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# Copper-Catalyzed Three-Component Domino Cyclization for the Synthesis of 4-Aryl-5-(arythio)-2-(trifluoromethyl)oxazoles

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Supporting Information

ABSTRACT: A copper-catalyzed oxidative cyclization of oxime, arylthiol, and trifluoroacetic anhydride for the construction of trisubstituted oxazoles has been developed. This transformation combines N-O bond cleavage, C-H functionalization, and intramolecular annulation, providing a practical protocol for the introduction of a trifluoromethyl  $(-CF_3)$  group at oxazole rings.

Organic

he oxazole skeleton is an important N-heterocyclic structural unit, existing in naturally occurring compounds and drugs.<sup>1</sup> Therefore, a series of efficient and simple methods for synthesizing polysubstituted oxazoles have been developed.<sup>2</sup> Despite these applications, few methods for the introduction of a trifluoromethyl  $(-CF_3)$  group onto oxazole rings,<sup>3</sup> which may change the metabolic stability, lipophilicity, and biological properties of oxazole compounds,<sup>4</sup> have been reported. In this regard, the development of efficient methods for preparing trifluoromethyl-decorated oxazoles has long been a hot and challenging topic in molecular synthesis.

Oximes play an important role in the area of synthetic organic chemistry because of the prevalent molecular skeleton for various organic transformations.<sup>5</sup> In particular, O-acyl oximes have been among the most extensively studied in an attempt to provide viable precursors of active imino radicals due to their stability and ease of preparation.<sup>6</sup> Over the past decade, major improvements in the development of transition metal-catalyzed transformations of methylketoxime esters have been realized, especially in the synthesis of nitrogen heterocycles such as thiazoles,<sup>7</sup> pyridines,<sup>8</sup> pyrazoles,<sup>9</sup> pyrroles,<sup>10</sup> imidazo[1,2-*a*]pyridines,<sup>11</sup> etc. (Figure 1, a–e). Very recently, we have developed novel approaches for synthesis of benzo[4,5]thieno[3,2-d]thiazoles via C-H bis-heteroannulation of acetophenone oximes with elemental sulfur.<sup>12</sup> To the best of our knowledge, there are few methods for constructing the biologically important trifluoromethyl nitrogen-containing heterocycles using arylmethylketoximes as a versatile N-C-C building block.<sup>13</sup> Herein, we disclose the copper-catalyzed three-component oxidative annulation reactions of ketoximes, trifluoroacetic anhydride, and arylthiols, which provide efficient access to 4-aryl-5-(arythio)-2-(trifluoromethyl)oxazoles (Figure 1, f). Hence, salient features of our finding include (a) easy operational handling, (b) readily available starting materials,





Figure 1. Copper-catalyzed N-heteroarenes from methylketone oximes.

(c) access to highly functionalized trifluoromethyl oxazoles, and (d) broad functional group tolerance.

We commenced our studies by exploring the threecomponent tandem cyclization of acetophenone oxime 1a, 4methylbenzenethiol 2a, and trifluoroacetic anhydride 3a (Table 1). To our delight, the domino cyclization proceeded smoothly to afford product 4a in 65% yield using  $Cu(OAc)_2$  as the catalyst, N-iodosuccinimide (NIS) as the additive, and  $K_2S_2O_8$  as the oxidant in DCE (Table 1, entry 1). Encouraged

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Table 1. Optimization of the Reaction Conditions<sup>a</sup>

| Ph    | NOH +             | ו<br>} + (CF₃C | catalyst, o:<br>CO) <sub>2</sub> O <u>additiv</u><br>solven | kidant<br><u>e</u> Ph<br>t |                        |
|-------|-------------------|----------------|-------------------------------------------------------------|----------------------------|------------------------|
| 1a 2  |                   | 3a             |                                                             | 4a                         |                        |
| entry | catalyst          | additive       | oxidant                                                     | solvent                    | yield (%) <sup>b</sup> |
| 1     | $Cu(OAc)_2$       | NIS            | $K_2S_2O_8$                                                 | DCE                        | 65                     |
| 2     | $CuSO_4$          | NIS            | $K_2S_2O_8$                                                 | DCE                        | 58                     |
| 3     | $Cu(OTf)_2$       | NIS            | $K_2S_2O_8$                                                 | DCE                        | 76                     |
| 4     | Cu <sub>2</sub> O | NIS            | $K_{2}S_{2}O_{8}$                                           | DCE                        | 57                     |
| 5     | CuI               | NIS            | $K_2S_2O_8$                                                 | DCE                        | 49                     |
| 6     | CuCl <sub>2</sub> | NIS            | $K_2S_2O_8$                                                 | DCE                        | 56                     |
| 7     | $Cu(OTf)_2$       | $I_2$          | $K_2S_2O_8$                                                 | DCE                        | 52                     |
| 8     | $Cu(OTf)_2$       | ICl            | $K_2S_2O_8$                                                 | DCE                        | 67                     |
| 9     | $Cu(OTf)_2$       | $NaIO_4$       | $K_2S_2O_8$                                                 | DCE                        | 62                     |
| 10    | $Cu(OTf)_2$       | IBX            | $K_{2}S_{2}O_{8}$                                           | DCE                        | 60                     |
| 11    | $Cu(OTf)_2$       | NIS            | $Na_2S_2O_8$                                                | DCE                        | 40                     |
| 12    | $Cu(OTf)_2$       | NIS            | $(NH_4)_2S_2O_8$                                            | DCE                        | 42                     |
| 13    | $Cu(OTf)_2$       | NIS            | oxone                                                       | DCE                        | 50                     |
| 14    | $Cu(OTf)_2$       | NIS            | S                                                           | DCE                        | 37                     |
| 15    | $Cu(OTf)_2$       | NIS            | $K_{2}S_{2}O_{8}$                                           | PhCH <sub>3</sub>          | 70                     |
| 16    | $Cu(OTf)_2$       | NIS            | $K_2S_2O_8$                                                 | CH <sub>3</sub> CN         | 63                     |
| 17    | $Cu(OTf)_2$       | NIS            | $K_2S_2O_8$                                                 | TTCE                       | 64                     |
| 18    | $Cu(OTf)_2$       | NIS            | $K_2S_2O_8$                                                 | DMF                        | 0                      |
| 19    | $Cu(OTf)_2$       | NIS            | $K_2S_2O_8$                                                 | DMSO                       | 0                      |
| 20    | _                 | NIS            | $K_2S_2O_8$                                                 | DCE                        | 20                     |
| 21    | $Cu(OTf)_2$       | -              | $K_2S_2O_8$                                                 | DCE                        | 22                     |
| 22    | $Cu(OTf)_2$       | NIS            | -                                                           | DCE                        | 5                      |

<sup>a</sup>Conditions: 1a (0.2 mmol), 2a (0.3 mmol), 3a (0.8 mmol), catalyst (5 mol %), additive (0.12 mmol), oxidant (0.3 mmol), solvent (1 mL), 80 °C, 20 h, air. <sup>b</sup>Isolated yield.

by this result, we screened other copper catalysts, including CuSO<sub>4</sub>, Cu(OTf)<sub>2</sub>, Cu<sub>2</sub>O, CuI, and CuCl<sub>2</sub> (entries 2-6, respectively). Among them,  $Cu(OTf)_2$  was the most effective, resulting in the formation of 4a in 76% yield (entry 3). Replacing N-iodosuccinimide with other iodine-containing additives such as I2, ICl, NaIO4, and IBX led to lower yields (entries 7-10, respectively). A suitable oxidant is crucial to this reaction. The use of  $Na_2S_2O_{81}$  (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, oxone, and sulfur significantly decreased the reaction yields (entries 11-14, respectively). Solvents also played an important role. Moderate yields were obtained when reactions were carried out in toluene, acetonitrile, and 1,1,2,2-tetrachloroethane (TTCE) (entries 15–17, respectively). DMF and DMSO as the reaction media completely quenched the reaction (entries 18 and 19, respectively). The control experiments in the absence of a copper catalyst or NIS decreased the product yields to  $\sim 20\%$ (entry 20 or 21, respectively). An only 5% yield of 4a was achieved without an external oxidant (entry 22).

With the effective oxidative cyclization procedure established, the substrate scope of the copper-catalyzed threecomponent oxidative annulation was probed. First, various aryl methyl ketoximes were reacted with 4-methylbenzenethiol (2a) and trifluoroacetic anhydride (3a) (Scheme 1). The isolated yield of model reaction product 4a is 76%. Moreover, when the reaction was conducted on a 6.0 mmol scale, the yield of 4a slightly decreased to 70%. Acetophenone oximes with methyl, isobutyl, and phenyl electron-donating groups were quite compatible, giving the desired products 4b-4d in 65%, 38%, and 61% yields, respectively. The structure of 4d Scheme 1. Scope of Ketoximes<sup>a</sup>



<sup>a</sup>Conditions: 1 (0.2 mmol), 2a (0.3 mmol), 3a (0.8 mmol), Cu(OTf)<sub>2</sub> (5 mol %), NIS (0.12 mmol),  $K_2S_2O_8$  (0.3 mmol), DCE (1 mL), 80 °C, 20 h, air. Isolated yield based on 1. <sup>b</sup>On a 6 mmol scale. <sup>c</sup>2a (2.4 mmol), 3a (3.2 mmol).

was confirmed by X-ray crystallography. Good to high yields were obtained with ketoximes with the fluoro, chloro, bromo, and trifluoromethyl substituents at the para or meta positions on aromatic rings (4e-4g and 4m-4o). The steric effect of the substituents is obvious in this oxidative cyclization (82% for p-Br vs 49% for o-Br). Other substrates with electronwithdrawing groups such as nitro and cyano performed well in the reaction, affording products 4h and 4i, respectively, in high yields. Notably, ester or sulfone groups on the benzene ring were also feasible for this transformation (4j or 4k, respectively). Aryl methyl ketoximes with two functional groups reacted well and gave the trisubstituted oxazole in good yields (4q and 4r). Furthermore, 1-(naphthalen-2yl)ethanone oxime and 1-(phenanthren-3-yl)ethanone oxime afforded products 4s and 4t in 60% and 47% yields, respectively. The heteroaromatic substrate bearing an indole moiety underwent cyclization with 2a and 3a to afford the corresponding product 4v, albeit in 28% yield. Active amino or hydroxyl group substituents on acetophenone oxime were acylated by trifluoroacetic anhydride, and the desired products 4w-4y were obtained in 55%, 63%, and 68% yields, respectively. However, the active hydroxyl group was not acylated by trifluoroacetic anhydride when using 1-(4-hydroxy-3-nitrophenyl)ethan-1-one oxime as a substrate, giving an excellent yield (4u). Notably, 4,4'-diacetylbiphenyl-derived oximes could give 4z in a 40% yield.

To further investigate the scope and limitation of this threecomponent system, various thiophenols were examined under the optimized reaction conditions (Scheme 2). Good yields were obtained with electron-donating groups  $(-^{t}Bu$  and



<sup>a</sup>Conditions: **1a** (0.2 mmol), **2** (0.3 mmol), **3a** (0.8 mmol), Cu(OTf)<sub>2</sub> (5 mol %), NIS (0.12 mmol),  $K_2S_2O_8$  (0.3 mmol), DCE (1 mL), 80 °C, 20 h, air. Isolated yield based on **1a**. <sup>b</sup>Diphenyl disulfide.

 $-OCH_3$ ) at the para position of the benzene ring (4ab and 4ac). Thiophenol derivatives containing halogen substituents, including fluoro, chloro, and bromo, were also available for cyclization, giving the desired products in moderate yields, with no cleavage of the aryl-halide bond observed (4ad-4af). The steric effects of the substituents are apparent. For example, when the methyl was located in the para, ortho, and meta positions on the benzene ring, the corresponding products 4a, 4ag, and 4ak were obtained in 76%, 48%, and 66% yields, respectively. Moderate yields were observed when 2-ethyl, 2-F, and 2-Br thiophenols were used as substrates (4ah-4aj, respectively). To be specific, upon treatment with 2,3dichlorobenzenethiol as the coupling reagent, the yield of the desired product 4al sharply decreased to 22%. Naphthalene-1thiol was compatible with the reaction but gave 4am in a lower yield. It is noteworthy that diphenyl disulfide was also a suitable substrate in this reaction (4an). In current systems, acetic anhydride afforded 4ao in 60% yield. Among other polyfluorinated substituted anhydrides, pentafluoropropionic acid anhydride and perfluorobutyric anhydride could also react to afford products (4ap and 4aq, respectively) in modest yields. Unfortunately, no corresponding products were obtained when alkylthiols were used as substrates.

To understand the mechanistic information, some control experiments were performed (Scheme 3). When we directly used O-trifluoroacetyl oximes 1a' as the substrate, the desired product 4a was formed in 70% yield (Scheme 3, a). During the optimization of the reaction, an only 5% yield of 4a was achieved in the absence of  $K_2S_2O_8$  (Table 1, entry 22), but thioenamine 4a' was observed in 62% yield when acetophenone oxime (1a) reacted with thiophenol (2a) and trifluoroacetic anhydride (3a) (Scheme 3, b). Finally, oxidative annulations of 4a' occurred to produce the corresponding product 4a in 83% yield with the assistance of only  $K_2S_2O_8$  (Scheme 3, c).

#### Scheme 3. Control Experiments



On the basis of the control experiments and previous reports, <sup>13,14</sup> a possible mechanism was proposed in Scheme 4.

Scheme 4. Tentative Reaction Mechanism



First, p-toluenethiol 2a was oxidized by NIS to form sulfur radical intermediate 2a'. On the other hand, oxime trifluoroacetate 1a' is generated in situ from the acylation of ketoxime. Then, imino radical A is formed from 1a' by singleelectron transfer (SET) with Cu(I), which is formed in situ by disproportionation from Cu(II). Intermediate A becomes intermediate B by sequential acylation and isomerization, which coordinates with Cu(I) and 2a' to generate alkyl-Cu(III) intermediate C. Then, reductive elimination of C followed by isomerization occurs to form enamine intermediate product 4a'. The SET oxidation of 4a' generates intermediate E, which further delivers intermediate F via a radical isomerization process. Cyclo radical intermediate G is generated by intramolecular cyclization of F, which may also be promoted by a copper catalyst. The single-electron oxidation of intermediate G followed by deprotonation gives final product 4a.

In summary, we have developed a copper-catalyzed oxidative system that enabled the highly selective formation of 4-aryl-5-(arythio)-2-trifluoromethylated oxazoles. With O-trifluoroace-

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tyl oximes generated in situ, N–O bond cleavages and new C– N bond formation were involved. This reaction system showed a good substrate scope, and various functional groups such as alkyl, ester, and nitro were well tolerated. Mechanistic studies revealed a cascade three-component enamine formation and oxidative 5-endo cyclization.

## ASSOCIATED CONTENT

#### **Supporting Information**

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.or-glett.9b02934.

Experimental procedures, characterization data, and <sup>1</sup>H, <sup>13</sup>C, and <sup>19</sup>F NMR spectra for all new products (PDF)

#### **Accession Codes**

CCDC 1954698 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data\_request/cif, or by emailing data\_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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#### Notes

The authors declare no competing financial interest.

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