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Sodium chlorate as a viable substoichiometric oxidant for cobalt-catalyzed oxidative annulation of aryl sulfonamides with alkynes

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### ARTICLE INFO

### ABSTRACT

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Keywords: Cobalt catalysis Sultam C–H activation Oxidative annulation In this work, NaClO<sub>3</sub> is demonstrated for the first time as an efficient and versatile oxidant in the catalytic C–H activation reaction. By using sodium chlorate as the oxidant, a highly regioselective cobalt-catalyzed oxidative annulation of aryl sulfonamides with alkynes has been developed and can be extended to the annulation of benzamide.

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Recently, transition-metal-catalyzed C-H functionalization has emerged as an efficient and powerful strategy for the construction of carbon-carbon and carbon-heteroatom bonds.<sup>1</sup> In particular, transition-metal-catalyzed oxidative C-H activation has attracted much attention due to their high atom- and stepeconomy and versatility in organic transformations.<sup>2</sup> In these reactions, oxidation represents a fundamental and important step to constitute the catalytic cycle, in which reoxidation of the catalytic metal center from a relatively low oxidation state to a higher oxidation state is accomplished. Furthermore, oxidant could work as an important variant to dictate the reaction selectivity as well.<sup>3</sup> Thus, various external oxidants, including metal salts (e.g., Cu(OAc)<sub>2</sub>, Ag<sub>2</sub>CO<sub>3</sub>, etc.), peroxides, hypervalent iodine(III) reagents, DDQ, and O<sub>2</sub> or air, are often employed. Although these common oxidants could support most of the currently developed reactions, they still suffer from some limitations, including: 1) The concomitant side-reactions caused by these oxidants complicate the transformations. For example, undesired acetoxylation could occur when  $PhI(OAc)_2$  and Cu(OAc)<sub>2</sub> are employed<sup>4</sup> and N-oxidation of the nitrogencontaining substrates could take place in the presence of peroxides;<sup>5</sup> 2) The wastes released from oxidants, such as heavy metals and aromatic iodides, would be harmful to environment; and 3) Stoichiometric or excess amounts of oxidants are required in most cases, thus diminishing the appeal of oxidative C-H activation. Therefore, exploitation of lowly toxic, inexpensive and environmentally benign oxidants that could work operatively with the catalytic system remains an important task in oxidative C-H activation.

Sodium chlorate is usually used in metal extraction and paper bleaching in modern chemical industry.<sup>6</sup> Despite its good oxidative ability, NaClO<sub>3</sub> is seldom used as an oxidant in organic synthesis. To the best of our knowledge, whether NaClO<sub>3</sub> can be applicable as an oxidant in transition-metal-catalyzed oxidative C-H functionalization has never been reported. We envisioned that NaClO<sub>3</sub> would be an ideal oxidant in catalytic C-H activation on the basis of the following considerations: 1) The high oxidation state of Cl (potential reduction from Cl(+5) to Cl(-1)) may minimize the amount of oxidant used; and 2) NaClO<sub>3</sub> is an inexpensive, easily-available and -handling oxidant, hence improving the reaction practicality.

On another hand, transition-metal-catalyzed oxidative C-H activation/annulation of arenes with alkynes is regarded as a highly efficient and expedient tactics to construct diverse carbocycles and heterocycles. In the past decade, numerous noble metals,<sup>7,8</sup> including Pd, Rh, and Ru, have been successfully employed in these catalytic annulation reactions. From the viewpoint of economy and sustainable chemistry, using an inexpensive and abundant metal to accomplish these catalytic annulations should be more attractive and practical.<sup>9</sup> Herein, we disclose a regioselective cobalt-catalyzed oxidative C-H activation/annulation of sulfonamides with alkynes by using NaClO<sub>3</sub> as the oxidant to synthesize sultams which are common motifs in pharmaceuticals and biologically active compounds (Scheme 1).<sup>10,11</sup> Notably, this is the first demonstration of NaClO<sub>3</sub> as an efficient and versatile oxidant in the catalytic C-H activation reactions.

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### Tetrahedron



**Scheme 1** Oxidative annulation of aryl sulfonamides with both terminal and internal alkynes by using NaClO<sub>3</sub> as the oxidant.

Our initial optimization focused on the effect of oxidants on the cobalt-catalyzed annulation of aryl sulfonamide 1a and phenyl acetylene 2a (Table 1). It was found that a mixture of regioisomers 3a and 3a' (67/29) could be obtained in the presence of Co(OAc)<sub>2</sub> (20 mol%), Ag<sub>2</sub>CO<sub>3</sub> (1.5 equiv), and PivOH (1.0 equiv) in 1,4-dioxane at 120 °C (Table 1, entry 1). The replacement of Ag<sub>2</sub>CO<sub>3</sub> with Ag<sub>2</sub>O could slightly improve the selectivity of products, while AgOAc gave a diminished ratio of 3a/3a' (entries 2 and 3). All the other common oxidants in our tests, including Mn(OAc)<sub>2</sub>/O<sub>2</sub>, Cu(OAc)<sub>2</sub>, PhI(OAc)<sub>2</sub>, Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, failed to promote this reaction (entries 4-7). To our delight, 3a was afforded in 87% yield as a single regioisomeric product when NaClO<sub>3</sub> was employed as the oxidant (entry 8). No annulated product 3a or 3a' was detected in the absence of PivOH, which indicated the essential role of PivOH in this reaction (entry 9). [Cp\*Co(CO)I<sub>2</sub>] gave a comparable yield (entry 10). Other cobalt salts exhibited inferior efficiency in the catalysis of annulation of sulfonamide 1a with phenyl acetylene 2a (entries 11 and 12). 1,4-Dioxane was found to be the best solvent for this transformation (Table S1, entry 11, see the Supporting Information). Interestingly, only 0.75 equiv of NaClO<sub>3</sub> was required under our conditions, which implied that the reductive intermediates of  $ClO_3^-$ , such as  $ClO_2^-$  and  $ClO_-$ , could also act as the oxidant for the regeneration of Co(III) species (entry 15).<sup>12</sup> No decrease in the yield was detected when cobalt(II) acetate tetrahydrate was used instead of anhydrous salt under air (entry 16).

$ \begin{array}{c} 0,0\\ S'N^{Q} + \\ Ph \end{array} + \begin{array}{c} 0,0\\ PivOH (1.0 equiv)\\ 1,4-dioxane\\ 120 \ ^{\circ}C, 24 \ h \end{array} + \begin{array}{c} 0,0\\ S'N^{Q} + \\ Ph \end{array} + \begin{array}{c} $				
1a	2a	<b>3a</b> (Q=8	8-quinolinyl) <b>3a'</b>	
Entry	Catalyst	Oxidant	3a/3a' Yield <sup>b</sup> (%)	
1	Co(OAc) <sub>2</sub>	Ag <sub>2</sub> CO <sub>3</sub>	67/29	
2	Co(OAc) <sub>2</sub>	$Ag_2O$	79/16	
3	Co(OAc) <sub>2</sub>	AgOAc	57/28.	
4	Co(OAc) <sub>2</sub>	Mn(OAc) <sub>2</sub> /O <sub>2</sub>	trace	
5	$Co(OAc)_2$	Cu(OAc) <sub>2</sub>	n.d.	
6	$Co(OAc)_2$	PhI(OAc) <sub>2</sub>	n.d.	
7	Co(OAc) <sub>2</sub>	$Na_2S_2O_8$	n.r.	
$8^d$	$Co(OAc)_2$	NaClO <sub>3</sub>	87/trace	
$9^c$	Co(OAc) <sub>2</sub>	$Ag_2CO_3$	n.r.	
$10^d$	$Co(acac)_2$	NaClO <sub>3</sub>	57/trace	
$11^{d}$	CoCl <sub>2</sub>	NaClO <sub>3</sub>	30/trace	
$12^d$	[Cp*Co(CO)I <sub>2</sub> ]	NaClO <sub>3</sub>	88/trace	
13 <sup><i>d</i></sup>	Co(OAc)2·4H2O	NaClO <sub>3</sub>	88/trace	
$14^{d,e}$	Co(OAc)2·4H2O	NaClO <sub>3</sub>	89/trace	
$15^{e,f}$	Co(OAc)2·4H2O	NaClO <sub>3</sub>	92/trace	
16 <sup><i>e,f,g</i></sup>	Co(OAc)2·4H2O	NaClO <sub>3</sub>	92/trace	

<sup>*a*</sup> **1a** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv), oxidant (1.5 equiv), catalyst (20 mol%) and PivOH (1.0 equiv) were stirred in 1,4-dioxane (1.0 mL) at 120 °C for 24 hours under an atmosphere of  $N_2$ .

- <sup>b</sup> Isolated yields.
- <sup>c</sup> Without PivOH.
- <sup>*d*</sup> NaClO<sub>3</sub> (1.2 equiv) was used.
- <sup>*e*</sup> Co(OAc)<sub>2</sub>·4H<sub>2</sub>O (10 mol%) was used.
- <sup>*f*</sup> NaClO<sub>3</sub> (0.75 equiv) was used.
- <sup>g</sup> Reaction was performed under air. n.d. = not detected. n.r. = no reaction.

With the optimized conditions in hand, we next examined the scope of this catalytic system with respect to sulfonamide derivatives as summarized in Table 2. Both the sulfonamides with electron-donating and -withdrawing groups worked smoothly under the standard conditions, giving the corresponding products with an excellent regioselectivity in moderate to excellent yields (Table 2, 3b-h). The positions of substituents on the aromatic rings did not show significant influence on the yields (3f and 3g). Sulfonamide with a trifluoromethyl group at the ortho position could also afford the desired product 3h in 79% yield, indicating that the reaction seems insensitive to the steric hindrance. It is notable that heteroaromatic and condensed aromatic substrates also displayed good reactivity and excellent regioselectivity under the current reaction conditions (3i and 3i). In addition, the absolute configuration of 3a was confirmed by single crystal X-ray analysis (Table 2).<sup>1</sup>

Table 2 Scope of aryl sulfonamides<sup>*a,b*</sup>



<sup>*a*</sup> **1** (0.2 mmol, 1.0 equiv), **2a** (2.0 equiv),  $Co(OAc)_2 \cdot 4H_2O$  (10 mol%), NaClO<sub>3</sub> (0.75 equiv) and PivOH (1.0 equiv) were stirred in 1,4-dioxane (1.0 mL) at 120 °C for 24 hours under air. <sup>*b*</sup> Isolated yields.

We next investigated the generality of alkynes 2 with various substitutions as depicted in Table 3. Both aromaric and aliphatic terminal alkynes reacted smoothly with sulfonamide 1a to give the corresponding sultam products with excellent regioselectivity in moderate to excellent yields. It is noteworthy that an array of functional groups, including alkoxy, halide, ester, alkenyl and trimethylsilyl, were tolerant with the reaction conditions, which could offer an opportunity for further transformations (Table 3, 4b-k). The reaction could also be extended to internal alkynes. Symmetrical alkynes such as diaryl- and dialkyl-substituted acetylenes could be efficiently converted to the desired sultams (4l-t). However, unsymmetrical aryl alkyl alkynes gave a mixture of regioisomers, but with an obvious preference having the phenyl substituent proximal to nitrogen (4u and 4v), which could be separated by column chromatography and crystallization.

To further examine the generality of our methodology, benzamide **5** was subjected to the standard conditions, giving a single regioisomer **6** in 82% yield (eqn (1)). In addition, NaClO<sub>3</sub> was also successfully employed as an oxidant in the cobaltcatalyzed oxidative annulation of benzamide **5** with styrene (eqn (2)), rhodium-catalyzed oxidative annulation of azobenzene with diphenylacetylene (eqn (3)) and cobalt-catalyzed homo-coupling of benzamide **5** (eqn (4)), which further proved the efficiency and versatility of NaClO<sub>3</sub> as an oxidant in oxidative C–H activation.

 Table 3. Scope of Alkynes<sup>a,b</sup>



<sup>*a*</sup> **1a** (0.2 mmol, 1.0 equiv), **2** (2.0 equiv),  $Co(OAc)_2 \cdot 4H_2O$  (10 mol%),  $NaClO_3$  (0.75 equiv) and PivOH (1.0 equiv) were stirred in 1,4-dioxane (1.0 mL) at 120 °C for 24 hours under air.

<sup>b</sup> Isolated yields.

<sup>c</sup> NaClO<sub>3</sub> (1.0 equiv), Co(OAc)<sub>2</sub>·4H<sub>2</sub>O (20 mol%) and PivOH (2.0 equiv).



To further gain insight of the reaction mechanism, several common radical inhibitors were added, including 2,2,6,6-tetramethylpiperidine-*N*-oxyl (TEMPO), 2,6-di-*tert*-butyl-*p*-cresol (BHT) and ascorbic acid (Table S3 in SI). All of these radical scavengers exhibited significant suppression effect on the reaction, thus indicating that a radical process might be involved.

Based on the above results as well as previous reports, a proposed reaction mechanism was illustrated in Scheme 2.<sup>9b,11b,c,14</sup> Initial coordination of cobalt complex to **1a** leads to a Co(III) species **7**, which then undergoes C–H metallation and alkyne insertion to deliver a seven-membered metallacycle intermediate **9**. Subsequent reductive elimination affords the desired sultam product **3a** and releases the Co(I) species. Finally, the reoxidation of Co(I) to the reactive Co(III) species by  $\text{CIO}_n^-$  completes the catalytic cycle.



#### Scheme 2 Proposed mechanism.

In conclusion, a cobalt-catalyzed annulation of aryl sulfonamides with both terminal and internal alkynes to synthesize sultams has been developed. A broad set of sulfonamides and alkynes could be converted to the desired products with high regioselectivity in moderate to excellent yields. For the first time, NaClO<sub>3</sub> has been proved to be an efficient and versatile oxidant to sustain the transition-metal-catalyzed oxidative C–H activation reaction.

### Tetrahedron

### Acknowledgments

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- 12. In the presence of  $NaClO_2$  or  $Ca(ClO)_2$ , the desired product could be obtained in 45% and 5% yield, respectively (Table S2 in SI).
- Crystallographic data for the compound 3a (CCDC 1444380 for 3a) in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data\_request/cif.
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#### **Supplementary Material**

Supplementary data associated with this article can be found, in the online version, at XXX.

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## Highlights

Title: Sodium Chlorate as a Viable Substoichiometric Oxidant for Cobalt-Catalyzed **Oxidative Annulation of Aryl Sulfonamides with Alkynes** 

- (1) Synthesis of sultams through cobalt-catalyzed oxidative annulation.
- (2) NaClO<sub>3</sub> proved to be a versatile oxidant in C–H activation reaction.
- (3) Moderate to excellent yields with excellent regioselectivity could be obtained.
- (4) A broad scope of functional groups could be tolerant.