



Communication

Direct synthesis of benzoxazinones via Cp*Co(III)-catalyzed C–H activation and annulation of sulfoxonium ylides with dioxazolones

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ABSTRACT

A highly novel and direct synthesis of benzoxazinones was developed via Cp*Co(III)-catalyzed C–H activation and [3+3] annulation between sulfoxonium ylides and dioxazolones. The reaction is conducted under base-free conditions and tolerates various functional groups. Starting from diverse readily available sulfoxonium ylides and dioxazolones, a variety of benzoxazinones could be synthesized in one step in 32%–75% yields.

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Benzoxazinones are an important class of fused heterocycles that are widely present in diverse natural products and pharmaceuticals [1], such as TEI-5624 [2], Cetilistat [3], and AX-9657 (Fig. 1) [4]. In addition, they are also valuable intermediates in organic synthesis [5]. For example, 2-substituted benzoxazinones could be converted to bioactive quinazolinone derivatives. As a result, developing efficient protocols for the synthesis of benzoxazinones has been an important research topic in organic synthesis [6–9]. Conventionally, benzoxazinones can be synthesized through the annulation of *N*-acyl anthranilic acid or anthranilic acid [7]. Furthermore, benzoxazinones can also be prepared via Pd-catalyzed carbonylation of *ortho*-haloanilines [8] and oxidation of indoles [9]. Despite all these progress, most of these approaches tend to suffer from either requiring prefunctionalized substrates or harsh reaction conditions. Recently, transition-metal-catalyzed C–H activation has become a powerful synthetic tool in contemporary organic synthesis due to its high atom/step economy [10]. Hence, developing direct and efficient methods for the construction of benzoxazinones via C–H activation mode can be of great significance.

Indeed, the preparation of benzoxazinones through transition-metal-catalyzed C–H activation has been reported (Scheme 1) [11]. Lloyd-Jones, Booker-Milburn [11a] and Yu [11b] reported that

benzoxazinones can be synthesized via Pd(II)-catalyzed C–H carbonylation of aniline derivatives (Scheme 1a). It should be noted that these reactions require the use of toxic carbon monoxide gas. Kim [11c], Wang [11d] and Dong [11e] reported Rh(III) or Ir(III)-catalyzed C–H amidation of aldehydes or their equivalents to deliver 2-aminobenzaldehydes, which could further undergo intramolecular oxidative cyclization to give benzoxazinones (Scheme 1b). In these cases, the additional oxidation step was essential for the synthesis of benzoxazinones. Very recently, Zou reported a Rh(III)-catalyzed cascade reaction of benzoic acids with dioxazolones for the construction of 2,5-substituted benzoxazinones (Scheme 1c) [11g]. Though highly efficient, due to the cost issues associated with the use of palladium, iridium and rhodium metals, it would be highly desirable if similar transformations can be accomplished by employing cheaper catalysts. Recent studies have shown that Cp*Co(III) catalysts are not only promising alternatives to Cp*Rh(III) catalysts, they also show unique catalytic reactivity in C–H activation [12]. It is worth noting that enormous advances in this area have been accomplished by the groups of Cheng [13], Kanai [14], Matsunaga [15], Chang [16], Ackermann [17], Li [18], Glorius [19], Shi [20] and others [21]. Inspired by these studies and in continuation of our research on C–H bond functionalizations [22], we herein present a direct and practical method for the synthesis of benzoxazinones via Cp*Co(III)-catalyzed C–H activation and [3+3] annulation reaction of sulfoxonium ylides [23] with dioxazolones (Scheme 1d). It should be mentioned that during the course of our investigation, Li

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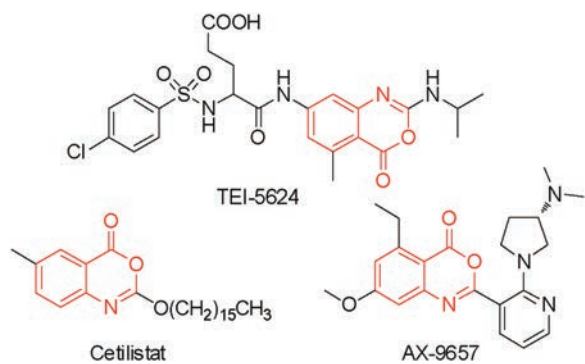
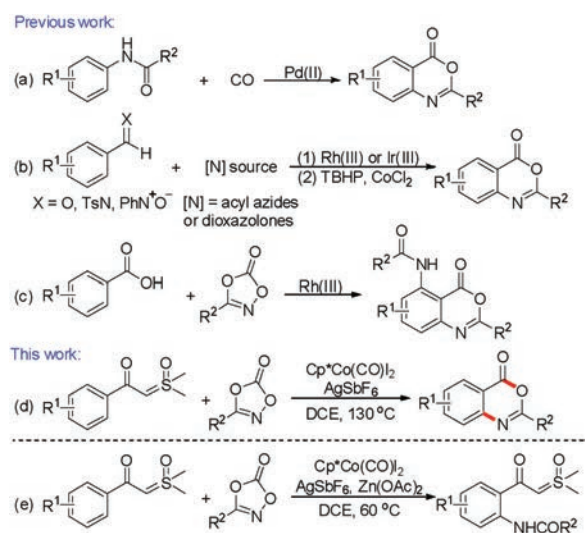


Fig. 1. Biologically active benzoxazinones.

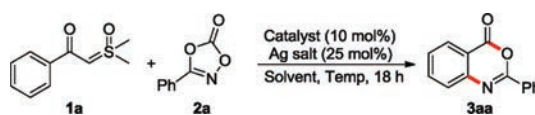


Scheme 1. Comparison of previous works with this work.

reported a similar cobalt(III)-catalyzed C–H functionalization of sulfoxonium ylides with dioxazolones under different reaction conditions and only amidation products were obtained (Scheme 1e) [24].

Our initial plan was to realize $Cp^*Co(III)$ -catalyzed C(aryl)–H amidation of sulfoxonium ylides with dioxazolones as the amidating reagents. Much to our surprise, when we treated sulfoxonium ylide **1a** (0.2 mmol) with dioxazolone **2a** (0.3 mmol) in the presence of $Cp^*Co(CO)_2$ (0.02 mmol), and $AgSbF_6$ (0.05 mmol) in DCE (2 mL) under N_2 at 130 °C for 18 h, benzoxazinone derivative **3aa** was obtained in 69% yield (Table 1, entry 1). Encouraged by this result, we further studied other reaction conditions for the transformation. It was found that introduction of additives such as NaOAc, $Zn(OAc)_2$, KOAc and PivOH could not improve the yield of **3aa** (entries 2–5). Subsequent screening of solvents suggested that DCE was the most efficient medium for the transformation, while other solvents such as 1,4-dioxane, CH_3CN , and TFE were totally ineffective (entries 6–8). Other cobalt catalysts such as $Co_2(CO)_8$ and $Co(acac)_3$ were also examined and found to be ineffective for this reaction (entries 9 and 10). It was observed that the choice of Ag salt had an important effect on the reaction. When $AgOTf$ was used in place of $AgSbF_6$, **3aa** could be isolated in 43% yield (entry 11). In sharp contrast, no desired product was detected when $AgOAc$ was employed (entry 12). The yield of **3aa** was not improved under elevated (67%, 140 °C) or lower (58%, 120 °C) reaction temperatures (entries 13 and 14). It is worthwhile to note that when the reaction temperature was

Table 1
Optimization of the reaction conditions.^a



Entry	Catalyst	Ag salt	Solvent	Additive	Yield (%) ^b
1	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	None	69
2	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	NaOAc	65
3	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	$Zn(OAc)_2$	61
4	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	KOAc	63
5	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	PivOH	58
6	$Cp^*Co(CO)_2$	$AgSbF_6$	1,4-dioxane	None	Trace
7	$Cp^*Co(CO)_2$	$AgSbF_6$	CH_3CN	None	ND ^g
8	$Cp^*Co(CO)_2$	$AgSbF_6$	TFE	None	ND ^g
9	$Co_2(CO)_8$	$AgSbF_6$	DCE	None	ND ^g
10	$Co(acac)_3$	$AgSbF_6$	DCE	None	ND ^g
11	$Cp^*Co(CO)_2$	$AgOTf$	DCE	None	43
12	$Cp^*Co(CO)_2$	$AgOAc$	DCE	None	ND ^g
13 ^c	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	None	67
14 ^d	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	None	58
15 ^e	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	None	71
16 ^f	$Cp^*Co(CO)_2$	$AgSbF_6$	DCE	None	68
17	None	$AgSbF_6$	DCE	None	ND ^g
18	$Cp^*Co(CO)_2$	None	DCE	None	ND ^g

^a Unless otherwise noted, the reactions were performed using **1a** (0.2 mmol), **2a** (0.3 mmol), $Cp^*Co(CO)_2$ (10 mol%), $AgSbF_6$ (25 mol%) and additive (20 mol%) in a solvent (2 mL) at 130 °C for 18 h under N_2 .

^b Isolated yield.

^c At 140 °C.

^d At 120 °C.

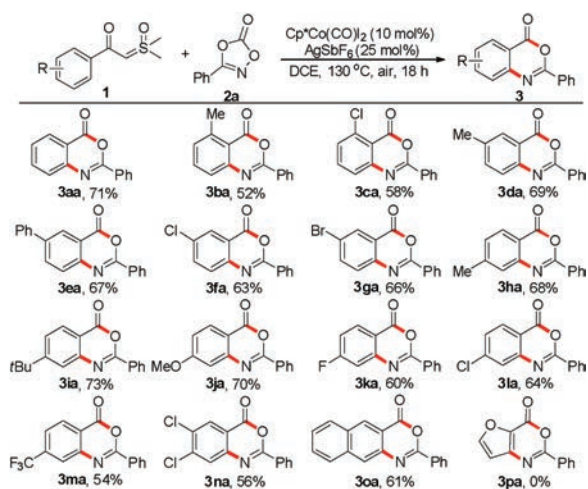
^e Under air atmosphere.

^f Under O_2 atmosphere.

^g ND: not detected.

lowered to 70 °C, only *ortho* C–H amidation product of sulfoxonium ylide was obtained, and no cyclization product **3aa** was detected. When the reaction was carried out under an air atmosphere, the yield of **3aa** could be improved to 71% (entry 15). Control experiments revealed that both $Cp^*Co(CO)_2$ and $AgSbF_6$ were necessary for the formation of **3aa** (entries 17 and 18). It should be mentioned that when another amidating reagent 5-phenyl-1,3,2,4-dioxathiazole 2-oxide was employed in place of **2a**, no desired product was detected (not shown in Table 1). Therefore, we decided to set reacting **1a** with 1.5 equiv. of **2a** in the presence of 10 mol% of $Cp^*Co(CO)_2$ and 25 mol% of $AgSbF_6$ in DCE under air at 130 °C for 18 h as our optimized reaction conditions. It should be noted that only 5 mol% of cobalt catalyst is enough to obtain similar yields when the reaction is run at larger scale, details see Supporting information.

We next examined the substrate scope with respect to the sulfoxonium ylides under the optimized conditions, and the results are summarized in Scheme 2. It was observed that sulfoxonium ylides substituted with electron-donating or electron-withdrawing groups on the phenyl rings all reacted smoothly with dioxazolone **2a** to provide the corresponding benzoxazinones in moderate to good yields (**3aa–3oa**). From the table, we can see that when *ortho*-substituted sulfoxonium ylides were used as the substrates, the corresponding products were obtained with lower yields (**3ba** and **3ca**), which is perhaps due to steric hindrance. As for the *meta*-substituted sulfoxonium ylides, the C–H activation occurred exclusively on the less hindered site (**3da–3ga**). It should be mentioned that 3,4-disubstituted sulfoxonium ylide **1n** was found to be a viable substrate, affording the desired product **3na** in 56% yield. Furthermore, sulfoxonium ylide with a naphthalene ring could also participate in the reaction to give the desired product **3oa** in 61% yield. Unfortunately, heterocyclic sulfoxonium ylides

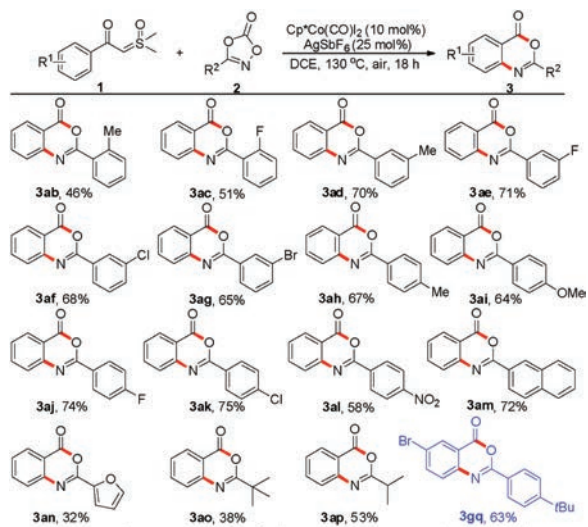


Scheme 2. Substrate scope of sulfoxonium ylides. Reaction conditions: **1** (0.2 mmol), **2a** (0.3 mmol), Cp*Co(CO)₂ (10 mol%), AgSbF₆ (25 mol%), DCE (2 mL), 130 °C, air, 18 h. Isolated yield.

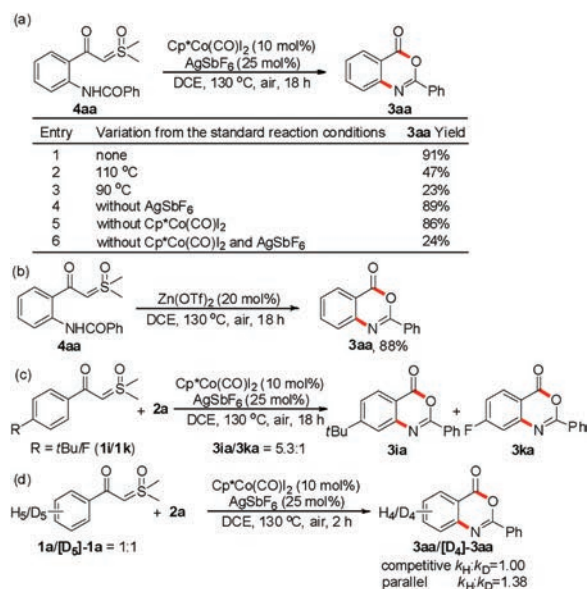
such as α -2-furoyl sulfoxonium ylide were found to be incompatible for the system (Scheme 2, **3pa**).

Subsequently, the scope of dioxazolones was explored as well (Scheme 3). From the scheme, we can see that aryl-substituted dioxazolones bearing either electron-donating (methyl and methoxy) or electron-withdrawing (fluoro, chloro, bromo, and nitro) groups are all viable in this system, delivering the corresponding products in moderate to good yields (**3ab–3al**). However, 2-methylphenyl and 2-fluorophenyl-substituted dioxazolones (**2b** and **2c**) were less reactive possibly due to steric effect. It should be noted that naphthalene ring and heterocycle substituted dioxazolones also reacted smoothly with **1a** to deliver **3am** and **3an** in yields of 72% and 32%, respectively. We were also delighted to find that alkyl-substituted dioxazolone **2o** and **2p** also proved to be viable substrates, giving **3ao** and **3ap** in 38% and 53% yield, respectively. It is worthwhile to point out that compound **3gq**, a high-density lipoprotein elevator [25], could also be synthesized in 63% yield under our reaction conditions.

To elucidate the reaction mechanism, several mechanistic experiments were conducted (Scheme 4). At first, to probe the



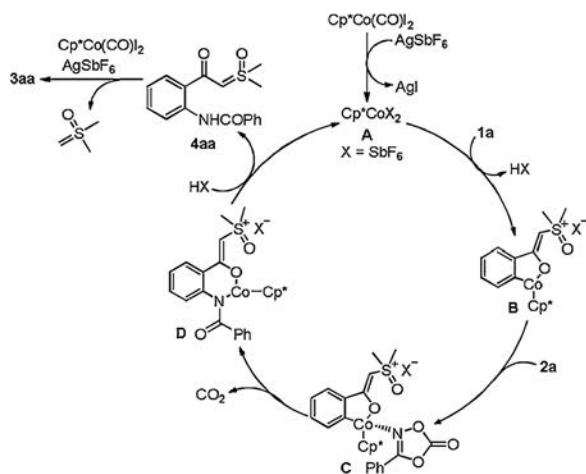
Scheme 3. Substrate scope of dioxazolones. Reaction conditions: **1** (0.2 mmol), **2** (0.3 mmol), Cp*Co(CO)₂ (10 mol%), AgSbF₆ (25 mol%), DCE (2 mL), 130 °C, air, 18 h. Isolated yield.



Scheme 4. Mechanistic studies.

annulation process, NH-benzoyl-substituted sulfoxonium ylide **4aa** was prepared [24]. Treatment of the compound **4aa** with 10 mol% of Cp*Co(CO)₂ and 25 mol% of AgSbF₆ in DCE at 130 °C afforded product **3aa** in 91% yield, indicating that **4aa** is an intermediate in the reaction (Scheme 4a, entry 1). Lowering the reaction temperature significantly decreased the yield of **3aa** (Scheme 4a, entries 2 and 3), suggesting that high temperature is necessary for the cyclization process to proceed. It is worthwhile to note that in the presence of Cp*Co(CO)₂ or AgSbF₆, **3aa** could be obtained in excellent yield (Scheme 4a, entries 1, 4 and 5). In sharp contrast, when both Cp*Co(CO)₂ and AgSbF₆ are omitted, **3aa** could only be obtained in 24% yield (Scheme 4a, entry 6). These results revealed that Cp*Co(CO)₂ and AgSbF₆ may play an important role in the cyclization process. We suspected that Cp*Co(CO)₂ and AgSbF₆ may serve as Lewis acid to activate the carbonyl of sulfoxonium ylides. To verify this conjecture, we performed a control experiment with Lewis acid Zn(OTf)₂ (Scheme 4b). Gratifyingly, treatment of **4aa** with 20 mol% of Zn(OTf)₂ delivered **3aa** in 88% yield, showing the importance of Lewis acid participation. Then, an intermolecular competition reaction between electronically different sulfoxonium ylides **1i** and **1k** were conducted (Scheme 4c). The result suggested that more electron-rich substrate **1i** was favored for the reaction. Furthermore, we carried out several experiments to study the kinetic isotopic effect (KIE). Both intermolecular competition reaction ($k_H/k_D = 1.00$) and parallel reaction ($k_H/k_D = 1.38$) gave relatively small KIE values, revealing that C–H activation may not be involved in the turnover-limiting step (Scheme 4d). Combined with Li's work [24], we suspected that the cyclization of intermediate **4** may be the rate-limiting step of the whole reaction process.

On the basis of mechanistic experiments and previous literatures [22f,26], a plausible mechanism for the reaction is proposed in Scheme 5. First, the active cationic Co(III) complex **A** is generated upon treatment of Cp*Co(CO)₂ with AgSbF₆. Then, **A** coordinates to the carbonyl oxygen of **1a** and activates the *ortho* C–H bond to give a five-membered cobaltacyclic intermediate **B**. Subsequent coordination of the nitrogen of dioxazolone **2a** to the Co center delivers the intermediate **C**, which subsequently is converted to intermediate **D** via extrusion of a molecule of CO₂ and migratory insertion process. Next, protonolysis of **D** regenerates the Co(III) catalyst for a new catalytic cycle and releases the key intermediate **4aa**. Finally, intramolecular nucleophilic attack of the



Scheme 5. Possible mechanism.

amide carbonyl oxygen on the sulfoxonium ylide carbonyl group with the assistance of $\text{Cp}^*\text{Co}(\text{CO})_2$ or AgSbF_6 gives the product **3aa** via the loss of a molecule of Corey's ylide (please see the Supporting information for its trapping experiment) [26d].

In conclusion, we have developed a highly novel and direct route for the synthesis of benzoxazinones via $\text{Cp}^*\text{Co}(\text{III})$ -catalyzed C–H activation and [3 + 3] annulation of sulfoxonium ylides with dioxazolones. This strategy employs cost-effective and air-stable $\text{Cp}^*\text{Co}(\text{CO})_2$ as the catalyst and features operational simplicity. Another remarkable feature is that the reaction is conducted under base-free conditions. Starting from diversely substituted sulfoxonium ylides and dioxazolones, a variety of benzoxazinones could be prepared in one step in moderate to good yields. Further mechanistic investigation and application of this protocol are in progress, and the results will be forthcoming.

Declaration of competing interest

The authors report no declarations of interest.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.ccl.2020.09.020>.

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