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# Synthesis of 2,5-diaryloxazoles through rhodium-catalyzed annulation of triazoles and aldehydes†

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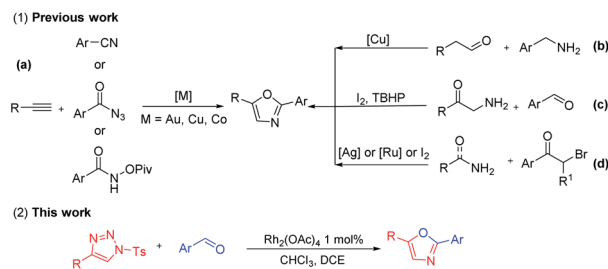
An efficient synthesis of a variety of 2,5-diaryloxazole derivatives *via* a rhodium-catalyzed annulation of triazoles and aldehydes is achieved. Various oxazole derivatives could be obtained in good to excellent yields. A concise synthesis of antimycobacterial natural products balsoxin and texamine has been achieved using this method.

Oxazoles, an important class of heterocycles, are present in a wide range of natural products and biologically active molecules,<sup>1</sup> as exemplified by salinazinone A,<sup>2</sup> peptide alkaloid (–)-muscoride A,<sup>3</sup> and antidiabetic agent AD-5061.<sup>4</sup> In particular, 2,5-diaryl substituted oxazoles are found in pharmacologically active molecules such as antipancreatic cancer agent PC-046,<sup>5</sup> antimycobacterial natural product texamine<sup>6</sup> and balsoxin.<sup>7</sup> Given this significant importance, several methodologies have been developed to access functionalized oxazole skeletons.<sup>8</sup> Some groups reported a metal-catalyzed synthetic method for 2,5-diaryl substituted oxazoles through the reaction of alkynes and aryl nitrile or aryl carbonyl azides or *N*-pivaloyloxyamides (Scheme 1(a)).<sup>9</sup> In 2012, a copper-catalyzed oxidative dehydrogenative annulation method of alkynes and amines was developed by Jiao's group (Scheme 1(b)).<sup>10</sup> Wang's group demonstrated a TBHP/I<sub>2</sub>-mediated tandem oxidative cyclization with 2-amino-1-arylethan-1-one and arylaldehyde to prepare 2,5-diaryl substituted oxazoles (Scheme 1(c)).<sup>11</sup> In addition,  $\alpha$ -bromoketones and arylamine or arylamides derivatives were utilized by Moses,<sup>12</sup> Zhang<sup>13</sup> and Cho<sup>14</sup> group for the synthesis of substituted oxazoles (Scheme 1(d)). Despite significant progress in this field, several limitations including low efficiency of activation, tedious side reactions and the lack of easy access to structural diversity, have hampered their further application. Thus, the development of efficient and economy strategy for the synthesis of 2,5-diaryl substituted oxazoles has been both a challenge and a focus of synthetic chemistry. Herein, we report an approach that *N*-sulfonyl 1,2,3-triazoles react with aldehydes to give 2,5-diaryloxazoles *via* a cyclization.

*N*-Sulfonyl-1,2,3-triazoles have recently emerged as structural motifs that are studied for synthesizing a variety of biologically active heterocycles,<sup>15</sup> including pyrrole,<sup>16</sup> imidazole,<sup>17</sup> oxazoline,<sup>18</sup> pyrroloindoline<sup>19</sup> and others.<sup>20</sup> In these transformations, Rh(II)-azavinylcarbene (Rh-AVC) was considered as the key intermediate, which derived from *N*-sulfonyl-1,2,3-triazoles under the treatment with rhodium(II) catalysts through denitrogenative reaction.<sup>21</sup> Due to its dipolar nature, Rh-AVC could serve as an aza-[3C]-synthons in various [3 + 2] cycloaddition reactions. So far, a wide range of unsaturated chemical bonds, including aldehyde, nitrile, have been well explored in the [3 + 2] cycloadditions.<sup>22</sup> 2008, Fokin and co-workers exploited the reactivity of Rh-AVC to achieve imidazoles in good to excellent yields with *N*-sulfonyl 1,2,3-triazoles and nitriles.<sup>17</sup> 2013, they reported that Rh-AVC react with aldehydes to give 3-sulfonyl-4-oxazolines through an intramolecular cyclization (Scheme 2(a)).<sup>18</sup> We were curious what would happen if *o*-cyanobenzaldehyde, containing aldehyde group and cyano group together, react with 1,2,3-triazoles? A variety of nitrogen-containing heterocycles,<sup>23</sup> such as 3-amino-substituted isoindolinones (Scheme 2(b1)),<sup>23a</sup> 3-(2-oxopropyl)-2-arylisoindolinone (Scheme 2(b2)),<sup>23b</sup> 3-aryl isoindolinones (Scheme 2(b3))<sup>23c</sup> and benzo[4,5]imidazo[2,1-*a*]isoquinolines (Scheme 2(b4)),<sup>23d</sup> could be constructed with *o*-cyanobenzaldehyde. As a continuation of our

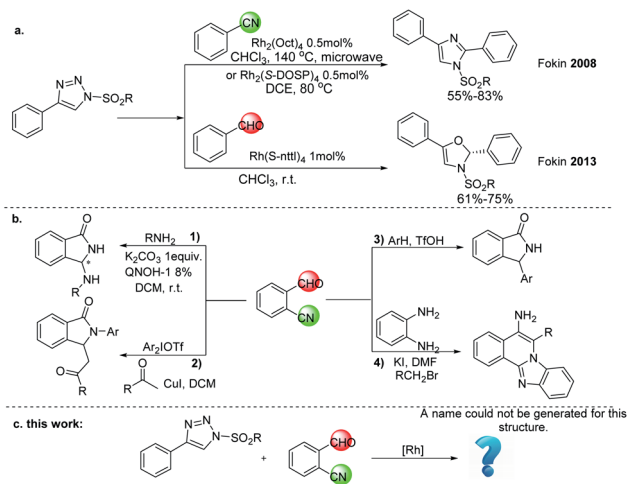
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Scheme 1 Selected synthetic strategies for 2,5-disubstituted oxazoles.





**Scheme 2** Reactions between *N*-sulfonyl 1,2,3-triazoles and nitriles/aldehydes.

research on cyano activation for the synthesis of nitrogen-containing heterocycles,<sup>24</sup> we envisioned that the [3 + 2] cycloadditions between *N*-sulfonyl 1,2,3-triazoles and *o*-cyanobenzaldehyde would afford a novel approach to the synthesis of nitrogen-containing heterocyclic compounds (Scheme 2(c)).

To test the hypothesis, we started the investigation by using 4-phenyl-1-tosyl-1*H*-1,2,3-triazole **1a** and *o*-cyanobenzaldehyde **2a** as model substrates in the presence of 1 mol% of Rh<sub>2</sub>(OAc)<sub>4</sub> in CHCl<sub>3</sub> at room temperature. Interestingly, we did not find the corresponding oxazoline product reported by Fokin's group. Instead, the oxazole **3a**, which is the product of the sulfinic acid elimination was isolated in 14% yield after 12 hours, and the starting materials **2a** was recovered in 68% yield. Due to the importance of oxazoles, we decided to optimize the reaction conditions with oxazoles as the target product. Increasing the temperature is beneficial to improve the yields of the products, the reactions afforded the desired product **3a** in 24% yield at 80 °C and 79% yield at 120 °C (Table 1, entries 2, 3). From the experiment we could conclude that the product was easier to obtain at higher temperature, it seems that high temperatures help to the elimination of sulfinic acid. Other rhodium catalysts were tested, but no better results were obtained for this transformation (Table 1, entries 5–7). For Rh<sub>2</sub>(OAc)<sub>4</sub> (1 mol%), the yields of the desired product **3a** in various solvents were as follows: DCE (52%), toluene (11%) and CH<sub>3</sub>CN (7%), none or trace of product was found in other solvents (entries 8–14).

With an optimized set of reaction conditions in hand, we assessed the scope of these new reactions with various 4-phenyl-1-tosyl-1*H*-1,2,3-triazole **1** and *o*-cyanobenzaldehyde **2**; the results are summarized in Table 2. As shown in Table 2, our cycloaddition reaction turned out to be widely applicable regardless of the electronic and steric properties of the aryl ring of the substrates **1**. For 1,2,3-triazole substrates bearing 4-substituted 4-phenyl-1-tosyl-1*H*-1,2,3-triazoles (**1a–1h**; R<sup>1</sup> = 4-XC<sub>6</sub>H<sub>4</sub>; X = F, Cl, Br, OMe, *n*-Bu, CO<sub>2</sub>Me, and Ph), rhodium-catalyzed reactions gave desired **3a–3h** in 79–88% yields. The reaction was found to be marginally affected by the electronic

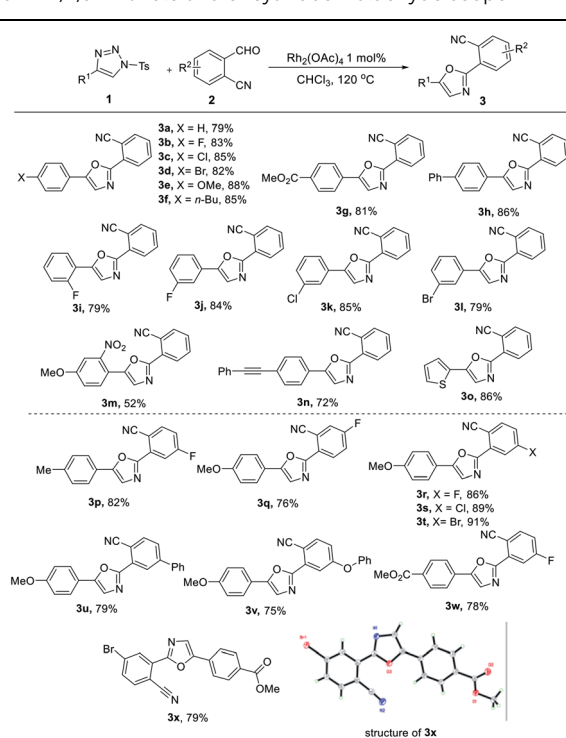
**Table 1** Optimization of the reaction conditions<sup>a</sup>

Entry	Catalyst	Solvent	Temperature (°C)	Time (h)	Yield <sup>b</sup> (%)
1	Rh <sub>2</sub> (OAc) <sub>4</sub>	CHCl <sub>3</sub>	rt	12	14
2	Rh <sub>2</sub> (OAc) <sub>4</sub>	CHCl <sub>3</sub>	80	12	24
3	Rh <sub>2</sub> (OAc) <sub>4</sub>	CHCl <sub>3</sub>	120	12	79
4	Rh <sub>2</sub> (OAc) <sub>4</sub>	CHCl <sub>3</sub>	150	12	47
5	Rh <sub>2</sub> (esp) <sub>2</sub>	CHCl <sub>3</sub>	120	12	21
6	Rh <sub>2</sub> (otc) <sub>4</sub>	CHCl <sub>3</sub>	120	12	28
7	Rh(Ph <sub>3</sub> P) <sub>3</sub> Cl	CHCl <sub>3</sub>	120	12	—
8	Rh <sub>2</sub> (OAc) <sub>4</sub>	DCE	120	12	52
9	Rh <sub>2</sub> (OAc) <sub>4</sub>	Toluene	120	12	11
10	Rh <sub>2</sub> (OAc) <sub>4</sub>	EtOH	120	12	—
11	Rh <sub>2</sub> (OAc) <sub>4</sub>	CH <sub>3</sub> CN	120	12	7
12	Rh <sub>2</sub> (OAc) <sub>4</sub>	DMF	120	12	—
13	Rh <sub>2</sub> (OAc) <sub>4</sub>	THF	120	12	—
14	Rh <sub>2</sub> (OAc) <sub>4</sub>	1,4-Dioxane	120	12	Trace

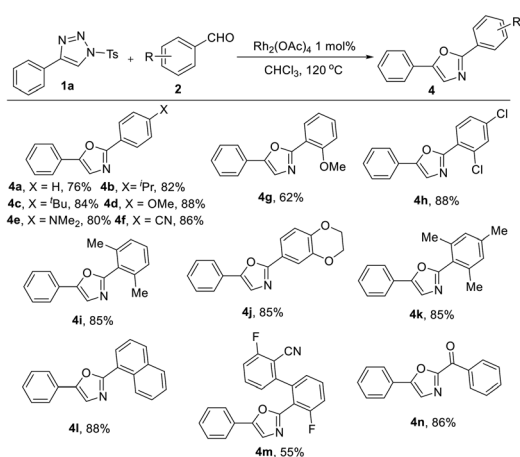
<sup>a</sup> Reaction condition: **1a** (0.6 mmol), **2a** (0.3 mmol) in solvent (2.0 mL), 1 mol% of catalyst, 12 h. <sup>b</sup> Isolated yield.

nature of the substituents on the aryl ring as shown by the reactions of *o*-cyanobenzaldehyde **2a** with triazoles bearing electron-donating substituents, which gave higher yields (**3e**

**Table 2** 1,2,3-Triazole and *o*-cyanobenzaldehyde scope<sup>a,b</sup>



<sup>a</sup> Reaction condition: **1** (0.6 mmol), **2** (0.3 mmol) in CHCl<sub>3</sub> (2.0 mL), 120 °C, 12 h. <sup>b</sup> Isolated yield.

Table 3 Aldehyde scope<sup>a,b</sup>

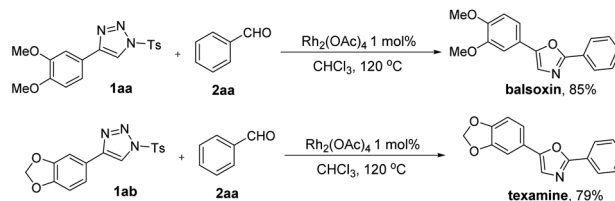
<sup>a</sup> Reaction condition: **1a** (0.6 mmol), **2** (0.3 mmol) in CHCl<sub>3</sub> (2.0 mL), 120 °C, 12 h. <sup>b</sup> Isolated yield.

and **3f**). We tested the reaction with 2-substituted and 3-substituted 4-phenyl-1-tosyl-1*H*-1,2,3-triazoles, and the corresponding products **3i–3l** were obtained in 79–85% yields. For disubstituted analogue **1m**, the corresponding product **3m** was produced in 52% yield. We also performed the reactions with reactants with 4-(phenylethynyl)-1-tosyl-1*H*-1,2,3-triazole **1n**, which afforded alkynyl-containing compound **3n** in 72% yield, this indicated that alkynyl group could be tolerated in the reaction conditions. The reaction was further compatible with heterocyclic substituted triazole (**1o**), yielding the desired thio-phen product **3o** in 86% yield.

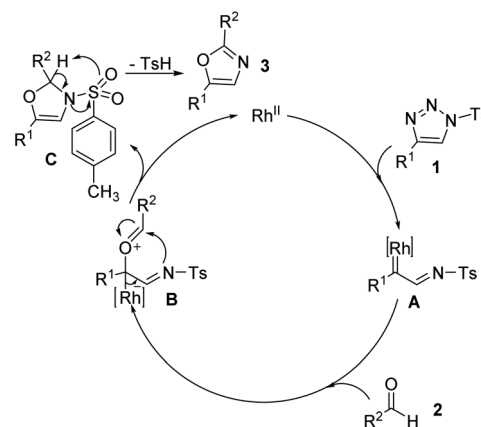
We next assessed the scope of *o*-cyanobenzaldehyde **2**. To our delight, with a series of substituents at different position of the *o*-cyanobenzaldehyde, including 4-F, 5-F, 5-Cl, 5-Br, 5-Ph and 5-OPh, the corresponding products **3p–3x** were formed in 75–91% yields (Table 2). The molecular structure of compound **3x** was confirmed by X-ray diffraction.

As cyano group was often employed as a ligand in transition metal-catalyzed reactions, we wondered whether the cyano group in *o*-cyanobenzaldehyde participated in the coordination with the rhodium metal in this reaction to promote the reaction. We chose unsubstituted benzaldehyde **2aa** as a substrate to react with 4-phenyl-1-tosyl-1*H*-1,2,3-triazole **1a** under the same conditions. It was surprising that the product 2,5-diphenyloxazole **4a** was collected in 76% yield. To demonstrate the generality of the present rhodium-catalyzed reactions of benzaldehydes, a series of aromatic aldehydes were investigated under the optimized reaction conditions, the results are summarized in Table 3.

For the scope with respect to the aromatic aldehyde substrate, the corresponding 2,5-disubstituted oxazole products were obtained in good yields in all cases. In terms of electronic effect, both electron-rich substrates (**2ab–2ad**, **2ag**, and **2ai–2ak**) and electron-deficient substrate (**2af**) could be tolerated (yield from 62% to 88%, Table 3), including isopropyl group (**4b**), *t*-



Scheme 3 One-step synthesis of natural products.



Scheme 4 Possible mechanistic pathways.

butyl group (**4c**), methoxy group (**4d**), methyl group (**4i**, **4k**), and cyano group (**3**, **4f**). Singly *ortho*-substitued (**2ah**, **2al**) or doubly *ortho*-substitued (**2ai**, **2ak**, **2am**) aldehydes lead to the corresponding products smoothly. Complex substrate 3,3'-difluoro-2'-formyl-[1,1'-biphenyl]-2-carbonitrile **2am** was prepared for this annulation, which proved to be applicable substrate and afforded compound **4m** in 55% yield. The reaction was further compatible with 2-oxo-2-phenylacetaldehyde **2an**, yielding the desired product **4n** in 86%. Several cases of aliphatic aldehydes, such as butyraldehyde, isopropanal, phenylacetaldehyde were also investigated, but none of the corresponding products were found under the standard conditions.

To demonstrate the synthetic utility of this protocol developed herein, we carried out the reaction with 4-(3,4-dimethoxyphenyl)-1-tosyl-1*H*-1,2,3-triazole **1aa** and benzaldehyde **2aa** in CHCl<sub>3</sub> for the preparation of natural product balsoxin (Scheme 3). Gratifyingly, the desired product balsoxin could be obtained in 85% yield in one step. With the same strategy, natural product texamine, could be produced in 79% yield in 12 hours.

With literature precedents in hand, a reaction pathway is proposed in Scheme 4. 1,2,3-Triazole **1**, upon reaction with Rh<sub>2</sub>(OAc)<sub>4</sub>, extrudes dinitrogen and forms Rh(II)-azavinyl carbene species **A**. Interaction of the carbonyl group with the carbene center leads to the formation of ylide **B**, which undergoes cyclization, leading to the formation of oxazoline **C**. Finally, removal of the *p*-toluenesulfonic acid followed afforded the desired oxazole **3**.

In conclusion, a rhodium-catalyzed annulation of 1,2,3-triazoles and aldehydes has been developed. The developed

methodology provides straightforward access to valuable 2,5-diaryl substituted oxazole derivatives. Applying this strategy, a concise synthesis of natural products balsoxin and texamine have been accomplished in good yields in one step. Further studies to explore the possibility for synthesis of various oxazoles are currently underway in our laboratory.

## Conflicts of interest

There are no conflicts to declare.

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