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C–H Activation

Cationic Cobalt(III)-Catalyzed Aryl and Alkenyl C–H Amidation: A Mild Protocol for the Modification of Purine Derivatives

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Abstract: A cationic cobalt(III)-catalyzed direct C–H amidation of unactivated (hetero)arenes and alkenes by using 1,4,2-dioxazol-5-ones as the amidating reagent has been developed. This transformation proceeds efficiently under external oxidant-free conditions with a broad substrate scope. Moreover, 6-arylpurine compounds, which often exhibit high potency in antimycobacterial, cytostatic, and anti-HCV activities, can be smoothly amidated, thus offering a mild protocol for their late stage functionalization.

Transition-metal-catalyzed directed C–H bond functionalization has emerged as a powerful synthetic methodology[1] that allows for the direct use of nonactivated substrates. Among the various transition-metal catalysts suitable for the directed C–H amidation of arenes, alkenes, and alkanes, Cp*-based (Cp* = pentamethylcyclopentadiene) catalytic systems of Rh^{III} and Ir^{III} have been well developed using organic azides as the amino source.[2] Despite their high catalytic activity and mild reaction conditions, these protocols suffer from the requirement of expensive rhodium and iridium catalysts, especially for large-scale synthesis. Since the first-row transition metals are earth abundant and cheaper than their 4d or 5d metal congeners, the development of efficient catalytic systems based on inexpensive first-row metals as alternatives to the precious metals is highly attractive.[3] Among the first-row transition metals, cobalt has emerged as one of the most promising catalysts for the direct C–H functionalizationthat leads to synthetically useful and cost-effective transformations.[4] Recently, low-valent cobalt catalysts were utilized by Nakamura, Yoshikai, Ackermann, and Daugulis for C–H transformations that were, until then, the domain of precious Rh, Pd, and Ru catalysts.[5] More recently, high-valent cobalt(III) catalysts were elegantly employed for chemoselective C–H functionalization by the groups of Matsunaga/Kanai,[6] Ackermann,[7] Glorius,[8] Ellman,[9] and Chang.[10]

With our continuing interest in inexpensive metal-catalyzed C–H nitrogenation reactions,[11,12] we have developed a cationic cobalt(III)-catalyzed C–H amidation of unactivated (hetero)arenes and alkenes that provides expedient access to diverse substituted amides in good to excellent yields (Figure 1). The significances of the present method are fourfold:

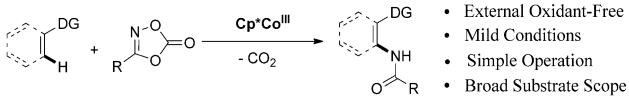

Figure 1. Cobalt-catalyzed C–H amidation.

1) Cost-effective, user-friendly cobalt catalyst is used under external oxidant-free conditions in the atmospheric environment. More attractively, compared to the reported Co catalysis, the present cationic cobalt(III) catalysts demonstrate high efficiency, which may promote the development of C–H functionalization by cationic Co catalysis.
2) 1,4,2-Dioxazol-5-ones are employed as amidating reagent with CO_2 as the single byproduct.
3) External oxidant is not required in this mild protocol.
4) The substrate scope is broad, including versatile arenes, heteroarenes and alkenes with high selectivity.

Thus, this methodology opens a new way to practical intermolecular C–N bond formation.

6-Arylpurine derivatives, which often exhibit high potency in antimycobacterial, cytostatic, and anti-HCV activities, are of high utility in medicinal chemistry. Besides, they also serve as useful building units in the synthesis of nucleosides.[13] In this regard, a selective installation of useful functional groups in 6-arylpurines skeleton for their late stage modification is very important. However, to date, only a few examples of direct C–H functionalization of 6-arylpurines have been reported.[14] We therefore commenced our studies by using 9-isopropyl-6-phenyl-9*H*-purine (**1a**) as a model substrate to react with 1.1 equivalents of diverse amidating reagents for the optimization of cobalt-catalyzed amidation reaction conditions (Table 1). After a series of preliminary screening, we were pleased to find that the desired amidation product (**3a**) could be obtained in 70% yield using 3-phenyl-1,4,2-dioxazol-5-one

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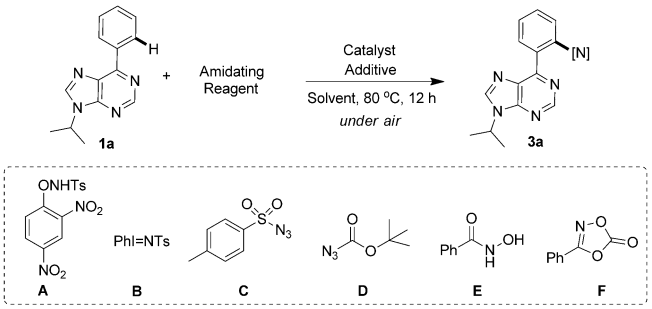
Table 1. Optimization of cobalt-catalyzed C–H amidation.[a]



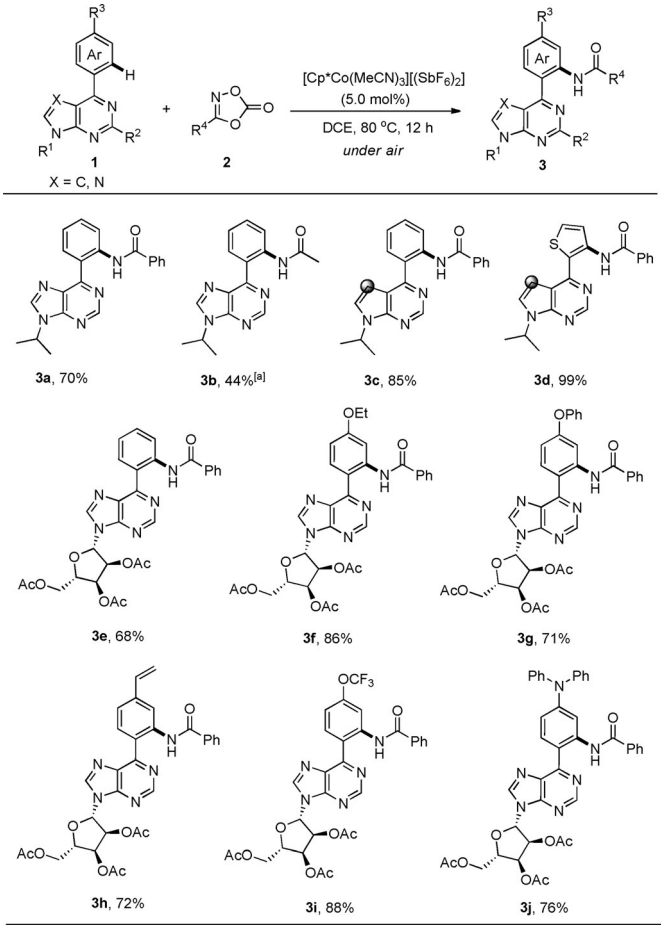
Entry	Amidating Reagent	Catalyst (mol%)	Solvent	Yield [%][b]
1	A	$[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5)	DCE	N.R.
2	B	$[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5)	DCE	N.R.
3	C	$[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5)	DCE	N.R.
4	D	$[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5)	DCE	N.R.
5	E	$[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5)	DCE	N.R.
6	F	$[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5)	DCE	70
7[c]	F	$[Cp^*Co(CO)I_2]$ (5)	DCE	30
8	F	$[Cp^*Co(CO)I_2]$ (5)	DCE	<5
9	F	$[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5)	MeCN	<1
10	F	$[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5)	THF	39
11	F	–	DCE	N.R.

[a] Reaction conditions: **1a** (0.2 mmol), amidating reagent (0.22 mmol), catalyst (2.5–5.0 mol%), solvent (1 mL), stirred at 80 °C under air for 12 h. [b] Isolated yields; N.R. = no reaction. [c] $AgSbF_6$ (10 mol%) was added.

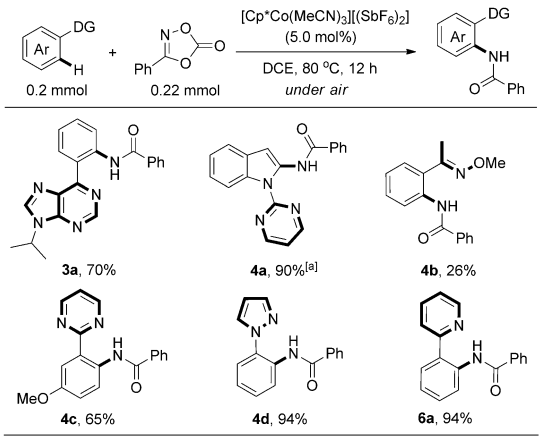
as an amidating reagent with cationic $[Cp^*Co(MeCN)_3][(SbF_6)_2]$—the preparation and catalysis of which were firstly reported by Glorius[8a]—as the catalyst (5 mol%) in DCE under air (Table 1, entries 1–6). Notably, 1,4,2-dioxazol-5-ones can be easily prepared from the corresponding hydroxamic acids, and can be used or stored without a special precaution.[15] Similar to acyl azides, they are known to generate *N*-acyl nitrenes through thermal or photo-initiated decomposition, which makes them a very good nitrene sources.[16] The direct C–N bond formation by using 1,4,2-dioxazol-5-one reagents has been elegantly developed by the groups of Bolm,[17] Dubé,[18] and Chang.[19] More recently, Chang and co-workers disclosed for the first time that these reagents can be used as efficient amino sources under a Cp^*Rh^{III}-catalyzed system.[19] Despite this progress, the application of 1,4,2-dioxazol-5-one as an amidating reagent in metal-catalyzed C–H amidation is still highly desired in organic synthesis. With this simple thought in mind, further optimization was conducted. It is noteworthy that the use of $[Cp^*Co(CO)I_2]$ (5 mol%) combined with $AgSbF_6$ (10 mol%) instead of cationic $[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (5 mol%) only afforded the desired product in 30% yield, which indicated that cationic $[Cp^*Co(MeCN)_3][(SbF_6)_2]$ is much more active than the in situ generated cationic Cp^*Co^{III} species in this reaction, and the reaction yield diminishes when the $AgSbF_6$ is omitted (Table 1, entries 7 and 8). DCE turned out to be the most effective solvent, while MeCN was found to be totally inhibitory (Table 1, entries 9 and 10). No reaction was observed in the absence of cobalt catalyst (Table 1, entry 11). Besides, several simple metal salts, such as $CoBr_2$, $Co(OAc)_2$, CuTc (Tc = thiophene-2-carboxylate), $[Fe(acac)_3]$ (acetylacetonate) were also tested, but they did not work (see the Supporting Information).

With the optimized conditions in hand, the substrate scope was then investigated. The amidation proceeded smoothly over a broad range of substrates (Scheme 1). Amidating reagent, 1,4,2-dioxazol-5-one, with a methyl group at the 3-position can also react smoothly to provide the corresponding product (**3b**). Notably, analogous substrates lacking a N7 nitrogen were amidated in high yields, which suggested that the N1 rather than the N7 atom serves as a chelating group to lead to the amidation (**3c**–**3d**). In addition, 6-(4-substituted phenyl)purine derivatives bearing a pendant *N*9-(*O*-acetyl-β-ribofuranosyl) group were also successfully amidated in good to excellent yields (**3e**–**3j**).

To expand the utility of this methodology, we turned our attention to study the reactivity of substrates with different directing groups (Scheme 2). Gratifyingly, this protocol can be applied for efficient C2 selective C–H amidation of indole with only 2.5 mol% catalyst loading (**4a**). Substrates using oxime ether as the directing group can also be functionalized (**4b**) albeit in low yield. Phenyl derivatives bearing pyrimidine, pyra-



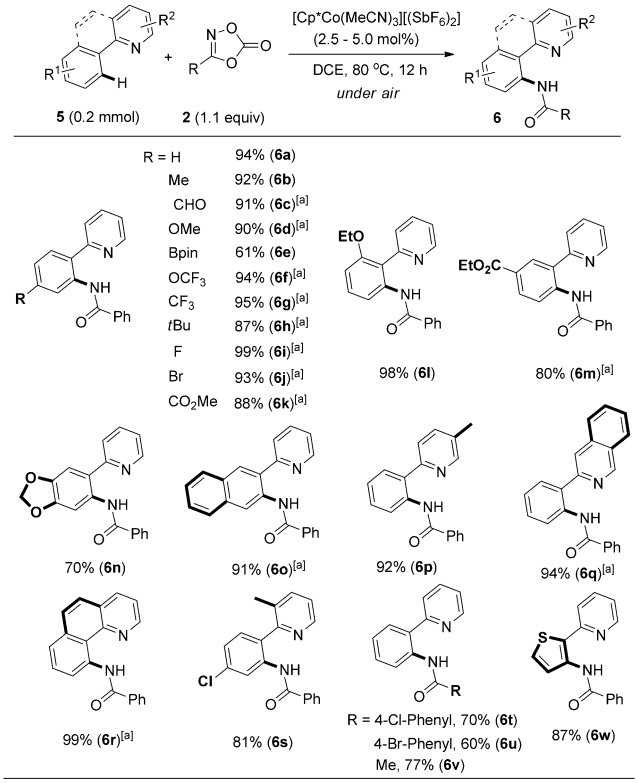
Scheme 1. Cobalt-catalyzed C–H amidation of 6-arylpurine. [a] $[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (10 mol%) was used 24 h.



Scheme 2. Cobalt-catalyzed C–H amidation with different directing groups. [a] $[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (2.5 mol%) was used.

zole, and pyridine as directing groups were effective and gave the corresponding products in good to excellent yields.

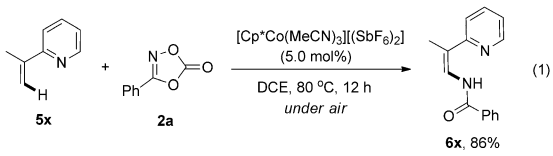
To display the excellent functional group tolerance of the present method, we therefore extended our protocol utilizing diverse 2-phenylpyridines as substrates. As depicted in Scheme 3, various 2-phenylpyridines regardless of electron-donating or -withdrawing groups on the aromatic ring reacted smoothly to furnish the products in good to excellent yields (**6a**–**6l**). The valuable functional groups tolerance, including methyl, methoxyl, aldehyde, borate, fluoro, bromo, ester



Scheme 3. Cobalt-catalyzed C–H amidation of 2-phenylpyridines. [a] $[Cp^*Co(MeCN)_3][(SbF_6)_2]$ (2.5 mol%) was used.

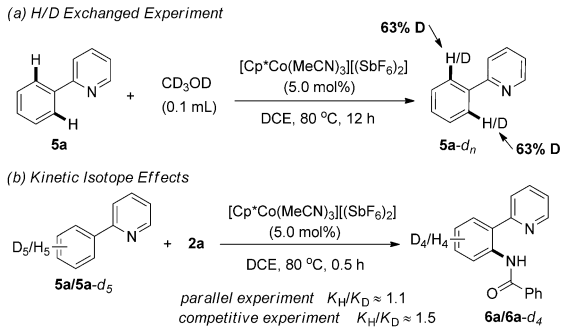
groups, offers ample opportunity for further derivatization. Additionally, when *meta*-substituted 2-phenylpyridines were used, a high regioselectivity favoring activation at the less hindered C–H bond was observed with the formation of the products **6m**–**6o**. The high efficiency was not affected even when the pyridine rings had additional substituents (**6p** and **6q**). In addition, the amidation of benzo[h]quinoline was almost quantitative (**6r**). A substrate bearing substituents at both the phenyl and the pyridine ring was also tolerated (**6s**). A variation on the amidating reagent was subsequently examined. Substituents (chloro and bromo) at the phenyl moiety, or 1,4,2-dioxazol-5-one with an alkyl substituent at the 3-position afforded the corresponding products in good yields (**6t**–**6v**). Notably, amidation of a thiophene derivative bearing a pyridine directing group is also feasible, leading to the corresponding product in 87% yield (**6w**).

Delightfully, the amidation reaction worked not only with (hetero)arenes, but also with olefins through alkenyl C–H activation. 2-(Propenyl)pyridine (**5x**) displayed a good reactivity in this reaction, selectively furnishing the desired product **6x** in 86% yield [Eq. (1)].

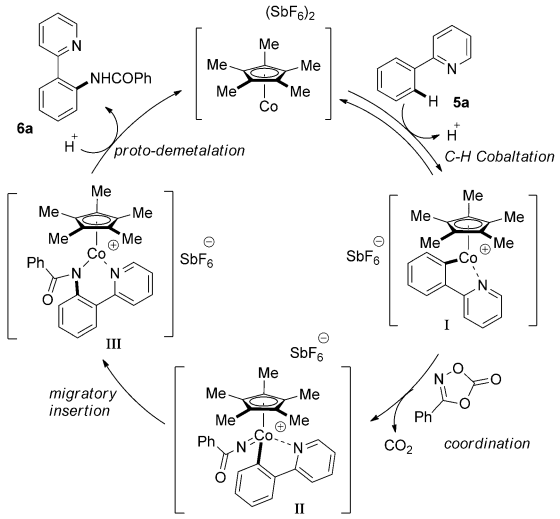


To gain insights into the mechanism, further experiments were conducted (Scheme 4). A significant level of deuterium incorporation (63%) was observed at the *ortho* position of 2-phenylpyridine when it was subjected to the present cobalt-catalyzed system in CD_3OD in the absence of amidating reagent, which indicated that the cobalt-mediated C–H bond cleavage is reversible (Scheme 4a). On the other hand, low level of primary kinetic isotope effects were observed (K_H/K_D = 1.1–1.5; Scheme 4b). The observed results suggested that the C–H bond cleavage may not be involved in the rate-limiting step.

Based on the mechanistic studies and the relevant reports,[6–10] a plausible catalytic cycle of the present cobalt-catalyzed C–H amidation reaction is proposed in Scheme 5. The reaction initiates with the cobalt-catalyzed C–H bond cleavage



Scheme 4. Mechanistic studies.



Scheme 5. Proposed catalytic cycle.

to furnish the five-membered metallacyclic intermediate **I**, which is subsequently coordinated by 3-phenyl-1,4,2-dioxazol-5-one to form the intermediate **II** with the release of CO_2. Subsequently, migratory insertion of intermediate **II** affords the intermediate **III**. Finally, protodemetalation of intermediate **III** liberates the desired product along with the regeneration of the active catalyst.

In summary, we have demonstrated an efficient cobalt-catalyzed C–H amidation of unactivated (hetero)arenes and alkenes by using 1,4,2-dioxazol-5-ones as amidating reagents. This reaction proceeded efficiently under external oxidant-free conditions with ample substrate scope, as well as excellent chemo- and regioselectivity. The employment of cationic cobalt(III) catalysts enables the high efficiency of this transformation, which may promote the development of C–H functionalization by cationic Co catalysis.

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Keywords: amidation · C–H functionalization · C–N bond formation · cobalt · purine compounds

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