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Amination

Carboxylate-Assisted Iridium-Catalyzed C–H Amination of Arenes with Biologically Relevant Alkyl Azides

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Abstract: An iridium-catalyzed C–H amination of arenes with a wide substrate scope is reported. Benzamides with electron-donating and -withdrawing groups and linear, branched, and cyclic alkyl azides are all applicable. Cesium carboxylate is crucial for both reactivity and regioselectivity of the reactions. Many biologically relevant molecules, such as amino acid, peptide, steroid, sugar, and thymidine derivatives can be introduced to arenes with high yields and 100% chiral retention.

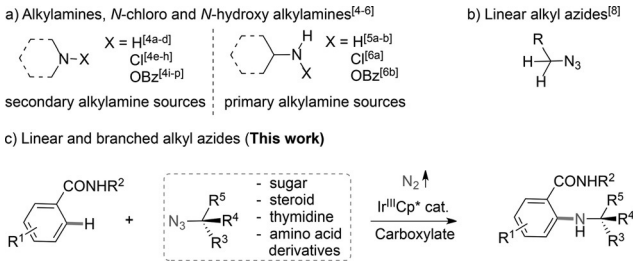

Figure 1. Alkylamine sources in directed C–H amination.

Given the ubiquity of arylamines in natural products, drug molecules, organic optoelectronic materials, and synthetic intermediates, facile methods to introduce nitrogen substituents onto aromatic rings have been a major focus of new methodology development.[1] Extensive research on Cu- and Pd-catalyzed C–N cross coupling reactions of aryl(pseudo)halide substrates led to the development of Ullmann–Goldberg, Buchwald–Hartwig, and Chan–Lam aminations.[2] As aryl(pseudo)halides are not always readily accessible, a metal-catalyzed amination of arenes through C–H activation has recently emerged as an alternative method for its evident advantage.[3] However, there are two major challenges of a direct C–H amination. First, the C–H bond has high energy barrier to dissociate; second, the newly formed amino groups have the tendency to retain coordination with the metal catalysts. As a consequence, the direct C–H aminations developed to date are most limited to amino sources with a strong electron-withdrawing group, such as sulfonyl and carbonyl group, conjugated to the nitrogen atom.[3] Recently, secondary alkylamines[4a–d] and their analogues, for example, *N*-chloroamines[4e–h] and *N*-hydroxyamines[4i–p] have also been widely used in the metal-catalyzed C–H amination of chelation-group-containing substrates, probably benefitting from the steric hindrance of newly formed tertiary amino groups (Figure 1a). However, to the best of our knowledge, primary alkylamines and their analogues have been underdeveloped as substrates for the direct C–H amination, even though they are abundant in nature (Figure 1a).[5,6] The reported methods normally require external oxidants and/or bases; this limits their practical application, as many compounds, especially biological active compounds, are oxidation/base sensitive. Therefore, a method of direct C–H amination with high functional group tolerance is still needed.

Azides are an important source of amines and have unique advantages. For example, they are easy to prepare, green (generate N_2 gas as the sole by-product), and reactions with azides could be conducted under mild conditions (oxidative or basic conditions are not necessary).[3f] In addition, due to the expanding popularity of click chemistry, many alkyl azides, including biologically relevant ones, are readily available.[7] However, applications of alkyl azides as an alkylamine source in metal-catalyzed direct C–H amination reactions are less explored. To the best of our knowledge, only three reports have been described recently that employs alkyl azides as the substrates in Rh[8a,b] and Ir[8c] catalytic systems. The major limitations of these methods are i) the azide substrates and/or arenes need to be electron-deficient to give high yields, and ii) only linear alkyl azides are used in these reactions; branched alkyl azides are not applicable (Figure 1b).

Inspired by the Ir-catalyzed direct C–H amination of arenes with organic azides[8c,9] and our previous studies,[10] we herein report a method of Ir-catalyzed intermolecular C–H amination of arenes. With the promotion of CsOAc, this method is well applicable to linear, branched, and cyclic alkyl azides; it is also suitable to benzamides bearing electron-donating and withdrawing groups (Figure 1c).

Amination of the C–H bond at the *ortho*-position of benzamide can produce anthranilamide, a motif found in many drug candidates.[11] Therefore, we chose benzamide and

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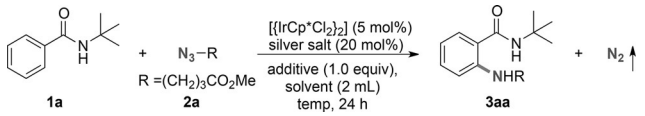
Table 1. Optimization of reaction conditions.[a]



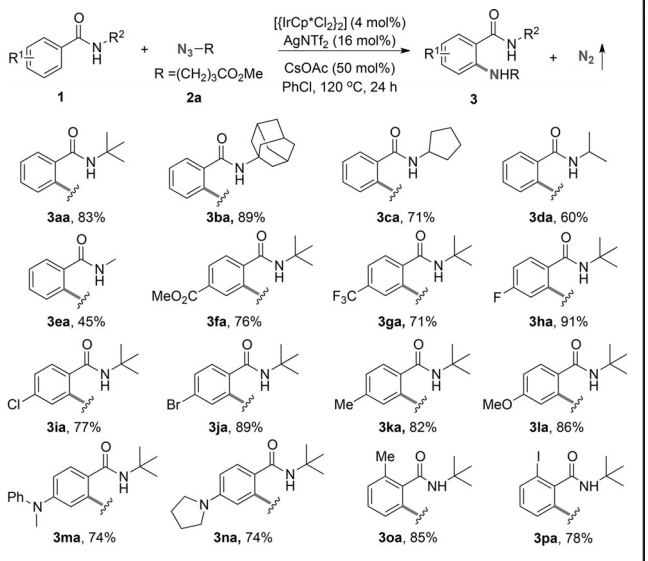
Entry	Silver salt	Additive	Solvent	T [°C]	Yield [%][b]
1	$AgNTf_2$	NaOAc	DCE	50	–
2	$AgNTf_2$	NaOAc	DCE	140	–
3	$AgNTf_2$	AgOAc	DCE	140	8
4	$AgNTf_2$	HOAc/Li_2CO_3[c]	DCE	140	28
5	$AgNTf_2$	KOAc	DCE	140	<5
6	$AgNTf_2$	HOAc	DCE	140	8
7	$AgNTf_2$	CsOPiv	DCE	140	13
8	$AgNTf_2$	CsOAc	DCE	140	57
9	$AgNTf_2$	CsF	DCE	140	–
10	$AgNTf_2$	Cs_2CO_3	DCE	140	–
11	$AgNTf_2$	CsOAc	dioxane	140	33
12	$AgNTf_2$	CsOAc	THF	140	36
13	$AgNTf_2$	CsOAc	benzene	140	30
14	$AgNTf_2$	CsOAc	*o*-xylene	140	54
15	$AgNTf_2$	CsOAc	CF_3CH_2OH	140	14
16	$AgNTf_2$	CsOAc	PhCl	140	77
17	AgOTf	CsOAc	PhCl	140	29
18	$AgBF_4$	CsOAc	PhCl	140	15
19	$AgSbF_6$	CsOAc	PhCl	140	58
20	$AgNTf_2$	CsOAc	PhCl	150	70
21	$AgNTf_2$	CsOAc	PhCl	120	88
22	**$AgNTf_2$**	**CsOAc**	**PhCl**	**120**	**87**[d]

[a] Reaction conditions: **1a** (0.36 mmol), **2a** (0.2 mmol). [b] Yield was calculated based on crude ^{1}H NMR spectra using CH_2Br_2 as the standard. [c] LiOAc (1.0 equiv)/Li_2CO_3 (1.0 equiv). [d] Optimized conditions: **1a** (0.3 mmol), **2a** (0.2 mmol), [{IrCp*Cl_2}$_2$] (4 mol%), $AgNTf_2$ (16 mol%), CsOAc (50 mol%), PhCl (2 mL).

$N_3(CH_2)_3CO_2Me$ (**2a**) as the substrates (Table 1 and Table S1 in the Supporting Information). We began our research with the Chang's optimized amination conditions;[8c] using [{IrCp*Cl_2}$_2$]/$AgNTf_2$ as the catalyst and NaOAc as the additive, no desired product was formed even after increasing the reaction temperature to 140 °C (Table 1, entries 1 and 2). Subsequently, a variety of carboxylates (entries 3–8) and cesium salts (entries 9 and 10) were screened in DCE, and we were delighted to discover that the desired product **3aa** was obtained in 57% yield when CsOAc was used as the additive (entry 8). After solvents (entries 11–16), silver salts (entries 17–19), temperatures (entries 20 and 21), and the stoichiometry of additive (entry 22) were screened, the optimal conditions for this amination were found (entry 22). No desired product was formed in the presence of other metals, such as [{RhCp*Cl_2}$_2$]/$AgNTf_2$,[8a,b] [RhCp*$(MeCN)_3$]$(SbF_6)_2$, [{Ru(*p*-cymene)Cl_2}$_2$], and $Pd(OAc)_2$ (Table S1 in the Supporting Information).

We then explored the scope of this reaction with a series of substrates (**1**; Table 2). Benzamides with various *N*-alkyl substitutions could be used in the reaction (**3aa**–**ea**). Both electron-withdrawing and -donating groups at the *para*-position of the phenyl ring were well tolerated (**3fa**–**na**). Functional groups, such as ester, halogen, methoxyl, and amino, were also tolerated. Finally, the reaction efficiency is independent of the *ortho*-substitution of the phenyl ring (**3oa**–**pa**).

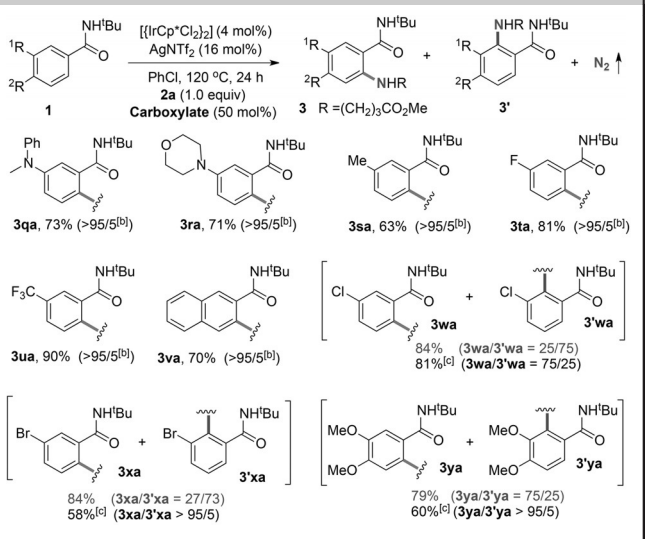
Table 2. Scope of benzamides **1**.[a]



[a] Reaction conditions: **1** (0.3 mmol), **2a** (0.2 mmol), isolated yield.

We then studied the effects of *meta*-substitution on the benzamide **1** (Table 3). *Meta*-substitution can affect both the accessibility and electron density of the reaction site, which are the major factors controlling and differentiating the reactivity and selectivity of C–H bonds.[12] In this reaction, the C–H amination generally takes place predominately at the less hindered position with high regioselectivity (**3qa**–**va**). However, when the *meta*-substituent on the benzamide was chlorine, bromine, and methoxyl, a mixture of regioisomers was obtained (**3wa**–**ya**, **3′wa**–**ya**). This can be explained by the *ortho*-directing

Table 3. Cesium carboxylate controlled regioselectivity of *meta*-substituted benzamides **1**.[a]

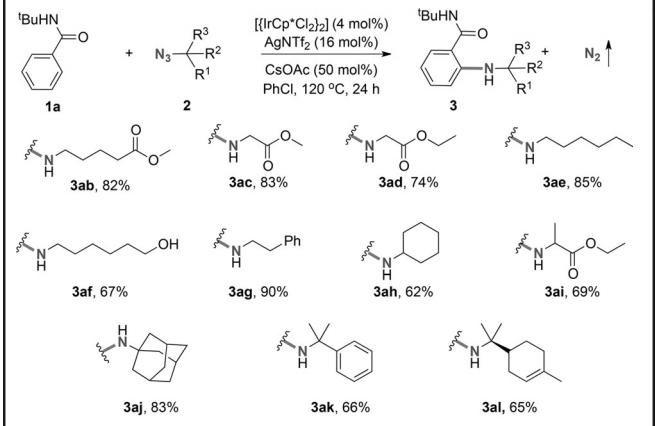


[a] **1** (0.3 mmol), **2a** (0.2 mmol), CsOAc (50 mol%), PhCl (2 mL), isolated yield. [b] The ratio of the products **3**/**3′**. [c] **1** (0.3 mmol), **2a** (0.2 mmol), [{IrCp*Cl_2}$_2$] (8 mol%), $AgNTf_2$ (32 mol%), CsOPiv (50 mol%), PhCl (2 mL).

effect of the strongly electronegative substituents, an outcome that was also observed in the rhodium, ruthenium, and cobalt-catalyzed C–H activation reactions.[12] Interestingly, after screening various additives (Table S2 in the Supporting Information), we found that both the reactivity and regioselectivity could be controlled by the carboxylate salts. When the cesium salt with a bulky carboxylate ligand (CsOPiv) was used as the additive, the high regioselectivity was regained and the reactions predominantly gave the products with substitutes at the less hindered site.

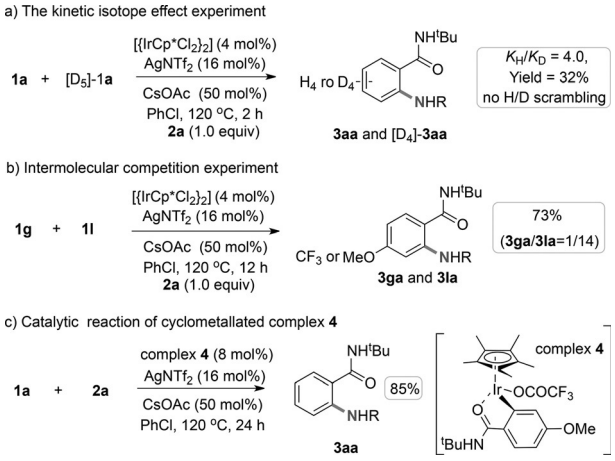
Next, we examined the scope of the alkyl azides **2** (Table 4). In addition to the linear azides with different functional groups, cyclic and branched alkyl azides were also well tolerated (**3 ab**–**ai**). α,α,α-Trisubstituted alkyl azides, substrates with a high steric hindrance, successfully formed the anthranilamides (**3 aj**–**al**), which are often difficult to synthesize through traditional Pd-catalyzed cross-coupling reactions.[13a] This is especially important because bulky alkyl-substituted amines are frequently found in biologically active molecules, in which they increase lipophilicity and/or improve metabolic stability of those molecules.[13b]

Table 4. Scope of alkyl azides **2**.[a]



[a] **1 a** (0.3 mmol), **2** (0.2 mmol), PhCl (2 mL), isolated yield.

The control experiments shows that i) $[\{Ir(Cp^*)Cl_2\}_2]$ and $AgNTf_2$ are essential for this reaction, while CsOAc is a great promoter, ii) $AgNTf_2$ and CsOAc cannot be replaced by AgOAc, iii) $AgNTf_2$ (8 mol%) is the lowest amount needed to afford the product in good yield, and iv) the radical inhibitor TEMPO (one equivalent) had no significant effect on the reaction (Table S3 in the Supporting Information). To study the reaction mechanism further, a series of experiments were also carried out (Scheme 1). A significant primary kinetic isotope effect was observed ($K_H/K_D = 4.0$), while no H/D scrambling occurred in the reaction between **1 a** and [D_5]-**1 a**. We then performed a competitive experiment, which revealed that electron-rich arenes are preferred in the reaction. The cyclometalated Ir^{III} complex **II** (Figure 2) was proposed to be involved in the catalytic cycle, because iridium complex **4** can catalyze direct C–H amination smoothly.



Scheme 1. Mechanistic studies.

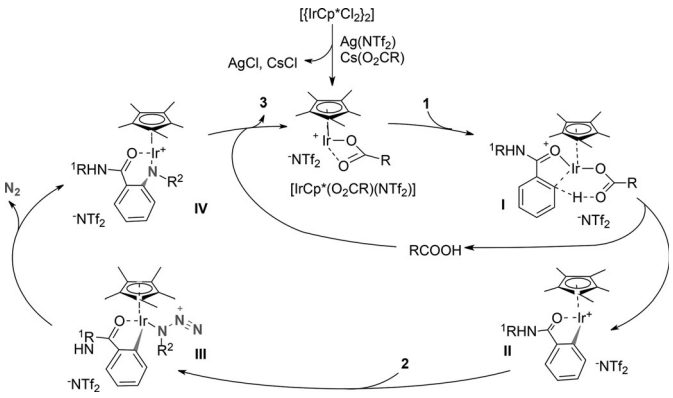
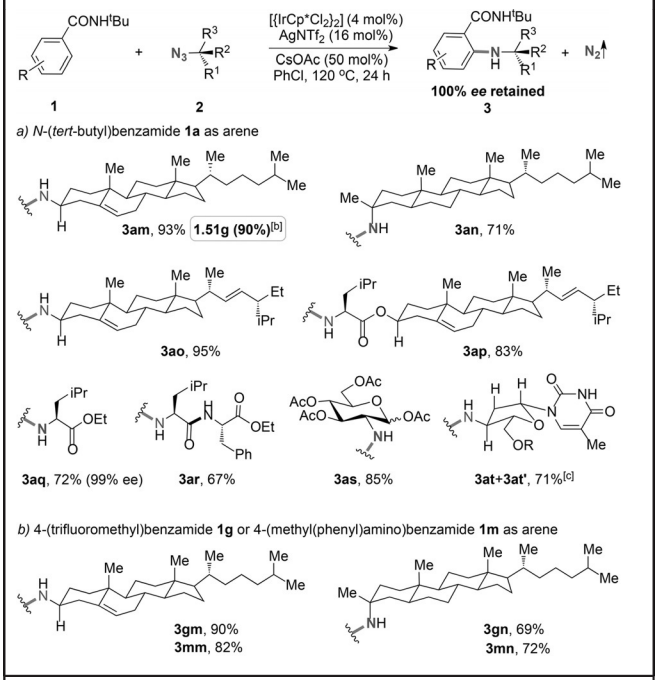


Figure 2. Proposed mechanism.

According to the aforementioned experiments and previous studies,[8,9] a possible mechanistic pathway is proposed in Figure 2. First, the active Ir species, $[IrCp^*(O_2CR)(NTf_2)]$, is formed by the $[\{IrCp^*Cl_2\}_2]$, $AgNTf_2$, and RCO_2Cs. This coordinates with benzamide **1**, inducing the C–H bond cleavage through transition state **I**, in which the regioselectivity can be controlled by both the substrate and carboxylate.[12a] This produces the cyclometalated Ir^{III} complex **II**. Coordination of the alkyl azide with the complex **II** forms **III**, which undergoes migratory insertion, releasing N_2, to give **IV**. Finally, protonolysis of **IV** provides the amination product **3** and regenerates the active Ir species.

Finally, we examined the compatibility of this method with several biological active molecules (Table 5). The azide derivatives of steroids, which were readily synthesized from their original bioactive structures (see the Supporting Information), were used as substrates to produce **3 am**–**ao** in high to excellent yields. Natural amino acid leucine and dipeptide-based alkyl azides were also compatible (**3 ap**–**ar**). Both of the derivative of glucosamine and drug zidovudine underwent this amination reaction with good yields (**3 as**–**at**). Steroid-based azides also reacted with both electron-withdrawing and -donating groups substituted benzamides to form the desired products in high yields (**3 gm**–**gn** and **3 mm**–**mn**). It is worth mentioning

Table 5. Applications of biologically relevant azides.[a]



[a] **1** (0.3 mmol), **2** (0.2 mmol), PhCl (2 mL), isolated yield. [b] Gram scale reaction. [c] Isolated yield of a mixture of **3at** and **3at′** (**3at**(R = Boc)/**3at′**(R = H) = 3.7/1), see Scheme S2 in the Supporting Information for details.

that the reaction can be scaled up to gram scale with a similarly high yield (**3am**; 1.51 g, 90%). Impressively, the chirality of all these products is 100% retained.

In summary, we have developed an Ir-catalyzed C–H amination of arenes using alkyl azides as the primary alkylamine source. We found that cesium carboxylate is crucial and can control both the reactivity and regioselectivity of the reaction. This method has very wide substrate scope; both linear and sterically hindered branched alkyl amino groups can be inserted to various benzamides with high yields. This reaction does not require oxidative conditions and is therefore friendly for many biologically relevant molecules, such as sugar, peptide, steroid, and thymidine derivatives. Moreover, a complete transfer of chirality is obtained under these reaction conditions. We expect that this method can serve as a robust method to synthesize anthranilamide derivatives, which are found in many drug molecules.

Experimental Section

To a screw-capped vial equipped with a spinvane triangular-shaped Teflon stir bar were added benzamide (**1**, 0.3 mmol), alkyl azides (**2**, 0.2 mmol), [{IrCp*Cl_2}$_2$] (6.4 mg, 4 mol%), $AgNTf_2$ (12.4 mg, 16 mol%), CsOAc (19.2 mg, 50 mol%), and chlorobenzene (2 mL) under N_2 conditions. The reaction mixture was stirred in a pre-heated oil bath at 120 °C for 24 h. The reaction was cooled to room temperature, filtered through a pad of celite, and then washed with CH_2Cl_2 (10 mL × 3). The solvents were removed under reduced pressure and the crude reaction mixture was purified by silica gel column chromatography with *n*-hexane/EtOAc as an eluent to give the desired product.

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Keywords: amides · azides · C–H activation · homogeneous catalysis · iridium

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