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Regioselective Annulation of Aryl Sulfonamides with Allenes through Cobalt-Promoted C−H Functionalization

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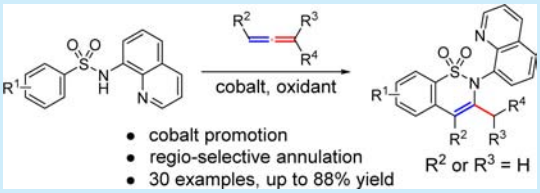
Supporting Information

ABSTRACT: The development of an efficient method for the construction of biologically relevant sultams is described, which represents the first case of cobalt-promoted C−H/N−H functionalization of sulfonamides with allenes. This newly developed annulation reaction demonstrated good functional group tolerance and excellent regioselectivity. Both terminal monosubstituted allenes and internal disubstituted allenes can be employed to give the desired sultams in good yields. This strategy can be successfully used to build a unique sultam library with novel structural diversity.



Cyclic sulfonamides (sultams) are an important class of chemicals that serve as structural motifs in different drugs and bioactive compounds (Figure 1).[1] For example, the attractive

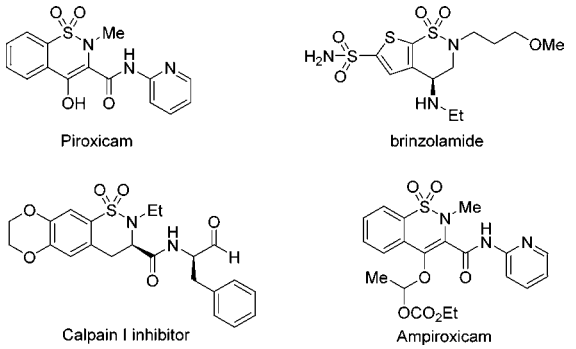


Figure 1. Drugs containing sultam motifs.

biological activities of sultams have been found in a wide range of fields including anti-inflammatory,[2] antibacterial,[3] anti-HIV,[4] and antimalarial,[5] etc.[6] Because of its significance in pharmaceutical development, a number of strategies have been developed for the synthesis of sultams.[7] However, most of the reported methods rely on intramolecular cyclization of elaborate precursors or intermolecular reactions starting from prefunctionalized starting materials. These methods usually suffer from multistep synthesis of precursors, tedious procedures, and low yields. Thus, further development of new, efficient, and general methods toward the synthesis of cyclic sulfonamides using readily available precursors is quite appealing.

In recent years, transition-metal-catalyzed annulation reactions through C−H bond functionalization have gradually emerged as a powerful tool for the synthesis of various heterocyclic compounds.[8] The straightforward synthesis of sultam scaffolds from simple starting materials through C−H functionalization reactions would be very attractive. To date, several annulation reactions generating sultams through transition-metal-catalyzed *ortho* C−H activation of aryl sulfonamides with alkynes and alkenes have been developed.[9] For example, in 2012, the Cramer[9a] group reported the synthesis of sultam scaffolds through Rh-catalyzed C−H functionalization of acylated aryl sulfonamides with alkynes. In 2014, Li and co-workers[9b] described the synthesis of five-membered sultams via Rh-catalyzed annulation reaction between acylated aryl sulfonamides and alkenes. More recently, the groups of Ribas[9c] and Sundararaju[9d] independently introduced the annulation reactions of aryl sulfonamides with alkynes through cobalt[10,11] catalysis. It should be noted that these methods generally employ alkynes and alkenes as coupling partners. In contrast, the practice of using allenes as annulation partners in C−H functionalization is still rare.[12] Different from alkenes and alkynes, the complexity of two orthogonal carbon−carbon double bonds of allenes can provide chances to access structurally unique products.

Because of the special reactivity of the 1,2-diene functionality, allenes have demonstrated great potential in organic chemistry.[13] In particular, the carbometalation reaction of allenes recently is one of the most interesting research field and has exhibited high potential for the preparation of novel and diverse scaffolds.[14] However, to the best of our knowledge, there are no reported examples of cobalt-promoted C(sp^2)−H functionalization of sulfonamides with allenes. We envisioned that the cobalt-promoted ortho C(sp^2)−H activation of sulfonamides might undergo an annulation reaction with allenes to produce corresponding sultams. Herein, we report the first example of regioselective sultam synthesis through cobalt-promoted C−H/N−H annulation of aryl sulfonamides with allenes (Scheme 1).

Initially, we began our investigations by screening different cobalt reagents in the reaction system, which consists of sulfonamide **1a**, allene partner **2a**, $Mn(OAc)_2$, KOAc, and

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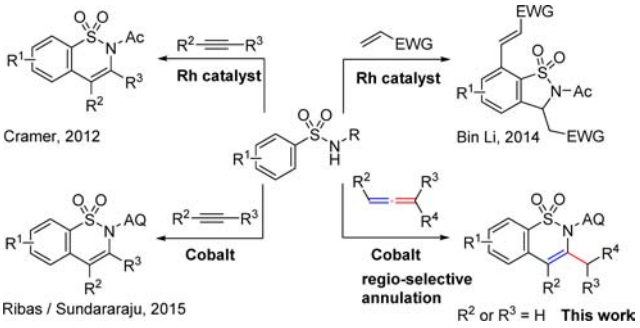
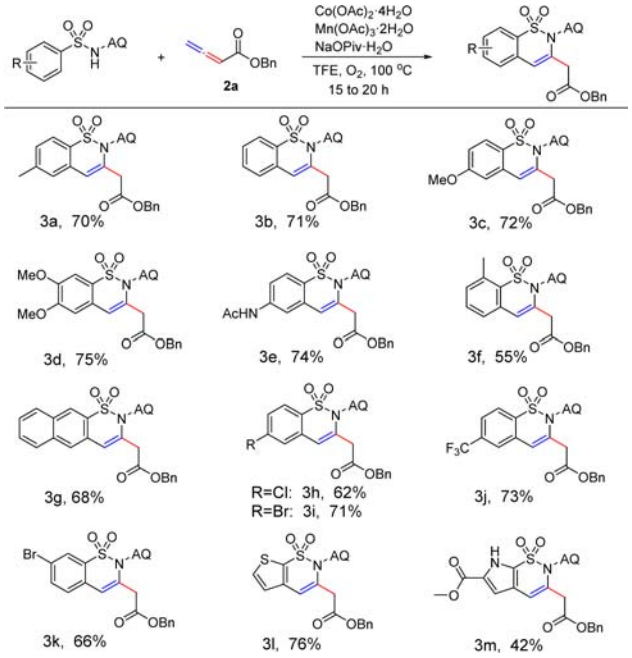
Scheme 1. *Ortho* C−H Functionalization of Aryl Sulfonamides



Scheme 2. Reaction Scope of Aryl Sulfonamides[a]

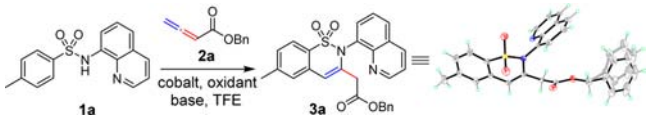


[a]Reaction conditions: aryl sulfonamide (0.1 mmol), **2a** (3.0 equiv), $Co(OAc)_2·4H_2O$ (20−50 mol %; in some cases $Co(NO_3)_2·6H_2O$ was used instead of $Co(OAc)_2·4H_2O$), $Mn(OAc)_3·2H_2O$ (2.0 equiv), $NaOPiv·H_2O$ (2.0 equiv), 1.5 mL of TFE, 100 °C, O_2.

trifluoroethanol (TFE) under air at 100 °C for 16 h. We found that $Co(OAc)_2·4H_2O$ was the optimal promoter among the four cobalt sources tested (Table 1, entries 1−5). When $Co(OAc)_2·4H_2O$ was used, the desired sultam product **3a** was observed in 13% NMR yield (entry 5; for the X-ray crystal structure, see the Supporting Information). Changing the oxidant from $Mn(OAc)_2$ to $Mn(OAc)_3·2H_2O$ resulted in an improved 35% NMR yield (entry 6). To our delight, we found that O_2 could promote the reaction significantly (entries 5, 7, and 12). Furthermore, a higher isolated yield (70%) can be obtained by replacing KOAc with $NaOPiv·H_2O$ (entry 10). For all of the conditions examined in Table 1, no obvious regioisomers can be observed. The control experiment showed that the omission of cobalt source resulted in the complete inactivity of the reaction (entry 14).

With the optimal conditions in hand, we next attempted to explore the substrate scope for this new reaction. A variety of substituted sulfonamides bearing 8-aminoquinoline directing groups were tested under the optimal conditions (Scheme 2). Electron-rich substrates (**3a**−**e**) could proceed smoothly to furnish the desired products in good isolated yields, whereas electron-deficient substrates (**3h**−**k**) only gave low to moderate isolated yields under the standard conditions with almost half of the starting material remaining. That may attribute to the increased difficulty of the C−H activation step caused by electron-withdrawing groups. To our satisfaction, these electron-deficient substrates could provide products in good yields when the $Co(OAc)_2·4H_2O$ loading was increased from 20 to 50 mol %. Functional groups such as −NHAc, −Cl, and −Br were well tolerated. Notably, some heterocyclic substrates were found to be compatible with the reaction system as well. For example, the 2-thiophenesulfonamide substrate can be readily transformed to the

Table 1. Optimization of Cobalt-Promoted C−H Activation and Annulation with Allene[a]



entry	cobalt source	oxidant	base	yield of **3a**[b] (%)
1	$Co(acac)_2$	$Mn(OAc)_2$	KOAc	<5
2	$Co(acac)_3$	Mn(OAc)2	KOAc	<5
3	CoF_3	$Mn(OAc)_2$	KOAc	<5
4	$CO(NO_3)_2·6H_2O$	$Mn(OAc)_2$	KOAc	<5
5	$Co(OAc)_2·4H_2O$	$Mn(OAc)_2$	KOAc	13
6	$Co(OAc)_2·4H_2O$	$Mn(OAc)_3·2H_2O$	KOAc	35
7	$Co(OAc)_2·4H_2O$	$Mn(OAc)_2/O_2$	KOAc	40
8	$Co(OAc)_2·4H_2O$	$Mn(OAc)_3·2H_2O/O_2$	KOAc	59[e]
9	$Co(OAc)_2·4H_2O$	$Mn(OAc)_2/O_2$	$NaOPiv·H_2O$	68[e]
10	$Co(OAc)_2·4H_2O$	$Mn(OAc)_3·2H_2O/O_2$	$NaOPiv·H_2O$	70[e]
11[c]	$Co(OAc)_2·4H_2O$	$Mn(OAc)_3·2H_2O/O_2$	$NaOPiv·H_2O$	64[e]
12[d]	$Co(OAc)_2·4H_2O$	$Mn(OAc)_3·2H_2O$	$NaOPiv·H_2O$	51
13	$Co(NO_3)_2·6H_2O$	$Mn(OAc)_3·2H_2O/O_2$	$NaOPiv·H_2O$	69[e]
14		$Mn(OAc)_3·2H_2O/O_2$	$NaOPiv·H_2O$	0

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (3.0 equiv), cobalt (20 mol %), oxidant (2.0 equiv), base (2.0 equiv), 1.5 mL of TFE (trifluoroethanol), 100 °C, 16 h, air. [b]Yields are calculated by ^1H NMR analysis of the reaction mixture using *p*-nitrobenzaldehyde as internal standard. [c]85 °C, 16 h. [d]N_2. [e]Isolated yields.

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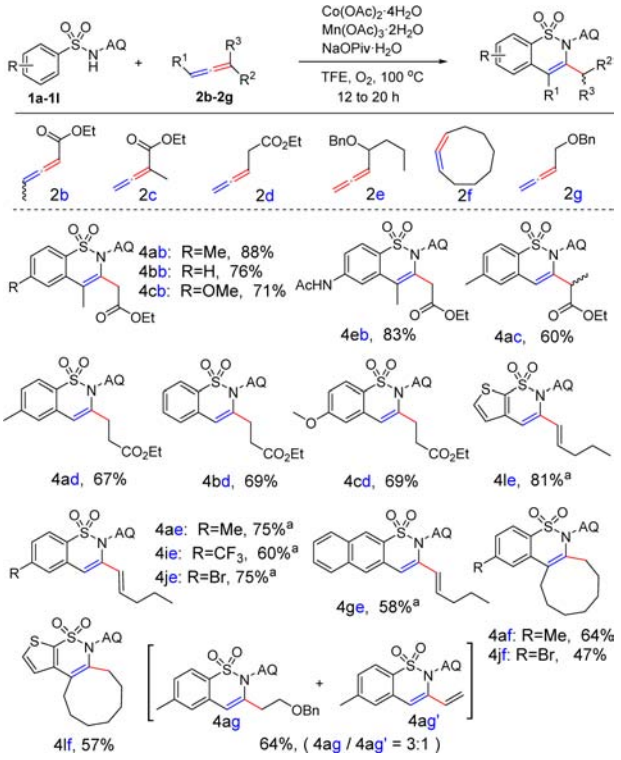
desired sultam (**3l**), which could be regarded as an analogue of brinzolamide. It is worth noting that in our case the 2-pyrrolesulfonamide substrate is well tolerated, even though the substrate contains the free N−H group.

Utilizing this annulation reaction, we then turned our attention to explore the scope and reactivity pattern of the allene partners. As shown in Scheme 3, both electron-poor and electron-rich

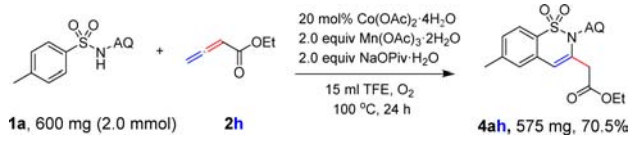
Scheme 3. Reaction Scope of Allenes*



*Reaction conditions: aryl sulfonamide (0.1 mmol), allene (**2b−g**) (3.0 equiv), $Co(OAc)_2{\cdot}4H_2O$ (20−50 mol %, in some cases $Co(NO_3)_2{\cdot}6H_2O$ was used instead of $Co(OAc)_2{\cdot}4H_2O$), $Mn(OAc)_3{\cdot}2H_2O$ (2.0 equiv), $NaOPivH_2O$ (2.0 equiv), 1.5 mL of TFE, 100 °C, O_2. [a]Product derived from **2e**.

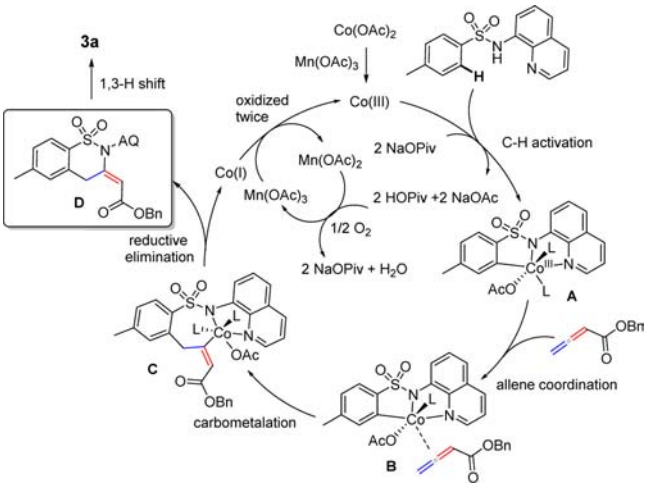
allenes (**2b−g**) were examined in the reaction system. It was found that the electronic properties of the allenes do not change the fashion of the annulation reaction. The new C−C bond formed preferentially between the *ortho* carbon of the aryl sulfonamides and the less hindered terminal carbon of the allenes. Under these conditions, all tested allenes could be regioselectively incorporated to generate the desired sultams in good isolated yields. For the internal disubstituted allene **2b**, the small steric hindrance difference between the methyl group and the ester group can be accurately recognized. It was interesting that when **2e** was used as the allene partner, 1,3-diene derivatives **4ae−je** formed as the final products. When **2g** was used as the allene partner, partial elimination led to a mixture of product (**4ag**) and the 1,3-diene derivative (**4ag′**) in a ratio of 3:1. This sequential reaction enriches the diversity of products derived from the annulation reaction. Satisfyingly, the nine-membered cyclic allene **2f** is also compatible with the reaction system to furnish the three-ring-fused heterocycle products **4af−lf**, which are difficult to obtain through known methods of sultam synthesis. This new reaction demonstrates excellent regioselectivity. Compared with rhodium- or cobalt-catalyzed annulation of aryl sulfonamides with alkynes, which may provide two regioisomers with terminal aliphatic alkynes,[9a,d] only one regioisomer can be observed from this reaction in all cases. It should be mentioned that all of the allenes used can be easily accessed on large scale through inexpensive and simple starting materials in one or two steps, except **2e**, which takes three steps for preparation. To further evaluate the practical utility of this new reaction, a larger scale reaction was performed by using **1a** (2 mmol, 600 mg) and allene partner **2h**, which gave **4ah** in 70.5% yield (see Scheme 4).

Scheme 4. Sub-Gram-Scale Synthesis of 4ah



As illustrated in Scheme 5, a plausible reaction mechanism was proposed. The first step involves the oxidation of Co(II) to give

Scheme 5. Proposed Reaction Pathway



Co(III) by $Mn(OAc)_3$. Next, chelation of Co(III) to the quinoline directing group and subsequent C−H activation gives intermediate **A**. The following step is the coordination of the allene to the Co(III) center of intermediate **A** to yield the intermediate **B**. Based on the relevant reports of allene-involved C−H functionalization[12g,15] and cobalt-catalyzed alkyne annulation,[9c,d,16] in which vinylmetal intermediates were proposed, the next step might be the insertion of the less hindered double bond of the allene into the carbon−cobalt bond to form intermediate **C** in this case. Following this step, the reductive elimination of intermediate **C** gives Co(I) and intermediate **D**. Intermediate **D** could be isolated and identified (for details, see Supporting Information). Finally, intermediate **D** undergoes 1,3-H shift to provide the desired product **3a**.[17] The Co(I) can be further oxidized to Co(III) for the next turnover. O_2 is likely to play a role as the terminal oxidant in the entire process.

In conclusion, we have established a new, effective method for the construction of biologically relevant sultam scaffolds that represents the first example of cobalt-promoted C−H/N−H functionalization of aryl sulfonamides with allenes. A broad range of aryl sulfonamides can be exploited to react with both terminal monosubstituted allenes and internal disubstituted allenes to give the desired sultams in good yields with excellent regioselectivity.

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Upon successful implementation, this strategy can be further employed to build a promising sultam library with novel structural diversity for biological activity screening.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b03510.

General experimental procedure, characterization data for the products, and X-ray crystal data for **3a** (PDF)

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Notes

The authors declare no competing financial interest.

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(17) The transformation could be observed even in $CDCl_3$ at room temperature (for details, see the Supporting Information).

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