



Palladium-catalyzed annulative allylic alkylation for regioselective construction of indole-fused medium-sized cyclic ethers[☆]

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ARTICLE INFO

Article history:

Received 16 January 2023

Revised 18 March 2023

Accepted 28 March 2023

Available online 31 March 2023

Keywords:

Palladium-catalysis

Annulative allylic alkylation

[5 + *n*] annulation

Regioselectivity

Medium-sized cyclic ethers

ABSTRACT

A new palladium-catalyzed annulative allylic alkylation (AAA) reaction of 2-(indol-2-yl)phenols with dual allylic electrophiles such as isobutylene dicarbonate and butene dicarbonate is described, leading to the regioselective synthesis of tetracyclic medium-sized cyclic ethers possessing a bridged aryl-indole scaffold, namely, benzo[2,3]oxocino[4,5-*b*]indoles and benzo[2,3]oxepino[4,5-*b*]indoles, in good to excellent yields. This protocol demonstrates a broad substrate scope, good compatibility with substituents and high regioselectivity, providing a catalytic and flexible method for creating bridged aryl-indole skeletons.

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Benzannulated medium-sized cyclic ethers and their analogs, such as benzo[*b*]oxocines and benzo[*b*]oxepines, are privileged structural motifs that commonly exist in natural products and biologically active molecules [1–5], as exemplified by heliannuol C [6], cyclomahanimbine [7], protosappanin A [8], caesappin A [9], caesalpinaphenol C [10], and anti-leishmanial agents [11] (Fig. 1). Interestingly, the latter four exhibit axially chiral biaryl architectures. Despite their immense importance, difficulties in the construction of these scaffolds are frequently encountered by standard cyclization methods, because of the inherent challenges of unfavorable transannular interactions and entropic penalties associated with their medium-sized ring formation [12,13]. To overcome these challenges, tremendous synthetic effort has been made to establish some efficient methods toward these two types of skeletally diverse oxycycles, and of these, many of the developed approaches have focused on the assembly of benzo[*b*]oxepines, including traditional multistep coupling [14], intramolecular cyclization [15–21], [5 + 2] [22] or [4 + 3] [23–27] annulation and ring expansion [28–30]. In contrast, few reports on the generation of benzo[*b*]oxocines are documented in the literature [31–34]. Given the great significance of these benzannulated medium-sized cyclic ethers, devel-

oping a broadly applicable and unified approach compatible with such two types of skeletons, especially those incorporating rotationally hindered bridged biaryl scaffolds, is highly desirable but full of challenge.

On the other hand, transition-metal-catalyzed annulative allylic alkylation (AAA) reactions between dual electrophiles and dual nucleophiles have been developed as powerful and versatile strategies for the rapid construction of ring size-varying heterocyclic architectures [35–39]. Specifically, both butene dicarbonate [40–45] and isobutylene dicarbonate [46–51] are usually used and highly reactive dual electrophiles for AAA reactions, which often involve normal [3 + 2], [4 + 2], [3 + 3] and [3 + 4] cyclizations to access cyclic structures (Scheme 1a). However, to the best of our knowledge, although impressive advances have been made in this field [35], there have been no reports using this protocol for the synthesis of indole-fused benzannulated medium-sized cyclic ethers through substrate-enabling [5 + *n*] (*n* = 2, 3) annulation reactions. To continue our interest in cyclization cascades [52–56], we envisioned that privileged medium-sized cyclic ether-bridged aryl-indoles could be synthesized through a metal-catalyzed [5 + *n*] annulation reaction starting from suitable indolyl nucleophiles bearing 1,5-dinucleophilic sites and dual allylic electrophiles such as isobutylene dicarbonate and butene dicarbonate (Scheme 1b). Such a catalytic strategy may encounter difficulty in the choice of 1,5-dinucleophilic indolyl substrates and control of regioselectivity. Herein, we report a Pd-catalyzed AAA reaction of 2-(indol-2-yl)phenols with isobutylene dicarbonate or butene

[☆] Dedication to Prof. Lixin Dai on the Occasion of His Centenary Birthday.

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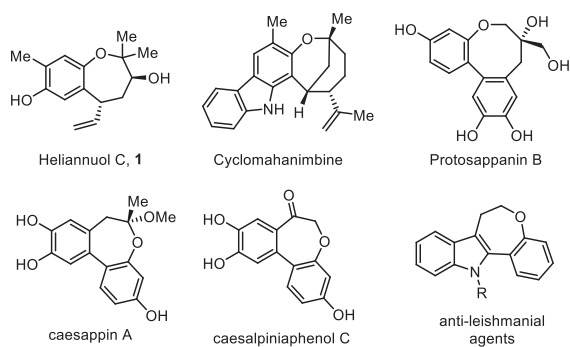
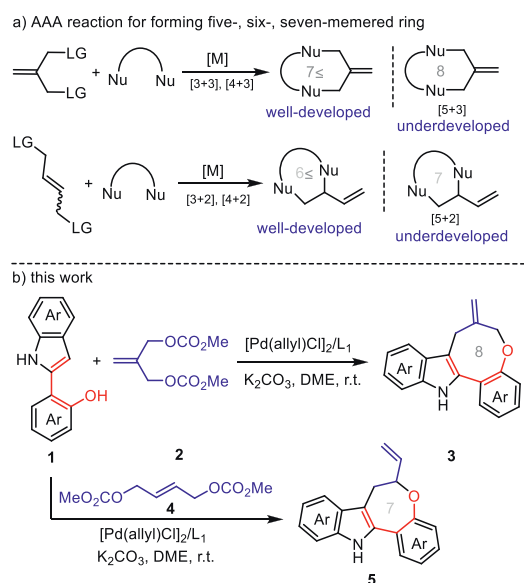


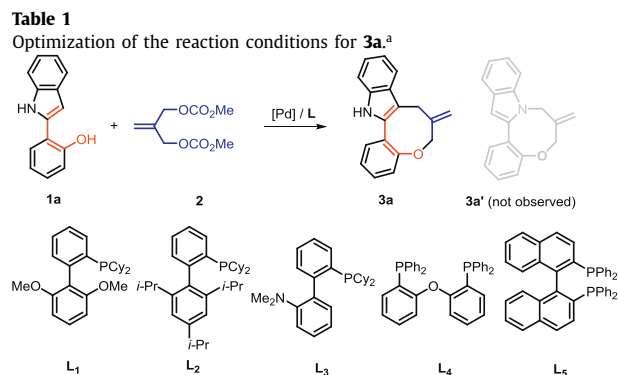
Fig. 1. Benzannulated medium-sized cyclic ether-containing natural products and bioactive molecules.



Scheme 1. Access to tetracyclic medium-sized cyclic ethers.

dicarbonate, in which the former [5 + 3] annulation led to the regioselective synthesis of unprecedented benzo[2,3]oxocino[4,5-*b*]indoles and functionalized benzo[2,3]oxepino[4,5-*b*]indoles could be obtained in the latter [5 + 2] annulation with complete regioselectivity. Notably, both annulation reactions demonstrate complete regioselectivity, enabling the direct formation of rotationally hindered bridged aryl-indole scaffolds.

We initiated our studies with 2-(1*H*-indol-2-yl)phenol (**1a**) as a 1,5-dinucleophile and isobutylene dicarbonate **2** as a 1,3-dielectrophile by exploiting [Pd(allyl)Cl]₂ and phosphine ligand as the catalyst. Details of the screening are summarized in Table 1. The reaction in tetrahydrofuran (THF) in the presence of Cs₂CO₃ by using [Pd(allyl)Cl]₂/SPhos (**L**₁) gave tetracyclic oxocine **3a** as the sole product in 64% yield without observation of its regioisomer **3a'** (Table 1, entry 1), implying that the reaction features complete regioselectivity. Exchanging [Pd(allyl)Cl]₂ to Pd₂dba₃ or Pd(*t*-BuCO₂)₂ led to product **3a** in extremely unsatisfactory yields (entries 2 and 3). Next, the effect of the ligands on this transformation was investigated. Several other mono (**L**₂-**L**₃) and diphosphine (**L**₄-**L**₅) ligands were assayed to enhance the efficiency of this reaction (entries 4–7). However, none of these attempts gave positive results. Taking [Pd(allyl)Cl]₂/**L**₁ as the catalyst, we investigated the effect of the solvent by examining several other aprotic solvents, such as dichloromethane (DCM), toluene, 1,4-dioxane and 1,2-dimethoxyethane (DME) (entries 8–11). The former three solvents were all not beneficial for this transformation, whereas the latter DME gave a higher yield of product **3a** than THF (72%, en-



Entry	[Pd] catalyst/L	Base	Solvent	Yield (%)
1	[Pd(allyl)Cl] ₂ / L ₁	Cs ₂ CO ₃	THF	64
2	Pd ₂ dba ₃ / L ₁	Cs ₂ CO ₃	THF	22
3	Pd(<i>t</i> -BuCO ₂) ₂ / L ₁	Cs ₂ CO ₃	THF	19
4	[Pd(allyl)Cl] ₂ / L ₂	Cs ₂ CO ₃	THF	47
5	[Pd(allyl)Cl] ₂ / L ₃	Cs ₂ CO ₃	THF	31
6	[Pd(allyl)Cl] ₂ / L ₄	Cs ₂ CO ₃	THF	60
7	[Pd(allyl)Cl] ₂ / L ₅	Cs ₂ CO ₃	THF	34
8	[Pd(allyl)Cl] ₂ / L ₁	Cs ₂ CO ₃	DCM	36
9	[Pd(allyl)Cl] ₂ / L ₁	Cs ₂ CO ₃	Toluene	27
10	[Pd(allyl)Cl] ₂ / L ₁	Cs ₂ CO ₃	1,4-Dioxane	56
11	[Pd(allyl)Cl] ₂ / L ₁	Cs ₂ CO ₃	DME	72
12 ^b	[Pd(allyl)Cl] ₂ / L ₁	Cs ₂ CO ₃	DME	63
13	[Pd(allyl)Cl] ₂ / L ₁	–	DME	N.R.
14	[Pd(allyl)Cl] ₂ / L ₁	DABCO	DME	N.R.
15	[Pd(allyl)Cl] ₂ / L ₁	DIPEA	DME	N.R.
16 ^c	[Pd(allyl)Cl] ₂ / L ₁	K ₂ CO ₃	DME	95
17	[Pd(allyl)Cl] ₂ / L ₁	K ₃ PO ₄	DME	58
18 ^{c,d}	[Pd(allyl)Cl] ₂ / L ₁	K ₂ CO ₃	DME	80
19 ^{c,e}	[Pd(allyl)Cl] ₂ / L ₁	K ₂ CO ₃	DME	89

^a Reaction conditions: **1a** (0.05 mmol), **2** (0.06 mmol), base (0.1 mmol), Pd catalyst (5 mol%), and ligand (6 mol%) in solvent (1.0 mL) at r.t., 2 h.

^b DME (1.5 mL);.

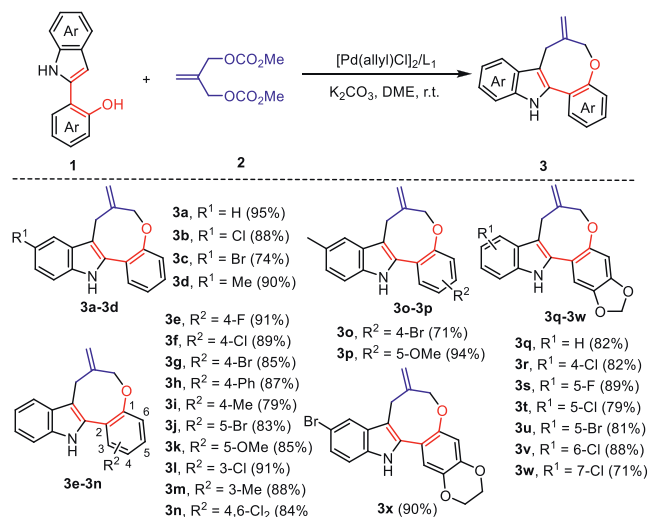
^c 1.5 h

^d Pd catalyst (1 mol%), **L**₁ (1.2 mol%).

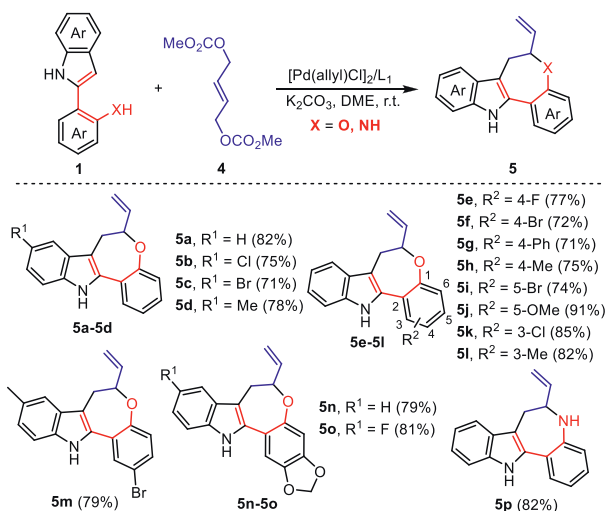
^e Pd catalyst (2.5 mol%), **L**₁ (3 mol%).

try 11). Increasing the amount of DME resulted in a significantly declining yield (63%, entry 12). Without the base, the reaction did not proceed (entry 13), suggesting that the base is crucial for this reaction. This result prompted us to screen various bases, such as DABCO, DIPEA, K₂CO₃ and K₃PO₄, for this transformation (entries 14–17). The results indicate that organic bases such as DABCO and DIPEA completely suppressed the reaction process (entries 14 and 15); in contrast, inorganic bases could drive the conversion into product **3a** (entries 16 and 17), and of these inorganic bases, K₂CO₃ proved to be a better choice because it could not only increase the yield of product **3a** but also shorten the reaction time (entry 16). Lowering the Pd catalyst loading was harmful for this transformation, because a lower conversion was observed (80% and 89%) when using 1.0 or 2.5 mol% Pd catalyst (entries 18 and 19).

With these acceptable reaction conditions in hand, we set out to examine the scope of this catalytic [5 + 3] annulation by exploiting a number of 2-(1*H*-indol-2-yl)phenols. As shown in Scheme 2, first, the electronic nature of substituents at the C5 position of the indole ring was probed. Both electron-withdrawing (chloro **1b** and bromo **1c**) and electron-donating (methyl **1d**) groups were compatible with the standard conditions, giving access to corresponding products **3b**–**3d** as sole regioisomers in 74%–90% yields. Next, the effects of changing the electronic properties and positions of substituents in the phenol ring of substrate **1** were carefully investigated. As expected, the reaction proceeded readily with the tolerance of various commonly encountered substituents, such as electronically poor (C4-fluoro, **1e**; C4-/C3-chloro, **1f** and **1i**; C4-/C5-bromo, **1g** and **1j**) and rich (C4-phenyl **1h**, C4-/C3-methyl, **1i** and **1m**; C5-methoxy, **1k**) groups at different positions of the



Scheme 2. Substrate scope for the synthesis of **3**. Reaction conditions: **1** (0.2 mmol), **2** (0.22 mmol), K₂CO₃ (0.4 mmol), [Pd(allyl)Cl]₂ (0.01 mmol), L₁ (0.012 mmol) and DME (1.0 mL), r.t., 2 h.

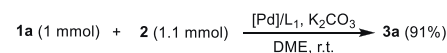


Scheme 3. Substrate scope for the synthesis of **5**. Reaction conditions: **1** (0.2 mmol), **4** (0.24 mmol), K₂CO₃ (0.4 mmol), [Pd(allyl)Cl]₂ (0.01 mmol), L₁ (0.012 mmol) and DME (1.0 mL), r.t., 3 h.

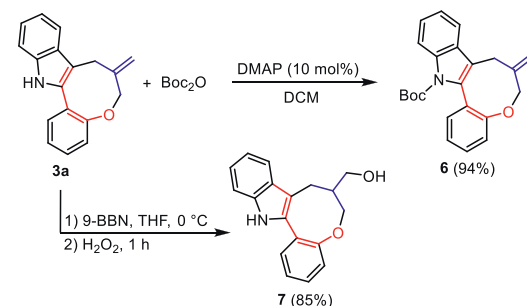
arene ring, and corresponding tetracyclic oxepine products **3e-3m** were regioselectively synthesized with yields ranging from 79% to 91%. Alternatively, substrate **1n** decorated with double chloro-substituents in the phenyl ring proved to be efficient (**3n**, 83%). Moreover, the reaction occurred smoothly with different functional groups on both indole and a phenol ring of substrates **1** (**3o** and **3p**). Furthermore, sesamol-derived counterparts **1q-1w** bearing C4- to C7-substituents at the indole ring were functional for this catalytic protocol, providing pentacyclic eight-membered ring ethers **3q-3w** in good yields. 2,3-Dihydrobenzo[*b*][1,4]dioxin-6-ol-derived substrate **1x** showed good reactivity and delivered target pentacyclic product **3x** in 90% yield (Scheme 3).

Having demonstrated that isobutylene dicarbonate is an excellent 1,3-dielectrophilic partner in the [5 + 3] annulation with 1,5-dinucleophilic indolyl reactants, we wondered whether butene dicarbonate as a 1,2-dielectrophilic component, instead of isobutylene dicarbonate, could lead to [5 + 2] annulations. This would be particularly relevant because the resulting products are also medium-sized oxycycles, privileged skeletons in biomedical chem-

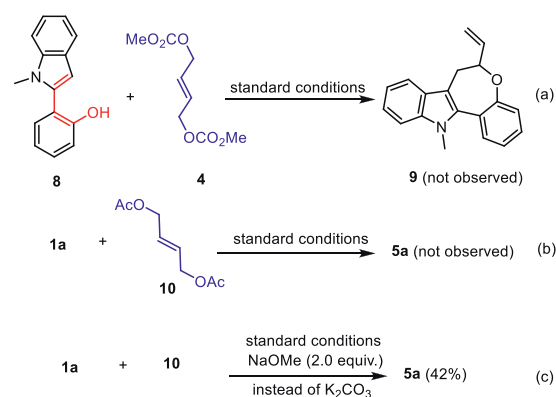
(a) Scale-up transformation into **3a**



(b) transformation of **3a**



Scheme 4. Synthetic potential of this protocol.

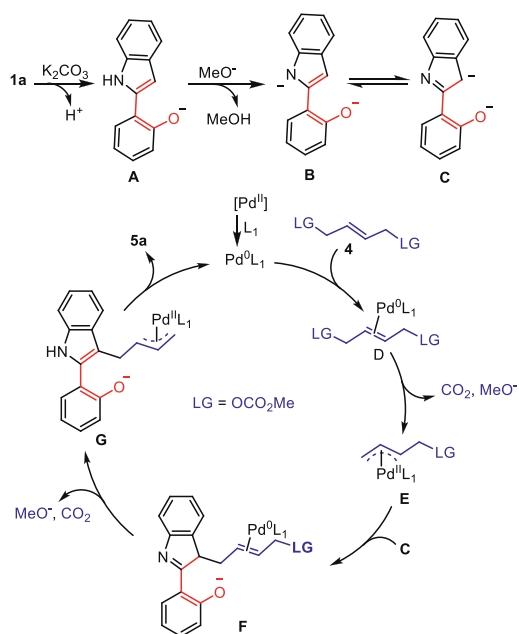


Scheme 5. Control experiments.

istry and pharmaceutical science (Fig. 1). Thus, butene dicarbonate was quickly prepared from the corresponding 2-butene-1,4-diol in a scalable manner [57]. For comparison purposes, we also considered using the above identical 2-(1*H*-indol-2-yl)phenols **1** as 1,5-dinucleophilic partners to react with butene dicarbonate **4**. Similarly, 1,5-dinucleophilic substrates also exhibited good functional group compatibility, irrespective of the position and electronic properties of substituents on both indole and phenol rings, and all performed well in the transformation to deliver the desired tetracyclic benzo[*b*]oxepines **5a-5o** featuring rotationally hindered bridged aryl-indole scaffolds with comparable efficiency. Various functional groups, such as halo (F, Cl and Br), methyl, phenyl and ether, were found to be applicable, enabling catalytic annulative allylic alkylation to access the expected polycyclic products. Finally, 2,3-dihydrobenzo[*b*][1,4]dioxin-6-ol-derived substrate **1u** was also possible in this [5 + 2] annulation. The structures of products **3h** (CCDC: 2236463) and **5m** (CCDC: 2236465) were confirmed by single-crystal X-ray diffraction analysis.

The scalability of this protocol was examined by synthesizing product **3a** on a 1.0 mmol scale. In this case, the desired product **3a** was obtained with almost no loss of yield (Scheme 4a). Subsequently, compound **3a** reacted with Boc₂O in the presence of 10 mol% 4-dimethylaminopyridine (DMAP), furnishing Boc-protected **6** in 94% yield. Subsequent hydroboration oxidation of **3a** gave the corresponding alcohol **7** in 85% yield (Scheme 4b) [58].

To understand the mechanistic features of this annulative process, *N*-methyl 2-(1*H*-indol-2-yl)phenol (**7**) was subjected to the reaction of **4** under standard conditions. Unfortunately, the desired product **9** was not observed as substrate **8** was not consumed at all (Scheme 5a), suggesting that free N-H is crucial for this trans-



Scheme 6. Proposed mechanism for forming product **5a**.

formation. Next, treatment of **1a** with butene diacetate **10** did not give product **5a** (Scheme 5b). Exchanging K_2CO_3 for NaOMe in the reaction system resulted in a 42% yield of **5a** (Scheme 5c), showing that the strong base could drive the reaction. From these results, it may be speculated that in the presence of a strong base, unstable indol-3-ide generated *in-situ* may be the key intermediate for allylic nucleophilic addition.

Based on the experimental results and previous works [35], a proposed catalytic cycle is depicted in Scheme 6. Initially, in the presence of K_2CO_3 , substrate **1a** loses a proton from the hydroxyl group to form 2-(1*H*-indol-2-yl)phenolate **A**, which loses another proton from the N-H bond in the presence of MeO^- from the decomposition of butene dicarbonate **4** to give intermediate **B**, followed by isomerization to yield unstable indol-3-ide intermediate **C** for the next allylic addition because the carbon anion favors chelation with Pd(II) species compared with normal nitrogen and oxygen atoms. Next, coordination of **4** with palladium species (Pd^L) from $[Pd(allyl)Cl]_2$ and phosphine ligand L_1 affords complex **D**, followed by oxidative addition to give Pd- π -allyl **E**, along with CO_2 and MeO^- . Intermediate **E** is captured by indol-3-ide **C**, enabling terminal allylic nucleophilic addition to form intermediate **F**, which undergoes proton transfer and second oxidative addition to afford Pd- π -allyl **G**, accompanied by CO_2 and MeO^- . The following intramolecular proximal O-allylation of **G** provides product **5a** and regenerates the Pd catalyst for the next catalytic cycle.

In summary, we have developed a new Pd-catalyzed annulative allylic alkylation reaction consisting of a twofold decarboxylative allylation process from 2-(indol-2-yl)phenols and dual allylic electrophiles such as isobutylene dicarbonate and butene dicarbonate, and a wide range of tetracyclic medium-sized cyclic ethers bearing a bridged aryl-indole scaffold were regioselectively synthesized with good to excellent yields. In all cases, complete regioselectivity was observed, and the construction of two types of medium-sized cyclic ethers, namely, oxocines and oxepines, was easily achieved. The present protocol features good functional group tolerance and a wide substrate scope, thus opening a new entry to fabricate bridged aryl-indole scaffolds through a catalytic AAA reaction. Further investigations and application of the AAA reaction for asymmetric synthesis are underway in our laboratory.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

We are grateful for the National Natural Science Foundation of China (Nos. 21971090 and 22271123).

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