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Communication

Rapid alkenylation of quinoxalin-2(1*H*)-ones enabled by the sequential Mannich-type reaction and solar photocatalysis

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ABSTRACT

Herein, a rapid alkenylation of quinoxalin-2(1*H*)-ones enabled by a combination of Mannich-type reaction and solar photocatalysis is demonstrated. A wide range of functional groups are compatible, affording the corresponding products in moderate-to-good yields. Control experiments illustrate that the *in situ* generated $^1O_2$ plays a central role in this reaction. This green and efficient strategy provides a practical solution for the synthesis of potentially bioactive compounds that containing a 3,4-dihydroquinoxalin-2(1*H*)-one structure.

Quinoxalin-2(1*H*)-one, as a significant heterocyclic unit, has been found important applications in synthetic chemistry, materials, natural products and pharmaceuticals because of their innate outstanding biological activities and excellent chemical characters [1], and their biological activities can be significantly influenced if the substituents is introduced into the N1- and C3-positions of the quinoxalin-2(1*H*)-one [2]. In particular, 3-substituted quinoxalin-2(1*H*)-ones have been developed into powerful drugs due to their strong pharmacological effects [3], such as ataquimast, antinicrobial, anticancer, Fxa coagulation inhibitors and glycogen phosphorylase inhibitor (Fig. 1) [4]. Therefore, a number of methods have been developed for their synthesis [5]. Generally, they are synthesized by cyclization of derivatives of aniline or 1,2-diaminobenzene with suitable partners. However, the disadvantages including pre-functionalization of the partners and multi-step synthesis limit its application [6]. In recent years, direct C—H bond functionalization at the C3-position of quinoxalin-2(1*H*)-one has become a straightforward access to the 3-substituted quinoxalin-2(1*H*)-one derivatives, and various remarkable work has been achieved [7–12]. For instances, our group in 2019 reported a first example of oxidative C—H fluoroalkoxylation of quinoxalinones with fluoroalkyl alcohols under transition-metal and solvent-free conditions [8b]. This method can also be extended to the facile and efficient synthesis of histamine-4 receptor. The same year, Sun's group presented an efficient electrochemical approach for the C(sp^2)–H phosphonation of quinoxalin-2(1*H*)-ones and C(sp^3)–H phosphonation of xanthenes [9a]. More interestingly, the group of Pan disclosed a photocatalyst-free visible-light-promoted sulfenylation of quinoxalinones with thiols *via* cross-dehydrogenative coupling [10b]. Shortly after this discovery, He's group demonstrated a visible-light-promoted amidation of quinoxalin-2(1*H*)-ones [11b]. In a very recent contribution, a mild and eco-friendly visible-light-induced decarboxylative acylation of quinoxalin-2(1*H*)-ones with α-oxo carboxylic acids using ambient air as the sole oxidant at room temperature was also established by the same group [12a]. In sharp contrast, the alkenylation of quinoxalin-2(1*H*)-ones was rarely reported.

Photocatalysis has become a powerful strategy for organic synthesis due to the advantages of low energy consumption and environmental protection [13]. For example, MacMillan *et al.* in 2016 reported a photocatalyzed C—H arylation of aliphatic amines with aryl bromides, providing a complement to existing cross-coupling technologies [13a]. In 2021, He' group developed the first example of visible-light induced one-pot tandem reaction of arylacrylamides, CHF_2CO_2H and $PhI(OAc)_2$, affording an eco-friendly and practical method to access various difluoromethylated oxindoles [13b]. The same year, Jin and coworkers developed

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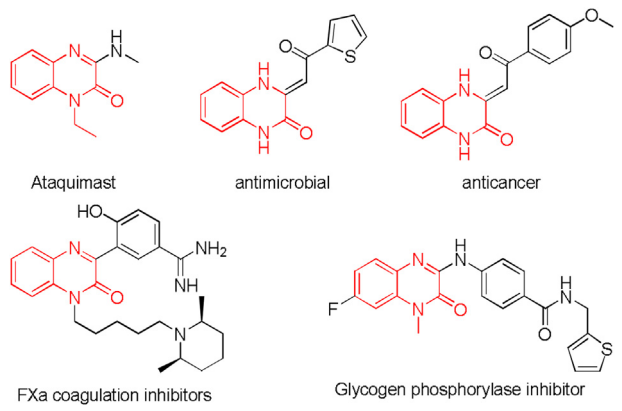
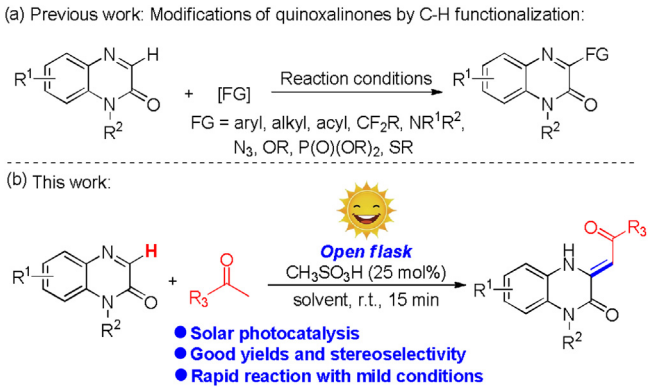


Fig. 1. Examples of quinoxalin-2(1*H*)-one skeleton-based bioactive molecules.

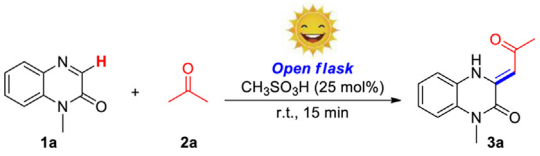
photocatalyst-free radical tandem cyclization of quinazolinones containing an unactivated alkene moiety with difluoro bromides under illumination, giving a practical method for the synthesis of fluorine-containing ring-fused quinazolinones [13f]. In recent years, with increasing attention to renewable energy, considerable efforts have been switched to the development of photocatalytic reactions that excited by the sunlight, which is known as a renewable and simple accessible light source [14]. Our research interests focus on the development of novel and effective methodologies for the direct modification of *N*-containing heterocycles [15], herein, we demonstrated a direct alkenylation reaction between quinoxalin-2(1*H*)-ones and methyl ketones. Compared with our previous work [15a], this transformation was achieved through a combination of Mannich-type reaction and solar photocatalysis, which could be completed within 15 min, providing a green and efficient solution for the synthesis of potentially bioactive compounds that containing a 3,4-dihydroquinoxalin-2(1*H*)-one structure (Scheme 1b).

1-Methylquinoxalin-2(1*H*)-one (**1a**) and acetone (**2a**) were chosen as starting materials to screen the reaction conditions. The target product (**3a**) was obtained in 80% yield when the reaction was performed by using 25 mol% of CH_3SO_3H as a catalyst under the irradiation of sunlight for 15 min (Table 1, entry 1). Other acid catalyst, such as CF_3COOH and HBF_4 gave the relative lower yield under the same conditions (Table 1, entries 2 and 3). No product was obtained in the absence of any acid catalyst (Table 1, entry 4). When used MeCN or DMF as solvent and 2.0 equiv. of acetone as substrate, 76% or 52% yield was obtained respectively (Table 1, entries 5 and 6). Extended reaction time to 30 min did not enhanced product yield (Table 1, entry 7). There was no desired product generated when the reaction was carried out under dark condition (Table 1, entry 8).



Scheme 1. Modification of quinoxalin-2(1*H*)-ones by C-H functionalization.

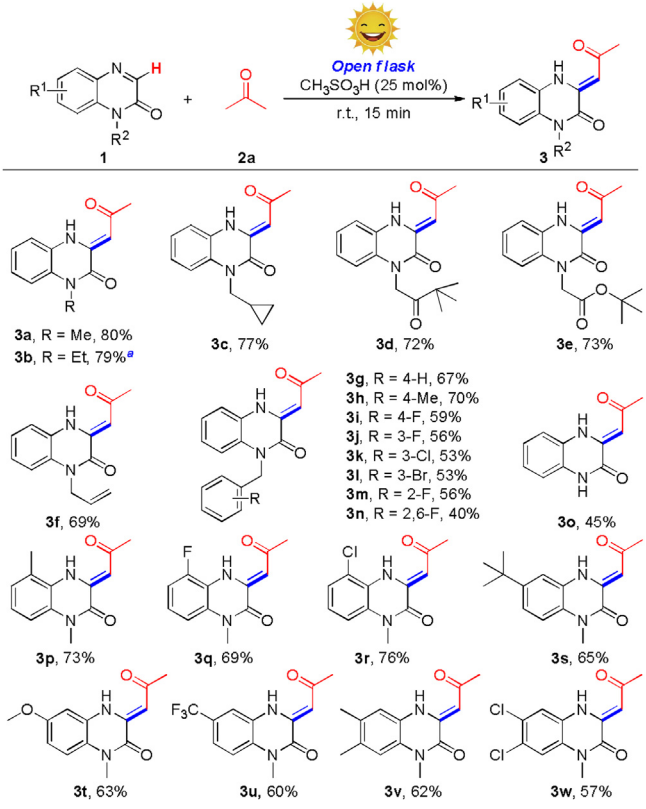
Table 1
Screening of reaction conditions.[a]



Entry	Variation from given conditions	Yield (%)[b]
1	none	80
2	CH_3SO_3H was replaced with CF_3CO_2H	48
3	CH_3SO_3H was replaced with HBF_4	36
4	No CH_3SO_3H	0
5[c]	acetone was replaced with MeCN	76
6[c]	acetone was replaced with DMF	52
7	extend reaction to 30 min	78
8	under dark condition	0

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (1.0 mL), CH_3SO_3H (25 mol%), open flask, sunlight, room temperature, 15 min.
[b] Isolated yields.
[c] 2.0 equiv. of acetone were used.

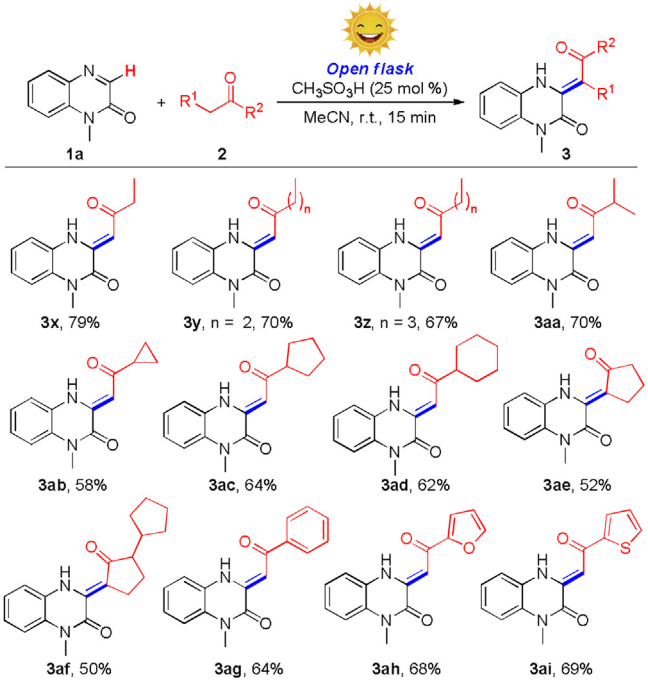
With the optimum reaction conditions in hands, we then examined the substrate scope of the reaction by employing various quinoxalin-2(1*H*)-ones (**1**) with acetone (**2a**) (Scheme 2). Firstly, the *N*-substituted groups such as *N*-methyl, *N*-ethyl, *N*-cyclopropylmethyl, *N*-keto and *N*-ester were well compatible under the standard conditions, giving the desired products (**3a-e**) in 72%–80% yields. It is worth mentioning that quinoxalin-2(1*H*)-one with a sensitive allyl group, which could be further functionalized, also



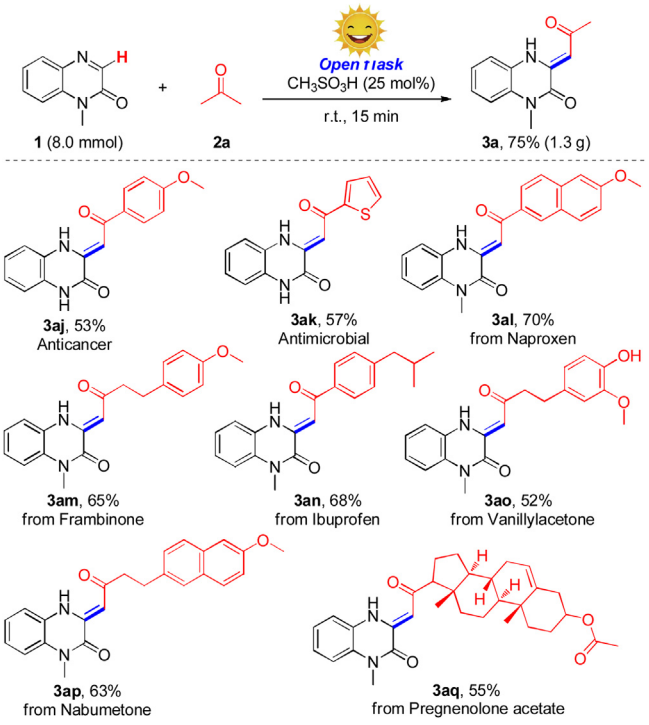
Scheme 2. Substrate scope of quinoxalin-2(1*H*)-ones. Reaction conditions: **1** (0.2 mmol), **2a** (1.0 mL), CH_3SO_3H (25 mol%), open flask, sunlight, room temperature, 15 min. Isolated yields. [a] Reaction was performed on a 1 mmol scale.

could give the product (**3f**) in 69% yield. A wide range of quinoxalin-2(1*H*)-ones with different benzyl groups, bearing both electron-donating and electron-withdrawing substituents at *ortho*-, *meta*-, or *para*-position could undergo the reaction smoothly, affording the corresponding products (**3g–n**) in 40%–72% yields. Importantly, the *N*-free quinoxalin-2(1*H*)-one could undergo the reaction smoothly, providing the product (**3o**) in 45% yield. Besides, plenty of quinoxalin-2(1*H*)-ones that bear the functional groups including methyl, halogen, *tert*-butyl, methoxy or trifluoromethyl at C5-, C6- or C7-position also gave the desired products in satisfactory yield (**3p**-**3w**). To expand the substrate scope of *N*-heterocycles, we also tested quinoline, isoquinoline, quinoxaline, benzimidazole and benzothiazole under standard conditions, however, no corresponding product was obtained (see Supporting information).

Subsequently, we evaluated the substrate scope of methyl ketones for the reaction (Scheme 3). To reduce the dosage of reactant, the reactions were performed with 2.0 equiv. of methyl ketones by using acetonitrile as solvent. To our delight, both long-chain and cycloalkyl methyl ketones could undergo the reaction smoothly, giving the corresponding products (**3x**–**3ad**) in 58%–79% yields. The molecular structure of **3y** was confirmed by X-ray crystallographic analysis (CCDC: 2060383). It was found that the molecular structure was more stable in (*Z*)-configuration probably because the effect of hydrogen bond interaction between amine and carbonyl group. Then, we found that cyclopentanone skeleton could also react with quinoxalin-2(1*H*)-one smoothly to deliver the target products (**3ae** and **3af**) in moderate yield. The subsequent exploration found that the aryl methyl ketones, such as acetophenone, 1-(furan-2-yl)ethan-1-one and 1-(thiophen-2-yl)ethan-1-one were also could be converted into target products (**3ag**-**3ai**) in acceptable yields. Unfortunately, the substrates like ethyl acetate, acetonitrile, nitromethane, ethyl acetoacetate, and acetylacetone were not compatible under standard conditions (Supporting information).

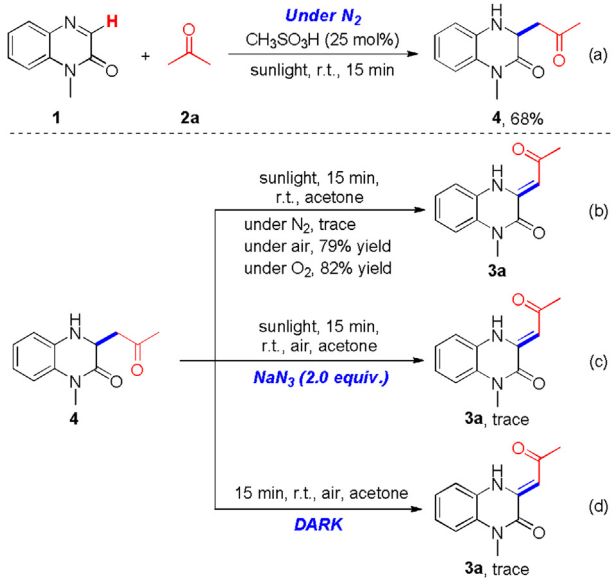


Scheme 3. Substrate scope of methyl ketones. Reaction conditions: **1a** (0.2 mmol), **2** (2.0 equiv.), CH_3SO_3H (25 mol%), MeCN (1.0 mL), open flask, sunlight, room temperature, 15 min. Isolated yields.

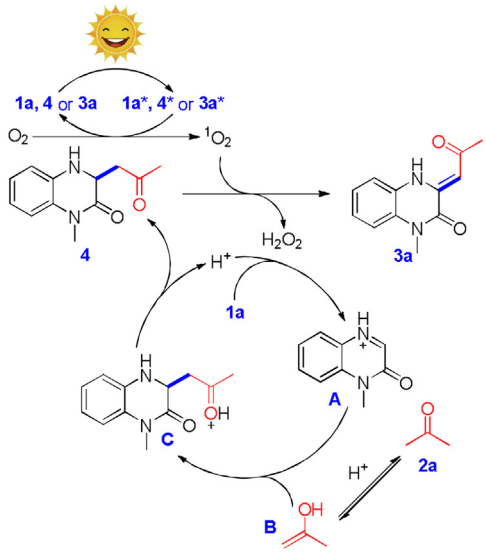


Scheme 4. Gram-scale synthesis and application. Reaction conditions: **1a** (0.2 mmol), **2** (2.0 equiv.), CH_3SO_3H (25 mol%), MeCN (1.0 mL), open flask, sunlight, room temperature, 15 min. Isolated yields.

To show the synthetic utility of this protocol, a gram-scale synthesis experiment was performed to give the target product (**3a**) in 75% yield (Scheme 4). Interestingly, the anticancer compound (**3aj**) and antimicrobial compound (**3ak**) were obtained in moderate yields by using our strategy [16]. Moreover, since the molecules that bearing a 3,4 dihydroquinoxalin-2(1*H*)-one framework are a promising class of biologically active compounds, in this regard, several bioactive molecules such as naproxen derivative, frambinone, ibuprofen derivative, vanillylacetone, nabumetone and pregnenolone acetate were selected to react with 1-methylquinoxalin-2(1*H*)-one directly,



Scheme 5. Control experiments.



Scheme 6. Plausible mechanism.

providing the potentially active molecules (**3al**-**3aq**) in 52%–70% yields.

To study the reaction mechanism, a series of control experiments were carried out. Product **4** was generated instead of target product **3a** when the reaction was performed under nitrogen atmosphere (Scheme 5a). This result showed that oxygen in air was included in the subsequent oxidation process. To confirm the assumption, the oxidation process of compound **4** was studied. First, target product **3** was formed in 0%, 79% and 82% yields when the reaction performed under nitrogen, air or oxygen atmosphere respectively (Scheme 5b). Second, the reaction was inhibited when singlet oxygen inhibitor (NaN_3) was involved in the transformation (Scheme 5c). Furthermore, compound **4** could not be converted into target product **3** when the reaction was performed in dark condition (Scheme 5d). These experimental results strongly supported that the singlet oxygen $^1O_2$, which was generated from triplet oxygen $^3O_2$ through photocatalysis, serves as the real oxidant.

On the basis of above results and previous reports [8–12], we proposed a possible mechanism for this reaction (Scheme 6). Firstly, substrate **1a** was transformed into intermediate **A** through a protonation process. Meanwhile, acetone **2a** was converted to the enol form **B** under acidic condition. Then, a Mannich-type reaction took place between intermediates **A** and **B** to give the intermediate **C,** which underwent a deprotonation process to provide the key compound **4**. It was found that organic molecules that containing a quinoxalin-2(1*H*)-one skeleton could act as a photosensitizer to generate $^1O_2$ from O_2 under the irradiation of visible light [12p]. In this regard, compounds **1a**, **4** or **3a** was excited by visible light to provide the excited-species **1a***, **4*** or **3a***, which acted as a photosensitizer and underwent an energy transfer (ET) process with O_2 to give $^1O_2$, along with the regeneration of ground-state compounds **1a**, **4** or **3a**. Finally, compound **4** underwent the single-electron-transfer (SET) process with $^1O_2$ to give the desired product with the generation of H_2O_2, which was detected by H_2O_2 test paper (Supporting information) [12a] [17]. We proposed that the electron-withdrawing effects of carbonyl group that exists in quinoxalin-2(1*H*)-one skeleton lower down the electron cloud density of the enamine moiety, making it difficult to be oxidized and can survive under this H_2O_2 oxidation conditions.

In conclusion, this study described a novel strategy for the olefination of quinoxalin-2(1*H*)-ones with methyl ketones. Various substrates were compatible under standard condition, providing the corresponding products in moderate to good yields. Control experiments revealed that a Mannich-type reaction and oxidative process were involved in the transformation.

Declaration of competing interest

The authors declare that they have no conflict of interest.

Acknowledgments

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.cclet.2021.04.016.

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