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Rhodium-Catalyzed Amination and Annulation of Arenes with Anthranils: C-H Activation Assisted by Weakly Coordinating Amides

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Abstract: A rhodium(III)-catalyzed C–H amination of benzamides and isoquinolones with anthranils has been realized under assistance of weakly coordinating amide, leading to a bifunctionalized amination product which can further cyclize to acridine under in situ or ex situ conditions.

Keywords: C–H amination; anthranil; benzamide; isoquinolone; bifunctionality

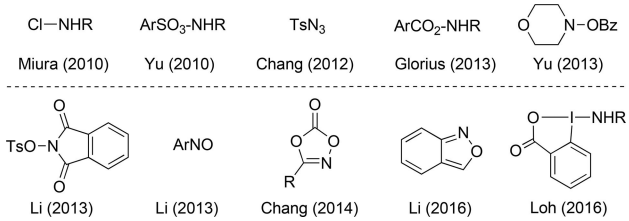


Figure 1. Electrophilic Aminating/Amidating Reagents in C–H Activation.

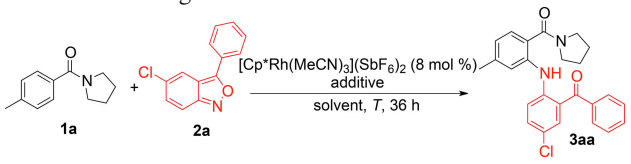
The C–N bond is a key linkage in organics and pharmaceuticals.[1] Efficient construction of C–N bond has been an ongoing task in the past decades. Traditional protocols of construction of C(aryl)-N bond include the Buchwald-Hartwig amination,[2] the Chan-Lam coupling,[3] and the Ullman coupling, among others. These methods required functionalized aromatic starting materials or harsh conditions which delivered products with limited atom-economy. With the increasing interest of C–H activation chemistry, transition metal catalyzed C–H amination/amidation[4] has been increasingly explored using palladium,[5] rhodium,[6] ruthenium,[7] iridium,[8] cobalt,[9] and copper[10] catalysts. Two classes of amination reactions have been developed using nucleophilic or electrophilic aminating reagents. The electrophilic aminating reagents are particularly attractive owing to high reactivity and redox-neutrality (Figure 1).[11–16]

While the strategy of C–H amination is appealing, it is desirable to introduce two functional groups in one single transformation. Ideally, this is realized by scission of an N–E bond of a ring (E=N, O, or C) and both the N and the E atoms are incorporated into the product, leading to bifunctionality. In this context, we and others independently applied anthranils as an aminating reagent for both sp^2 and sp^3 C–H bonds under chelation assistance.[14,17] In the cases when highly nucleophilic arene substrates such as indoles were used, further cyclization to acridines may arise due to the nucleophilic nature of the aniline intermediate and the electrophilicity of the resulting proximal carbonyl group.[17a,b] Despite the progress, the directing group of such systems have been strictly limited to strong nitrogen chelators.[14,17] Applications of weak oxygen chelators[18] in amination have been rare in general, and secondary amides were mostly employed.[19] Thus, it is important to develop amination systems using tertiary amides[20] as directing groups that are both readily available and easily transformable. However, challenges remain not only in the weak directing effect of an amide directing group but also in the relative instability of the anthranils, which readily decompose to the *ortho* amino ketones via (reductive) ring scission.[14]

We initiated our studies with the optimization of the reaction conditions of the coupling of *N*-Benzoylpyrrolidine **1a** and anthranil **2a** (Table 1). In the presence of a cationic rhodium(III) catalyst, an *ortho* C–H amination reaction occurred in DCE at 120 °C to give the aminated product **3aa** in 52% yield (entry 1),

together with an *ortho*-aminobenzophenone, a typical decomposition product of this anthranil. Omission of the HOAc additive led to a poor coupling efficiency (entry 2). The reaction is sensitive to choice of solvent, and only halogenated solvents seem applicable to ensure good efficiency (entries 3–7). Thus, 1,2-dichlorobenzene (DCB) was identified as the optimal solvent (entry 4). Switch of the additive to zinc acetate raised the yield to 86% (entry 9), while other Zn(II) salts or metal acetates all resulted in lower yields (entry 10–16), indicating that both the cation and anion of $Zn(OAc)_2$ played an important role. Further lowering or increasing the reaction temperature all gave diminished yields (entries 17 and 18). In all cases, only a small amount (<5%) of the amination-annulation product was obtained, but a fair amount of the decomposition product of anthranil was observed.

Table 1. Screening of Reaction Conditions[a].



entry	additive (equiv)	solvent	T (°C)	yield (%) [b]
1	HOAc (2)	DCE	120	52
2	–	DCE	120	40
3	HOAc (2)	PhCl	120	56
4	HOAc (2)	DCB	120	65
5	HOAc (2)	$PhCF_3$	120	50
6	HOAc (2)	MeOH	120	0
7	HOAc (2)	THF	120	0
8	PivOH (2)	DCB	120	50
9	$Zn(OAc)_2$(0.3)	DCB	120	86
10	$Zn(OTf)_2$ (0.3)	DCB	120	60
11	$ZnSO_4$ (0.3)	DCB	120	41
12	$ZnBr_2$ (0.3)	DCB	120	0
13	NaOAc (0.3)	DCB	120	62
14	KOAc (0.3)	DCB	120	50
15	$Ca(OAc)_2$ (0.3)	DCB	120	65
16	AgOAc (0.3)	DCB	120	78
17	$Zn(OAc)_2$ (0.3)	DCB	135	62
18	$Zn(OAc)_2$ (0.3)	DCB	110	70

[a] The reaction was carried out using **1a** (0.2 mmol), **2a** (0.4 mmol), $[Cp^*Rh(MeCN)_3](SbF_6)_2$ (8 mol%), additive in a solvent (2 mL) at T °C for 36 h under Ar.
[b] Isolated yield.

With the optimized reaction conditions in hand, we next explored the scope and limitations of this coupling system (Scheme 1). The scope of the arene was first examined in the coupling with anthranil **2a**. Benzoylpyrrolidines bearing halogens (**3ha, 3ia**), electron-donating (**3ca**, **3da**), and -withdrawing (**3fa**) groups at the *para* position were all applicable, although an electron-withdrawing group tends to attenuate the yield (**3fa**, **3ia**). Introduction of different *meta* substituents is also tolerated, and the amination reaction tend to occur at the less hindered *ortho* site with moderate yield (**3ja**, **3ka**). An exception was observed for meta-F group, where two isomeric products (**3la** and **3la′**) were obtained due to the secondary directing effect. Besides the amination of a benzene ring, the reaction has been extended to a thiophene substrate (**3ma**) under modified conditions.

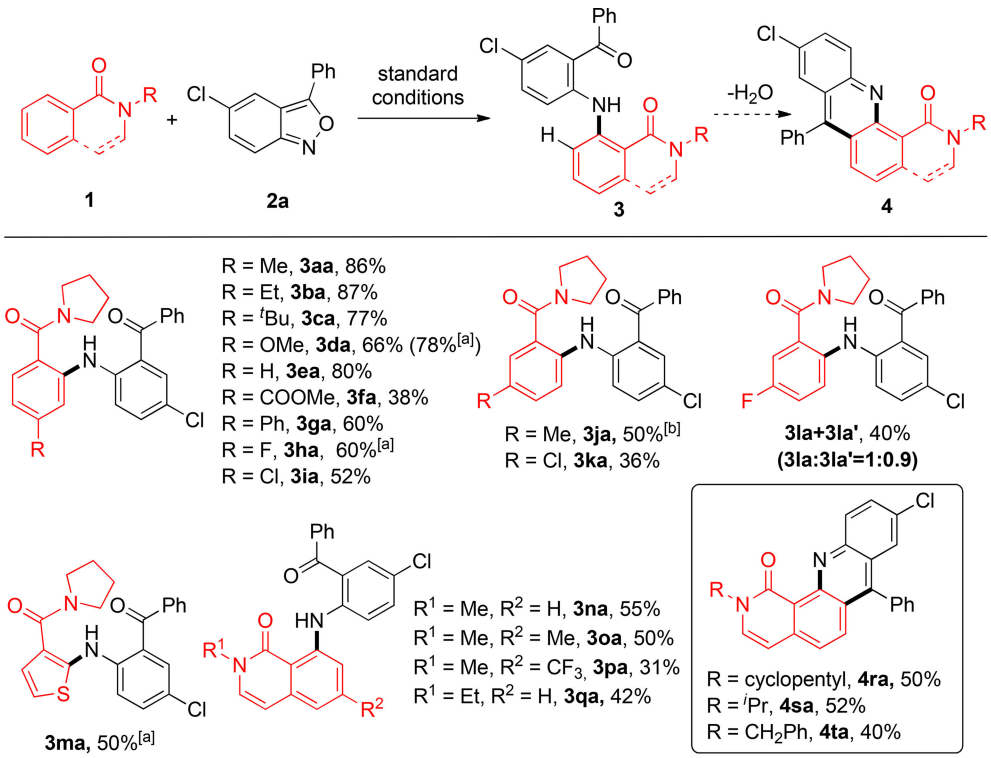
Extension of the benzamide substrate to isoquinolones, another class of arene, proved successful. Interestingly, substrate dependent selectivity was observed for this type of arene. Thus, *N*-methyl and -ethylisoquinolones underwent C(8)-H activation and coupling with **2a** to afford the corresponding amination product as the major pathway (**3oa–3ra**). In contrast, *N*-isopropyl, -cyclopentyl, and -benzylisoquinolones coupled under the same conditions to afford fused acridines as the major products via amination-cyclization condensation (**4sa–4ua**). The switch of the reaction selectivity is dictated by the subtle differences of the *N*-substituent in terms of electronic and steric effects. Electronically, the cyclization is favored by a more electron-donating *N*-substituent.

The scope of anthranil was then examined using both benzoylpyrrolidine and isoquinolone as the arene substrate (Scheme 2). The amination reaction proceeded smoothly for various anthranils bearing halogen and alkyl substituents in the aryl ring and in the anthranil ring (50–80%). The halogen groups in the final aminated product should provide handles for further chemical functionalization.

To better define the scope of anthranils, 3-unsubstituted anthranils were applied. In contrast to the *ortho*-amination reaction in Scheme 2, the corresponding aldehyde was not observed. Instead, the amination-cyclization condensation products were isolated, and the condensation was favored by the more reactive aldehyde intermediate. Thus, anthranils bearing different substituents in the backbone underwent smooth coupling with **1a** to afford the corresponding annulated product in moderate to good yields (Scheme 3)

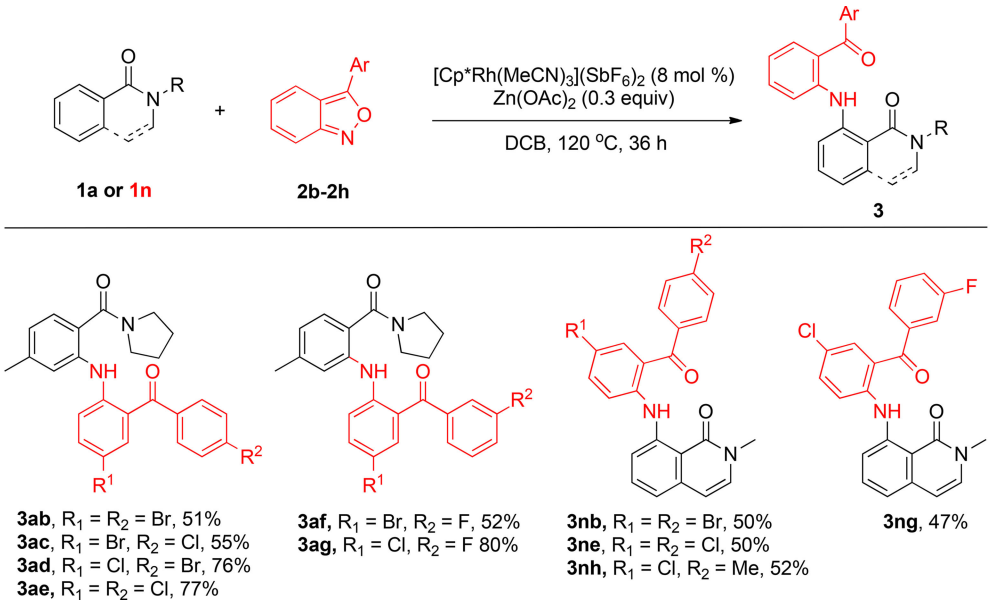
To realize the above acridine synthesis from the corresponding 3-substituted anthranils, the electrophilicity of the aminated ketone intermediate must be enhanced. Thus, treatment of these ketones with TFA as a solvent led to smooth condensation in excellent yield (**4aa–4ja**, Scheme 4). Alternatively, telescoping synthesis of such products from benzamide **1a** and anthranil was also realized in an overall good yield (eq 1).

Besides the condensation reaction, derivatization of two aminated products was also conducted. Selective reduction of **3aa** using $NaBH_4$ afforded the alcohol **5** in high yield. When alcohol **5** was reacted

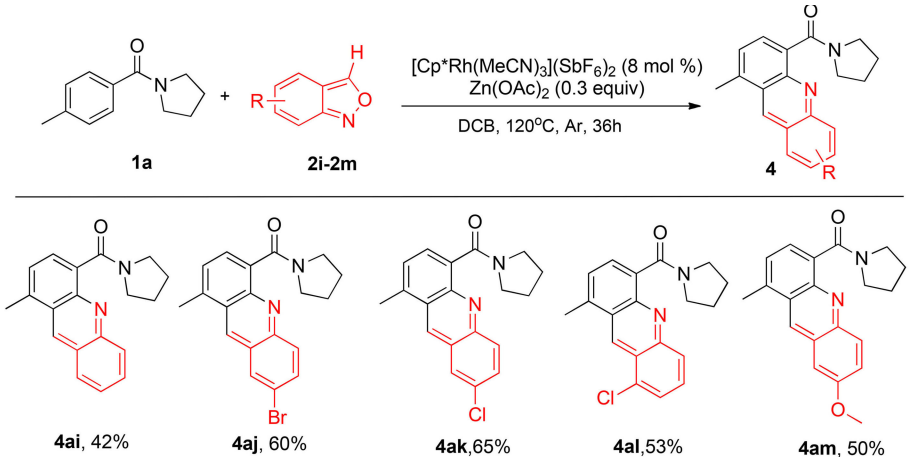


[a] $[Cp^*Rh(CH_3CN)_3](SbF_6)_2$ (8 mol %), $Zn(OAc)_2$ (0.3 equiv), DCE (2 mL), 120 °C, 36 h.
[b] $[Cp^*RhCl_2]_2$ (4 mol %), $AgNTf_2$ (16 mol %), $Zn(OAc)_2$ (0.3 equiv), DCB (2 mL), 120 °C, 36 h.

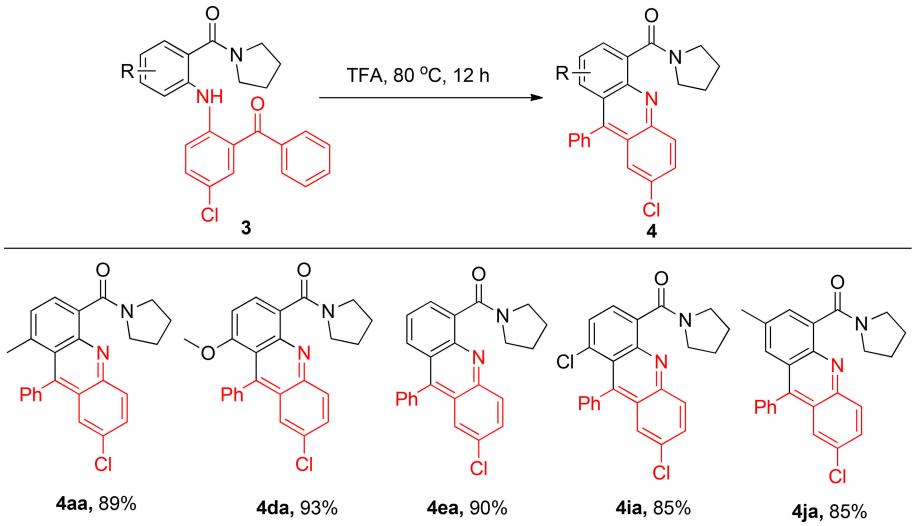
Scheme 1. Substrate Scope of Amides. *Reaction conditions*: **1** (0.2 mmol), **2a** (0.4 mmol), $[Cp^*Rh(MeCN)_3](SbF_6)_2$ (8 mol %), $Zn(OAc)_2$ (0.3 equiv), DCB (2 mL), 120 °C, 36 h, isolated yield after column chromatography.



Scheme 2. Scope of Anthranils in Amination. *Reaction conditions*: amide (0.2 mmol), anthranil (0.4 mmol), $[Cp^*Rh(MeCN)_3](SbF_6)_2$ (8 mol%), $Zn(OAc)_2$ (0.3 equiv), DCB (2 mL), 120 °C, 36 h, isolated yield after column chromatography.

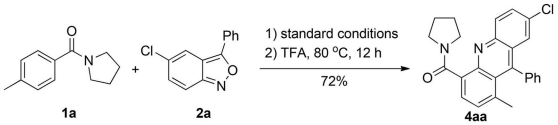


Scheme 3. Amination-Cyclization Using 3-Unsubstituted Anthranils. *Reaction Conditions*: **1a** (0.2 mmol), anthranil (0.4 mmol), $[Cp^*Rh(MeCN)_3](SbF_6)_2$ (8 mol%), and $Zn(OAc)_2$ (0.3 equiv), DCB (2 mL), 120 °C, 36 h, isolated yield after column chromatography.



Scheme 4. Transformations of Some Coupled Products. *Reaction conditions*: compound **3** (0.1 mmol), TFA (1 mL), 80 °C, 12 h, isolated yield after column chromatography.

with 2-methylfuran, the arylated derivative **6** was obtained in high yield. Treatment with *m*-CPBA and TFA afforded the transannulated product **7** in overall moderate yield (eq 2).[21] Reduction of **3ja** with LAH led to reduction of both the amide and the ketone functional groups (eq 3). To further display the synthetic utility, a 2 mmol-scale reaction has been performed and the product **3ba** was obtained in 83% yield (eq 4).



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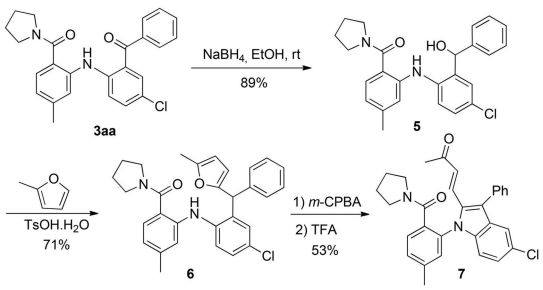

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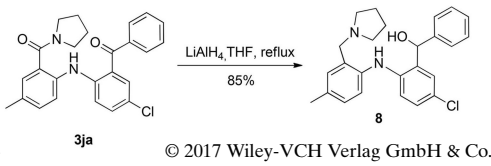
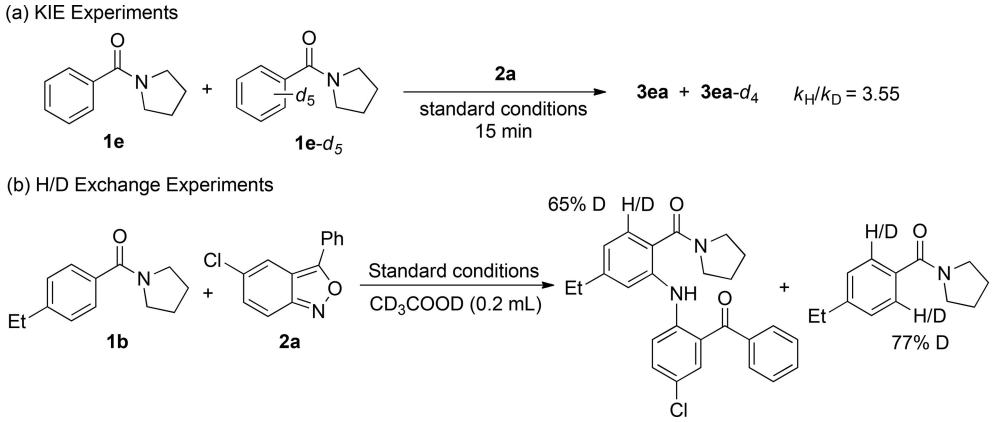
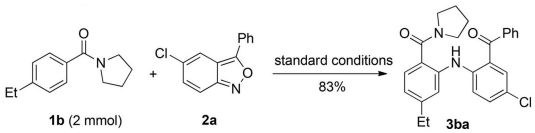


(3)



Scheme 5. Mechanistic Studies.



(4)

Preliminary studies have been performed to explore the mechanism of this amination process. H/D exchange was performed for the coupling of **1b** and **2a** in the presence of CD_3COOD, from which **1b** was recovered with 77% deuteration at the ortho positions. Also, the aminated product was obtained with 65% deuteration at the *ortho'* position. Measurement of the kinetic isotope effect (KIE) under intermolecular competition conditions using **1a** and **1a**-d_5 gave $k_H/k_D = 3.55$. These data collectively suggested the relevancy of C–H activation (Scheme 5).

In summary, we have applied tertiary amide as a weak directing group for the C–H amination of arenes using anthranils. Both benzoylpyrrolidines and *N*-alkylisoquinolones are viable arene substrates. The reaction could afford terminal amination products or further cyclized products, and this selectivity was under substrate control. This coupling system expended the scope of arenes in C–H amination assisted by a weak directing group. Studies on C–H activation of arenes and alkenes assisted by other weakly coordinating directing groups are underway and will be reported in due course.

Experimental Section

General Procedure for the Synthesis of Compound 3, 4sa–4ua and 4ai–4am

Arene **1** (0.2 mmol, 1 equiv), anthranil **2** (0.4 mmol, 2 equiv), $[Cp^*Rh(MeCN)_3](SbF_6)_2$ (8 mol%) and $Zn(OAc)_2$ (0.3 equiv) were charged into a Schlenk tube, to which was added anhydrous DCB (2.0 mL) under argon. The reaction mixture was stirred at 120 °C for 36 h. After cooled to room temperature, the solvent was removed under reduced pressure and the residue was purified by silica gel chromatography using acetate/petroleum ether/$NH_3.H_2O$ (1:7:0.2~1:1:0.2) to afford the desired product.

Acknowledgements

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