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# Visible Light-Mediated Oxidative [3+2] Cycloaddition of Enol and Alkynes to Access Furans

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**Abstract:** Visible light-mediated formation of furans *via* direct oxidative [3+2] cycloaddition of 1,3-diones and alkynes has been described. This protocol provides a simple and mild way to access poly-substituted furans in moderate to good yields. Besides, preliminary mechanistic studies suggest that this reaction most likely undergoes a radical addition/cyclization pathway.

[3+2] cycloaddition has developed to be a well-known methodology for the synthesis of five-membered heterocyclic compounds, such as furans, pyrroles.<sup>1</sup> Until now, classical 1,3-dipolar cycloaddition, as the main strategy for [3+2] cycloaddition reaction, are still limited to some specific substrates such as azomethine ylides, azides, nitrones, nitrile imines and nitrile oxides (Scheme 1a).<sup>2</sup> Moreover, the synthesis of these substrates requires many steps. Thus, direct oxidative [3+2] cycloaddition with the use of simple substrates is undoubtedly an urgent way (Scheme 1b). Although important progress has been made in this field,<sup>3</sup> there still remains a great challenge in optimization of reaction conditions, efficiency and selectivity.

(a) Classical 1,3-dipolar cycloaddition



Poly-substituted furans are found in many natural products, pharmaceuticals and agrochemicals,<sup>4</sup> and also widely utilized as

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synthetic intermediates in organic synthesis (Scheme 2).5 Because of the remarkable applications of furans, chemists focused on using carbonyl compounds as starting materials and spared no effort to develop new routes to synthesize furans under mild and green conditions. More recently, direct oxidative  $\alpha$ -C(sp<sup>3</sup>)–H functionalization of carbonyl compounds, most likely via SET oxidation/radical cyclization, usually requires large excess of metal oxidants, such as Mn(III) and Ce(IV) salts.6 Meanwhile, certain transition-metal catalytic procedures have also been reported for the synthesis of furans.<sup>7,8</sup> Besides, our group reported a similar reaction with molecular iodine as the under oxidative conditions.9 However, promoter this transformation could only tolerate  $\beta$ -ketoesters and gave the corresponding products with a relative low yield at a high temperature of 90 °C. Therefore, developing much greener and milder methods to construct furan motifs from simple alkynes is still in urgent demand.



Anti-pneumocystis carinii activity of carbamates Novel inhibitors of ATP-citrate lyase

Scheme 2. Examples of significant furan-containing pharmaceuticals.

Recently, visible-light photoredox catalysis has been utilized as a powerful tool to construct C-C and C-heteroatom bonds via a single-electron transfer (SET) pathway, playing an important role in large-scale renaissance of radical-based methodologies. Pioneers, such as MacMillian,<sup>10</sup> Stephenson,<sup>11</sup> Nicewicz,<sup>12</sup> Yoon,<sup>13</sup> Xiao<sup>14</sup> and Wu<sup>15</sup> have done lots of excellent work in cross-coupling reactions and intermolecular cycloadditions. Nevertheless, only a few cases were reported about the synthesis of furans through visible-light-mediated [3+2] cycloaddition, which still utilized halogenated substrates as reactants.<sup>16</sup> From the perspective of sustainable chemistry and atom-economy, the possibility of direct C-H functionalization, especially the oxidative coupling between two C-H bonds, provides a highly attractive strategy for an ideal chemical synthesis. Herein, a visible light-mediated oxidative [3+2] cycloaddition of 1,3-diones and alkynes to access furans was reported, which illustrated an efficient example of constructing furans in one step under mild conditions.

Our initial efforts were focused on the reaction between ethyl cyclohexane-1,3-dione **1a** and phenylacetylene **2a** by using  $Ru(bpy)_3Cl_2$  as the photocatalyst and  $(NH_4)_2S_2O_8$  as an oxidant under the irradiation of 3 W blue LEDs at room temperature. As a result, the desired 2-phenyl-6,7-dihydrobenzofuran-4(5H)-one product **3a** can be obtained in 35% yield (Table 1, entry 1). To modify this model reaction, photocatalysts such as  $Ir(ppy)_3$ ,

eosin Y, Acr<sup>+</sup>-MesClO<sub>4</sub><sup>-</sup>, rhodamine B and methylene blue will be firstly alternated and compared (Table 1, entries 2 - 6). When the methylene blue was employed as the photocatalyst, the yield of product could increase to 83% (Table 1, entry 6). In addition, as the oxidant was changed to other peroxodisulfates, such as Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, the yield dropped significantly (Table 1, entries 7, 8). The formation of product was corresponding to the equivalents of diacrbonyls, when **1a** reached to 4 equivalents (0.8 mmol), the desired product was able to obtained in 83% yield. Continuously increase **1a** didn't improve the yield (for details, see the Supporting Information (Figure 1)). The control experiments showed that only trace product could be detected in the absence of light, photocatalyst or (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (Table 1, entries 9 - 11).

**Table 1.** Optimization of conditions for the reaction of cyclohexane-1,3-dione **1a** and phenylacetylene **2a**.<sup>[a]</sup>



[a] Conditions: **1a** (0.8 mmol), **2a** (0.2 mmol), Photocatalyst (6 mol%) and Peroxodisulfate (2.0 equiv.) in 3 mL CH<sub>3</sub>CN under a nitrogen atmosphere, irradiation with 3 W blue LEDs, room temperature, 12 h. [b] Isolated yields. [c] Without light.

With the optimized conditions in hand, we firstly focused on investigating the scope of terminal aryl alkynes (Table 2). The reaction was readily extended to different aryl-substituted terminal alkynes. Good substrate tolerance of alkynes could be achieved with both electron-withdrawing and electron-donating functional groups, such as H-, CH<sub>3</sub>-, (CH<sub>3</sub>)<sub>2</sub>CH-, C<sub>5</sub>H<sub>11</sub>-, Ph-, CH<sub>3</sub>O-, naphthyl- (Table 2, **3a**, **3b** – **3d**, **3e**, **3f**, **3g**, **3h**, **3o**) and EtOOC-, CF<sub>3</sub>-, NO<sub>2</sub>- (Table 2, **3i** and Table 3, **3v**, **3w**). In addition, halogen substituted groups, regardless of *ortho, meta* or *para*, such as fluoro, chloro and bromo could also give the corresponding furans in good yields (Table 2, **3j** – **3n**). Alkynes with heterocyclic aromatic ring, for example 1,3-benzodioxolyl and thiophenyl alkyne, could also afford a moderate yield of the desired products (Table 2, **3p**, **3q**). Unfortunately, under the

standard conditions, although the furan product could be achieved by the reaction of 1,3-diones **1** and terminal alkynes **2**, the alkyl alkynes could not be tolerated in this reaction (for details, see the Supporting Information (Figure 2a)). A possible explain is that the stability of olefinic carbon radicals, which is generated from the radical addition of  $\alpha$ -carbonyl alkyl radicals to terminate alkynes, has a significant effect on the yield of this cycloaddition reaction. Besides, the conjugated effect of the aromatic ring would make the olefinic carbon radical more stable. Thus, the aromatic ring of terminal alkynes is necessary. On the other hand, some internal alkynes have also been tested in this reaction (for details, see the Supporting Information (Figure 2b)). Yet the steric effect of these substrates made the  $\alpha$ -carbonyl alkyl radical difficult to accomplish the radical addition to internal alkynes.

 Table 2.
 Substrate scope for visible light-mediated oxidative
 [3+2] cycloaddition of 1,3-cyclohexanedione 1a and Alkynes 2.<sup>[a]</sup>



[a] Conditions: **1a** (0.8 mmol), **2** (0.2 mmol), methylene blue (6 mol%) and  $(NH_4)_2S_2O_8$  (2.0 equiv.) in 3 mL CH<sub>3</sub>CN under a nitrogen atmosphere, irradiation with 3 W blue LEDs, room temperature, 12 h; isolated yields. [b] 1 mL acetone was added in the system.

The scope of the 1,3-dione components have been explored as well (Table 3). For instance, 5-phenylcyclohexane-1,3-dione, 5,5-dimethylcyclohexane-1,3-dione, 5-dimethylcyclohexane-1,3dione, 4,4-dimethylcyclohexane-1,3-dione and chromane 2,4dione could be well tolerated under the reaction conditions (Table 3, 3r - 3x). Interestingly, 4,4-dimethylcyclohexane-1,3dione and chromane-2,4-dione could give two different isomers in nearly 1/1 ratio (Table 3, 3u, 3x). Five-membered 1,3-dione could also be tolerated under the reaction conditions, yet it resulted in a relative low yield compared to the six-membered 1,3-dione (Table 3, 3y).  $\beta$ -Ketoester and acetylacetone were also tried with our protocol. Some efforts were made to realize this transformation by using ethylacetoacetate as the model substrate. Unfortunately, no matter which photocatalysts and oxidants we have tried, the reaction could not proceed (for details, see the Supporting Information (Figure 3a and 3b)).

To gain more insight of the reaction mechanism, radical trapping reagents such as TEMPO, BHT and 1,1-stilbene have been introduced to the reaction. Interestingly, only 10% of the desired product was obtained with BHT; TEMPO could totally

**Table 3.** Substrate scope for visible light-mediated oxidative [3+2] cycloaddition of 1,3-Dione **1** and Alkynes **2**.<sup>[a]</sup>



[a] Conditions: **1** (0.8 mmol), **2** (0.2 mmol), methylene blue (6 mol%) of and  $(NH_4)_2S_2O_8$  (2.0 equiv.) in 3 mL CH<sub>3</sub>CN under a nitrogen atmosphere, irradiation with 3 W blue LEDs, room temperature, 12 h; isolated yields. [b] Increasing the temperature to 80 °C.

inhibit the reaction. Additionally, when 1,1-stilbene was added, it would compete with phenylacetylene and cyclize with 1,3-dione to generate the 2,2-diphenyl-3,5,6,7-tetrahydroben-zofuran-4(2H)-one **4a** in 70% yield (Scheme 3). In some cases, 1,1-stilbene can be used as a radical-trapping reagent as well. Thus, these results suggested that the cyclization reaction probably proceeds through a radical process.



Scheme 3. Mechanistic studies

Based on the above results, we proposed a general mechanism for this oxidative [3+2] cycloaddition between **1a** and **2a** (Scheme 4). Firstly, the photocatalyst can be activated to the excited-state (PC\*, methylene blue) by the visible light irradiation (3 W blue LEDs).<sup>17</sup> The oxidant, persulfate  $(S_2O_8^{-2})$ , then was reduced by the PC\* to form the sulfate radical anion  $(SO_4^{-1})^{18}$ . Following the hydrogen atom transfer (HAT) between **1a** and sulfate radical anion, a new carbon radical (**Int-A**) was generated. Subsequently, radical addition of **Int-A** to **2a** gives an olefinic carbon radical (**Int-B**), which further cyclized to the carbonyl oxygen to form a five-membered ring (**Int-C**) through

intramolecular radical addition. Finally, the product **3a** is obtained after the oxidation of **Int-C** by the PC<sup>++</sup> and the deprotonation procedure.



Scheme 4. Proposed mechanism.

In summary, we have developed a visible light-mediated oxidative [3+2] cycloaddition of enol and alkynes to access furans. The use of cheap and innocuous methylene blue in combination with  $(NH_4)_2S_2O_8$  as a terminal oxidant could well accomplish these transformations. Synthetically, the present protocol provides an atom-economic and highly-selective way to construct the poly-substituted furans in good yields under the metal free, mild and simple conditions. Mechanistically, the reaction most likely undergoes a radical addition/cyclization mechanism.

#### **Experimental Section**

1,3-Dione **1** (0.8 mmol, 89.6 mg), methylene blue (0.012 mmol, 3.5 mg) and (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.4 mmol, 91.2 mg) (from glove box) were added in a clean sealed Schlenk tube equipped with a stir bar. After flushing the Schlenk tube three times with nitrogen atmosphere, CH<sub>3</sub>CN (3 mL) and alkyne **2** (0.2 mmol, 20.4 mg) were injected in the tube through a syringe in turn. The reaction mixture was irradiated with 3 W blue LEDs and stirred at room temperature. After completion of the reaction, as indicated by TLC and GC-MS, the mixture was then purified by column chromatography on silica gel using petroleum ether and ethyl acetate as eluent to afford pure product **3**.

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We have developed a visible light-mediated oxidative [3+2] cycloaddition of enol and alkynes to access furans. This protocol provides a simple and mild way to access poly-substituted furans in moderate to good yields.

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