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Cobalt-Catalyzed Oxidative C-H/N-H Cross-Coupling: Selective and Facile Access to Triarylamines

Cong Du, Peng-Xiang Li, Xinju Zhu, Jiao-Na Han, Jun-Long Niu,* and Mao-Ping Song*

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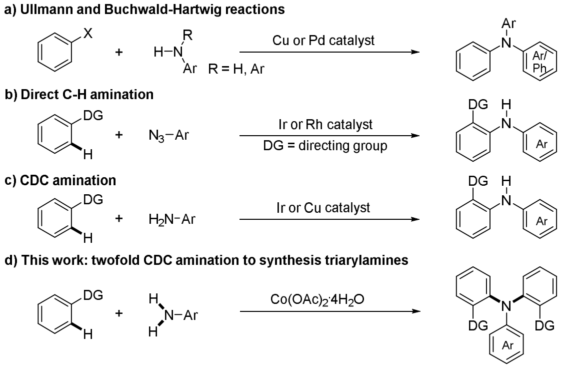
ABSTRACT: As a powerful synthetic tool for the formation of aromatic C–N bonds, the reported transition-metal-catalyzed direct C–H amination has been ineffective for the synthesis of triarylamines for a long time. Herein, an elegant strategy for the preparation of triarylamines was disclosed using inexpensive $Co(OAc)_2 \cdot 4H_2O$ as the catalyst. It is noteworthy that unactivated arenes and simple anilines were employed as the starting materials with good functional group tolerance. In addition, an organometallic Co(III) species was isolated and identified by X-ray crystallography, thus providing some in-depth insights into the mechanism.

KEYWORDS. cobalt catalysis, C-H activation, C-H/N-H cross-coupling, amination, triarylamine

Triarylamines are widely utilized in optoelectronic materials due to their prominent physical and electrochemical properties.[1] Conventional methods for their synthesis rely on the Pd or Cu catalyzed coupling reactions between aryl halides and anilines (Ullmann and Buchwald–Hartwig reactions), which typically encounters pre-activated starting materials, multistep syntheses and stoichiometric amounts of metal halide byproducts (Scheme 1a).[2] Although some technical improvements have been made in recent years,[3] these strategies still did not overcome the inherent restrictions. Therefore, it is highly desirable to develop an efficient and sustainable strategy for constructing these structures avoiding the use of aryl halides.

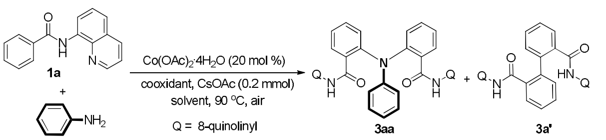
Alternatively, transition-metal-catalyzed C-H amination has emerged as a straightforward and step-economical synthetic methodology to form aromatic C-N bonds.[4] To date, several amine sources have been successfully employed in direct C_{sp2}-H amination (Scheme 1b).[5] In particular, transition-metal-catalyzed cross-dehydrogenative coupling (CDC) amination is highly desirable, in which non-preactivated aromatic amine were directly utilized as a reactant (Scheme 1c).[6] Pioneering studies in this area have been reported by Chatani[6a] and Chang,[6b] who have demonstrated Cu-mediated and Ir-catalyzed direct C–H amination with arylamine, respectively. Furthermore, Cu-mediated or catalyzed CDC amination with simple anilines was disclosed by Yu,[6c] Daugulis[6d] and Jana.[6e] Despite this great success, these strategies were unfortunately limited to the synthesis of diarylamine products.

On the basis of above discussion, the syntheses of triarylamines *via* direct C–H functionalization from readily available starting materials is highly attractive, especially if the first-row transition metals could be used as the catalysts. Recently, the earth-abundant cobalt catalysts have received particular attention due to their high activities in C–H functionalizations.[7] In particular, several catalytic systems have exhibited distinct reactivity on the construction of C-C bond.[8,9] Inspired by the previous progress, we attempted to explore an efficient method for the construction of C-N bond using simple cobalt salts as the catalyst.[10,11] Herein, a new protocol for cobalt-catalyzed CDC amination is reported, which allows for selective and facile access to triarylamines. (Scheme 1d). The main challenges for the strategy were listed as follows: 1) The cobalt catalyst is easily susceptible to the highly nucleophilic aniline, which may decrease the catalytic performance. 2) The sensitive anilines requires a finely tuned oxidant system. 3) The amination should be preferred to diarylamine products compared with the sterically congested triarylamines.



Scheme 1. Representative Synthetic Methods for Diarylamine and Triarylamine

Table 1. Optimization of the Cobalt-Catalyzed Amination



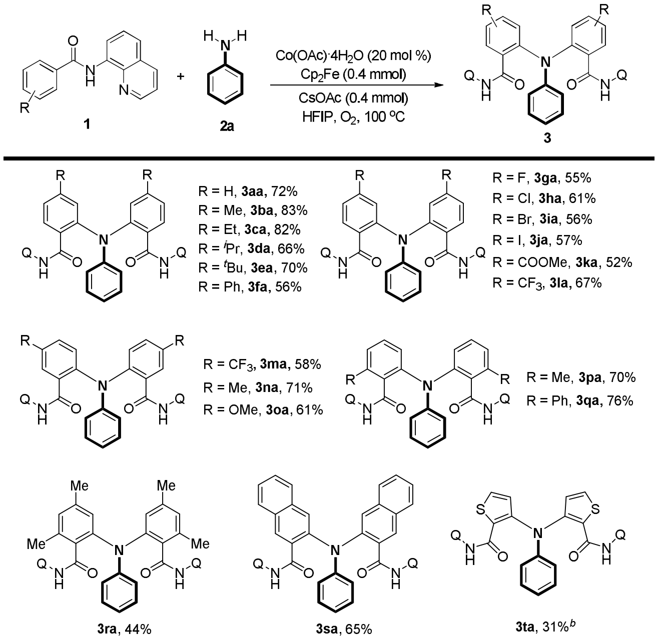
entry	solvent	cooxidant	ratio (**3aa**:**3a'**)^b	**3aa** (%)^c
1	MeOH	Ag_2CO_3	49:51	15
2	TFE	Ag_2CO_3	67:33	18
3	HFIP	Ag_2CO_3	94:6	25
4	HFIP	Ag_2SO_4	50:50	16
5	HFIP	$Mn(OAc)_2 \cdot 4H_2O$	43:57	9
6^d	HFIP	-	50:50	21
7^d	HFIP	Cp_2Fe	97:3	56

8[d,e]	HFIP	Cp_2Fe	100:0	72

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.2 mmol), $Co(OAc)_2{\cdot}4H_2O$ (20 mol %), cooxidant or [Ag] (0.4 mmol), CsOAc (0.2 mmol), solvent (1.0 mL), air atmosphere, 12 h, 90 °C. [b] The ratio was determined by ^1H NMR analysis. [c] Isolated yield. [d] O_2 atmosphere. [e] **2a** (0.6 mmol), CsOAc (0.4 mmol), HFIP (0.5 mL), 100 °C.

Our investigation to explore CDC aromatic amination began with the reaction of 8-aminoquinoline amide (**1a**) and aniline (**2a**) (Table 1 and Table S1-6 in the Supporting Information). Alcohol solvents (entries 1-3) can promote the amination reaction to produce triarylamine product (**3aa**), and non-protonic solvents, such as DMSO, PhMe, DCE, and CH_3CN were ineffective. Various catalytic systems were also investigated and exhibited a great effect on the reaction efficiency. The previously reported catalytic systems in this area, such as Co(II)/Ag(I), Co(II)/Mn(II or III), and Co(II)/O_2 catalytic system,[9,11] all displayed some catalytic activities, yet proved to be less effective (entries 4-6). When ferrocene (Cp_2Fe) was used as cooxidant, the yield of **3aa** was improved to 56% and the selectivity increased to 97:3 (entry 7). After a series of adjustments,[12] the highest yield (72%) and selectivity (100%) was obtained (entry 8). It is noteworthy that the diarylamine product was not detected during the optimization of the reaction conditions even with an excess of aniline.[12]

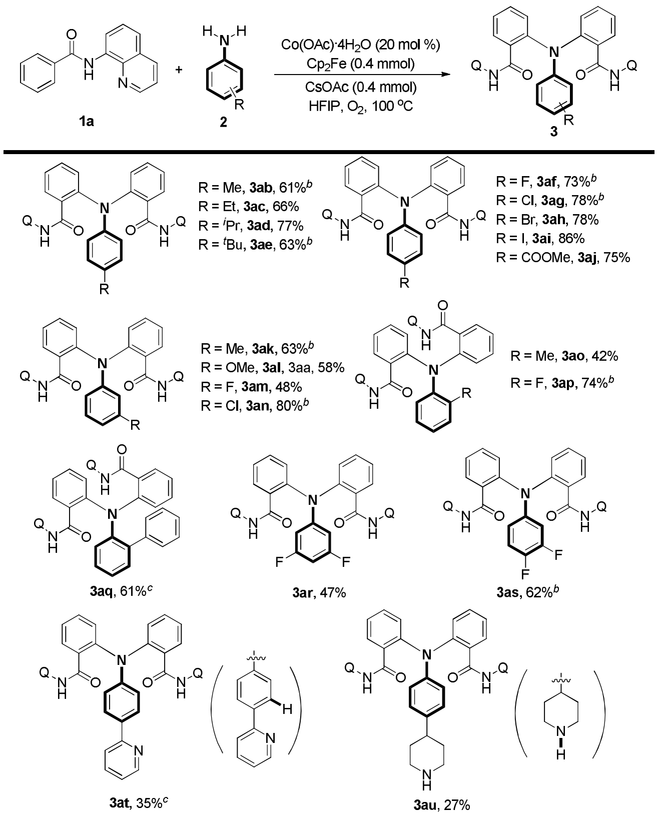
Table 2. Scope with Respect to Aromatic Amides



[a] Reaction conditions: **1** (0.2 mmol), **2a** (0.6 mmol), $Co(OAc)_2{\cdot}4H_2O$ (20 mol %), Cp_2Fe (0.4 mmol), CsOAc (0.4 mmol), HFIP (0.5 mL), O_2 atmosphere, 12 h, 100 °C. [b] 48 h.

With the optimized reaction conditions in hand, the scope of benzamide substrates was firstly explored (Table 2). A variety of functional groups, such as alkyl groups (Me, Et, ^{i}Pr, ^{t}Bu), halogens (F, Cl, Br, I), phenyl, ester, and CF_3, are well tolerated, providing aminated products in 52-83% yields (**3ba-la**). For unsymmetrical *meta*-substituted benzamides, the amination mainly occurred at the sterically less-hindered position (**3ma-oa**). This amination strategy further proved to be effective for *ortho*-monosubstituted benzamides (**3pa-qa**). Sterically congested disubstituted amides were also compatible with the transformation, thus furnishing the aminated product with two tetrasubstituted aromatic rings (**3ra**). Additionally, the substrates were not limited to substituted benzamides, direct amination of naphthalene and thiophene were also accessible (**3sa-ta**).

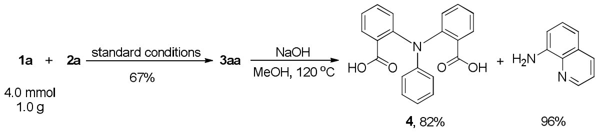
Table 3. Scope with Respect to Anilines



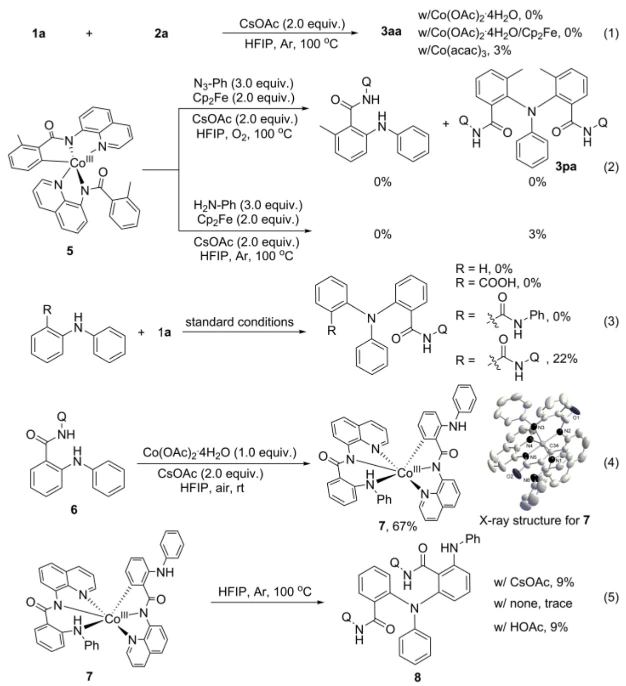
[a] Reaction conditions: **1a** (0.2 mmol), **2** (0.6 mmol), $Co(OAc)_2{\cdot}4H_2O$ (20 mol %), Cp_2Fe (0.4 mmol), CsOAc (0.4 mmol), HFIP (0.5 mL), O_2 atmosphere, 12 h, 100 °C. [b] 6 h. [c] 24 h.

Subsequently, the scope of the anilines was investigated (Table 3). A variety of substituted anilines were compatible with the Co-catalyzed amination regardless of the electronic properties of the substrates (**3ab-an**). It is noteworthy that the valuable groups (F, Cl, Br, I, COOMe) could be readily synthesized, which provides an opportunity for further elaboration. Although triarylamines containing three *ortho*-monosubstituted aromatic rings are highly challenging even for traditional methods, the *ortho*-substituted anilines underwent reactions successfully to give the corresponding aminated products (**3ao-aq**). Specially, the steric hindrance 2-aminodiphenyl (**2q**) could be coupled with benzamide. For disubstituted anilines, the reaction was also found to proceed smoothly. The strongly coordinating groups (arylpyridine and piperidine), which were employed as reagents for direct C-H functionalization,[9f,11b-c] were fully tolerated with high chem- and regioselectivity (**3at-au**).

To further demonstrate the potential application of this strategy, the gram-scale reaction of **1a** and **2a** was carried out and aminated product **3aa** was obtained in 67% yield under slightly modified conditions.[12] Next, the 8-aminoquinoline auxiliary could be easily removed and recovered by the simple base hydrolysis to afford the triarylamine (**4**) containing *ortho*-carboxy-substituted benzene rings (Scheme 2).

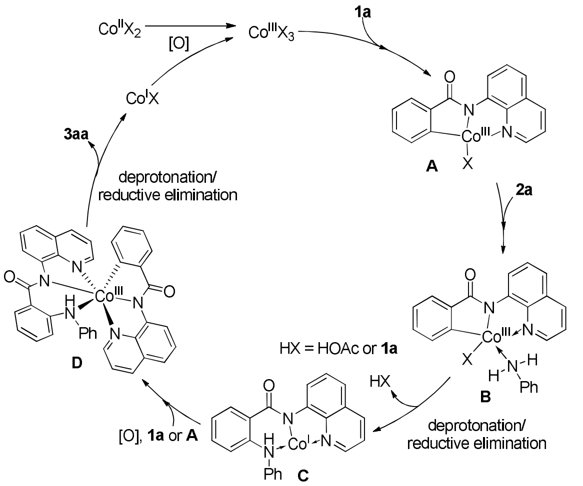


Scheme 2. Gram Scale Synthesis of Triarylamine (3aa) and Removal of the 8-Aminoquinoline Auxiliary



Scheme 3. Control Experiments and Mechanistic Studies

To provide some insights into the mechanism of the Co-catalyzed amination, a sequence of control experiments and mechanistic studies were carried out (Scheme 3). Under an argon atmosphere, $Co(OAc)_2·4H_2O$ and Cp_2Fe could not promote the reaction (Scheme 3, eq 1), whereas this transformation could occur with Co(II)/Ag(I), Co(II)/Mn(II or III), and Co(II)/O_2 catalytic system (Table 1). Without any cobalt salt, the amination did not occur.[12] These facts indicate that oxygen should be the actual oxidant and Cp_2Fe should be the cooxidant. When stoichiometric $Co(acac)_3$ was employed under Ar, the aminated product **3aa** could be obtained. Benzamide or cyclometalated Co(III) species **5**[13] did not react with phenylazide, whereas the organometallic species **5** and aniline could be transformed into **3pa** (Scheme 3, eq 2). These facts indicate that the reaction process probably initiates from a cobalt(III) species and should not involve a higher valence Co-imido species. Next, several diarylamines were attempted for C-H amination with benzamide **1a** (Scheme 3, eq 3), which showed only the compound **6** could give triarylamine product. These results indicate that this amination reaction might undergo a stepwise pathway and the 8-aminoquinoline directing group is the key moiety for subsequent amination. Furthermore cyclometalated Co(III) compound **7** could be obtained from the reaction of **6** with stoichiometric amounts $Co(OAc)_2·4H_2O$ at room temperature under air (Scheme 3, eq 4). When the compound **7** was placed under basic or acidic conditions, the dimerization product **8** was generated (Scheme 3, eq 5), which indicated that two C-H/N-H activation steps might occur in a stepwise manner involving an Co(III)/Co(I) cycle. In addition, a kinetic isotope effect (KIE) value of 1.6 was observed in the reaction of **1a** and $[D_5]$-**1a** with **2a**; 2.4 was observed in the reaction of **1a** and $[D_5]$-**1a** with **6**. Meanwhile, the KIEs of 1.2 (for the reaction of **1a** or $[D_5]$-**1a** and **2a**) and 1.5 (for the reaction of **1a** or $[D_5]$-**1a** and **6**) were observed in parallel reactions, suggesting that the C-H bond cleavage was not involved in the rate-limiting step.[12]



Scheme 4. Proposed Reaction Mechanism

Based on the above mechanistic studies and relevant literatures,[6,9-11] a plausible mechanism is proposed in Scheme 4. Initially, cobalt salt was oxidized to Co(III) species in the presence of Cp_2Fe and O_2, which activated the *ortho* C–H bond of benzamide to form the intermediate **A**. After the coordination of aniline, the intermediate **B** was obtained. As shown above in Scheme 3, when phenylazide was utilized instead of aniline, neither the diarylamine nor triarylamine could be detected. Also, benzamide (**1a**) and species **5** could be converted to triarylamines under an argon atmosphere. So, it is more likely that the intermediate **B** underwent deprotonation to give intermediate **C**. However, the dissociative diarylamine could not be detected during the reaction optimizations probably due to the strong chelation of the diarylamine **6** and cobalt salt. Subsequently, the intermediate **C** was oxidized to Co(III) species, followed by coordination with benzamide *via* C-H bond activation, which led to key intermediate **D**. It is also possible that intermediate **D** was generated *via* a ligand exchange between intermediate **C** and **A**. Finally, the aminated product **3aa** was obtained by deprotonation and reductive elimination, accompanied with the regeneration of Co(III) by the oxidant (O_2) and cooxidant (Cp_2Fe). Although the above mentioned experiments suggest a Co(III)/Co(I) cycle, the Co(IV)/Co(II) cycle could not be completely excluded.

In conclusion, we have successfully demonstrated Co(II)-catalyzed oxidative C-H/N-H cross-coupling reactions between unactivated arene and simple aniline to accomplish the triarylamines using commercially available $Co(OAc)_2·4H_2O$ as the catalyst and Cp_2Fe as the cooxidant. The significant aspects of our work include: 1) The sterically congested triarylamines were obtained with high chemoselectivity *via* twofold CDC amination. 2) A wide range of functional groups including electron-donating and electron-withdrawing groups were compatible under the current methodology. This protocol could provide new insight into Co-catalyzed C-H functionalization for the formation of C–N bonds, which paves the way for other Co-catalyzed carbon–heteroatom bond cross-coupling reactions.

ASSOCIATED CONTENT

Supporting Information. Experimental procedures, spectral data for new compounds and single crystal X-ray diffraction data for compound **3aa**, **7**. "This material is available free of charge via the Internet at http://pubs.acs.org."

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Notes

The authors declare no competing financial interest.

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