of cinnamyl tethered anilides (Scheme 2). It was found that anilides containing typical electron donating and electron withdrawing functional groups, such as *p*-Me, *p*-Et, *p*-ⁱPr, *p*-^tBu, *p*-OMe, *p*-F, *p*-Cl and *p*-CF₃, were well tolerated. All the indoline-forming reactions delivered diasteremeric mixtures (\leq 2:1), and so all the studied examples were subjected to hydrolysis and followed by oxidation to afford the corresponding indoline derivatives **4a-i** in good yields.

The products of this reaction could be easily converted into useful entities (Scheme 3). The hydrolysis is non-selective and both acetyl and picolinamide groups could be readily removed in one pot by using aqueous 10% NaOH at 0 °C to afford (1*H*indol-3-yl)(phenyl)methanol, which upon selective Nprotection with (Boc)₂O and oxidation of hydroxyl group with Dess–Martin periodinane afforded the 3-acylindole derivative **5a**. The related 3-acylindoles represent an important class of biologically active heterocyclic compounds such as pravadoline, ramosetron and BPR0L075,¹¹ and our method provides a rapid access to the core structure of these compounds. Interestingly, when we perform the hydrolysis Published on 21 September 2017. Downloaded by University of Sussex on 21/09/2017 19:35:02.

Scheme 2 Synthesis of indoline derivatives^a



^aReaction conditions: (1) **3** (0.3 mmol), Pd(OAc)₂ (0.03 mmol), PhI(OAc)₂ (0.75 mmol), PyCICN (0.12 mmol), toluene (5 mL), N₂, 110 °C, 3 h. (2) K₂CO₃, MeOH/H₂O, r.t, 3 h. (3) DMP, DCM, r.t, 2 h. ^bYield was that of the isolated product. See Supporting Information for details.

using NaOH in MeOH/H₂O as solvent produced methoxy substituted indole derivative **6a** via alkylideneindolenine intermediate **(Int-I)**.¹² Moreover, compound **2a** was transformed into various heteroatom substituted indole derivatives **7a** and **8a** using propane-2-thiol and benzylamine,





^aSee Supporting Information for details.

respectively. The scope of this synthesis and the application of alkylideneindolenine intermediates for the synthesis of indole alkaloids are under study.

To gain some mechanistic insight, the picolinyl protected 3benzylindole (**9a**) was synthesized to examine the possibility of acetoxylation at the benzylic position. As shown in Scheme 4, under the optimized conditions, the reaction did not afford the desired product **2a** (see ESI). This result suggest that the mechanism for arylacetoxylation of **1a** is not via the stable 3benzylindole intermediate.

Scheme 4 Attempted acetoxylation on 3-benzylindole 9a



On the basis of our observations and previous mechanistic studies,¹³ a plausible mechanism for the formation of indoles/indolines is depicted in Scheme 5. The difunctionalization may proceed via arene palladation with subsequent olefin insertion followed by oxidation to high-valent palladium (IV) intermediate with diacetoxyiodobenzene as the oxidant, which undergoes sequential acetoxylation and reductive elemination to afford the compound **2a**. In case of substrate **3a**, due to methyl group substitution, isomerization restricted and acetoxylation afforded the compound **4a**.

Scheme 5 Plausible reaction mechanism



In summary, we have developed a method to access indole and indoline derivatives from simple substrates in a straightforward and efficient manner. Bearing picolinamide as a directing group, the cinnamyl anilines (**1a-u**) cyclized to afford indoles (**2a-u**) in moderate to good yields via direct oxidative arylacetoxylation, while the 2-methyl substituted cinnamyl anilines (**3a-i**) furnished the indoline derivatives (**4a-i**) in good yields via sequential oxidative arylacetoxylation, hydrolysis and oxidation steps. Studies to establish an asymmetric oxidative arylation are currently ongoing in our laboratory. COMMUNICATION

Conflicts of interest

There are no conflicts to declare.

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Page 5 of 6

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9



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