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DTBP-promoted decarbonylative alkylation of quinoxaline-2(1*H*)-ones with aldehydes

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ABSTRACT

An efficient DTBP-promoted direct alkylation of quinoxaline-2(1*H*)-ones with aldehydes has been developed under metal-free conditions. The reaction undergoes sequential decarbonylation and radical addition using aliphatic aldehyde as a cheap and abundant alkyl radical source. A convenient and efficient approach for the synthesis of various 3-alkylquinoxaline-2(1*H*)-ones was provided.

Introduction

Direct C—H alkylation of *N*-heteroarenes remains a formidable challenge to the synthetic organic chemist due to their broad pharmacological activity and ability to tune physicochemical properties [1,2]. Recently, a number of methodologies have been developed by using different alkylating agents such as alkyl halides, alkyltrifluoroborates, boronic acids, acid chlorides etc. [3]. Moreover, C—H alkylation products of azaarenes can also be obtained via cross dehydrogenative coupling (CDC) reaction of simple alkanes [4]. However, the use of aliphatic aldehyde for alkylation still remains a challenge. Aliphatic aldehydes have been utilized as sources of both acyl radicals and alkyl radicals, but it is very difficult to selectively control acylation or alkylation [5]. Thus, great efforts are devoted to the selective formation and reaction of alkyl radicals from aldehydes via oxidative decarbonylation [6]. On the other hand, aldehydes are cheap and abundant chemicals and have been directly used as precursors for (oxidative) decarbonylative couplings catalyzed by ruthenium or rhodium, as shown by the extensive studies of Li and co-workers since 2009 [7]. After that, Paul's group reported another metal-free oxidative decarbonylative coupling of aliphatic aldehydes with azaarenes in 2015 [8]. However, majority of these methods had several disadvantages such as the use of over stoichiometric amount of peroxide as initiator and oxidant, metal salt as additive and so on [9]. Therefore, an environmentally friendly method to make aldehyde as alkylation reagent is still in great demand (Fig. 1).

Quinoxalin-2(1*H*)-ones, as significant heterocyclic units, have found important application in synthetic chemistry, material, natural products and pharmaceuticals because of their innate outstanding biological activities and excellent chemical characters [10], Particularly, 3-alkylated quinoxalin-2(1*H*)-ones possess a number of biological activities such as anticancer, antiviral, and c-Met kinase inhibitory properties [11]. Generally, 3-alkylquinoxalin-2(1*H*)-ones are prepared by the reaction of aryl-1,2-diamines with alkyl-substituted keto acids or esters through a multistep procedure [12]. Recently, the C3 − H functionalization of quinoxalin-2(1*H*)-ones has emerged as a powerful method to construct various 3-substitued quinoxalin2(1*H*)-ones [13]. Among them, the 3-alkylation of quinoxalin-2(1*H*)-ones have also been reported [14]. For instance, in 2018, Guo's group described Fe-catalyzed synthesis of 3-cyanoalkylated quinoxalin-2(1*H*)-ones through the alkylation reaction of cyclobutanone oxime esters with quinoxalin-2(1*H*)-ones [15]. To the best of our knowledge, direct C-3 alkylation of quinoxaline-2(1*H*)-ones using aliphatic aldehyde as alkylation reagent has not been reported. Herein, we describe an effective and transition metal-free oxidative consecutive decarbonylation coupling of aliphatic aldehydes with quinoxaline-2(1*H*)-ones in the presence of DTBP (*Di-tert*-butyl peroxide) (Scheme 1c).

Results and discussion

Initially, the alkylation of 1-methyquinoxalin-2(1*H*)-ones with isobutyraldehyde was selected as a model reaction for the

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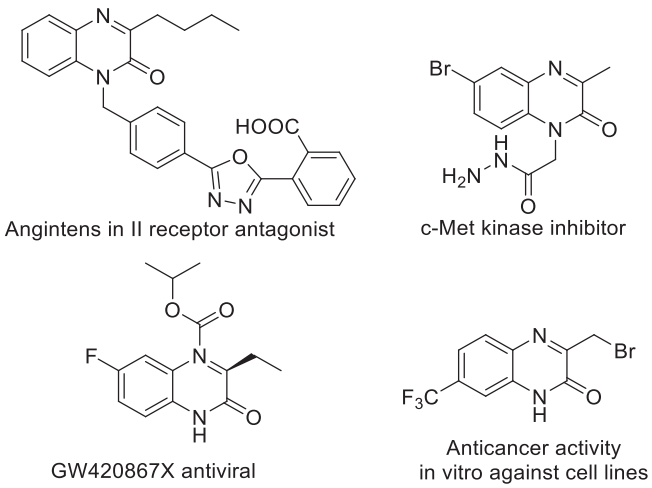
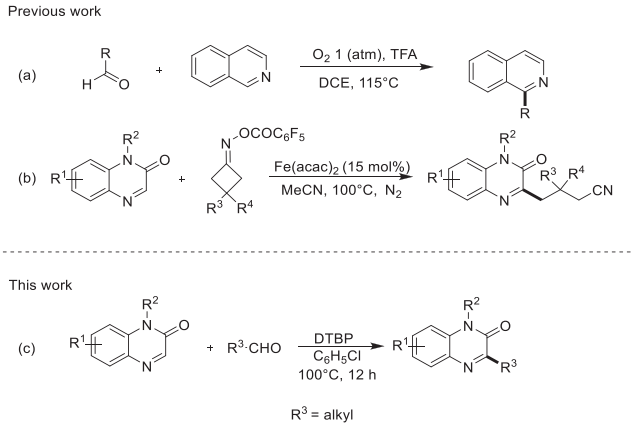


Fig. 1. Examples of bioactive 3-substituted quinoxalin-2(1*H*)-one derivative molecules.



Scheme 1. Synthesis of 3-alkylquinoxaline-2(1*H*)-ones and aldehyde as alkylation reagent.

optimizztion of reaction conditions. Gratifyingly, the treatment of 1-methylquinoxalin-2(1*H*)-one (**1a**, 0.2 mmol) with isobutyraldehyde (**2a**, 0.4 mmol) in the presence of DTBP (3 equiv) in 2 mL of 1,2-DCB (1,2-Dichlorobenzene) at 100 ℃ for 12 h under the atmosphere of air leads to the desired product **3aa** in 65% yield; the result is illustrated in Table 1. The structure of **3aa** was unambiguously confirmed by NMR and MS. Motivated by the above result, a screening of the reaction conditions with respect to solvent, peroxide, catalyst, temperature and time was performed. Notably, the product yield increased by about 17% when the reaction solvent changed from 1,2-DCB to the chlorobenzene (Table 1, entry 6). However, use of other solvents such as DMF (*N, N*-Dimethylformamide), toluene, EtOH, and CH_3OH, only gave trace amount of product (Table 1, entries 2–5). Different peroxide such as TBHP (*Tert-butyl* hydroperoxide) (70% in water), H_2O_2 (30% in water), DCP (Dicumyl peroxide), CHP (Cumene hydroperxide) and $K_2S_2O_8$ were also tested in this reaction but the yield of the product decreased instead (Table 1, entries 7–11). Reducing the amount of DTBP from 3 equiv to 1.5 equiv not resulted in a decrease of yield (Table 1, entries 12–13). That the reaction hardly occurred in the absence of DTBP indicated that the alkylation was mainly mediated by DTBP. Various catalyst including $Ni(CH_3COO)_2{\cdot}4H_2O$, $CuBr_2$, $Mn(OAc)_2{\cdot}2H_2O$, $Mn(OAc)_2{\cdot}4H_2O$ and $Mn(OAc)_2$ were investigated for this transformation (Table 1, entries 14–18). The yield of **3aa** was not improved but only a trace amount of product was obtained instead. Moreover, in view of the experimental results and current trend in organic synthesis, metal catalyst was not employed for this reaction. Then, the effect of temperature was studied, and 100 ℃ was founded to be the best choice (Table 1, entries 19–20). Finally, the reaction time was also screened, and 12 h was proved be optimal time (Table 1, entries 21–22). Further optimization found that the yield was not increased when the reaction was protected by nitrogen atmosphere.

Overall, the optimal reaction conditions were set as follows: **1a** (0.2 mmol), **2a** (2.0 equiv), DTBP (1.5 equiv) in chlorobenzene (2 mL) under air at 100 ℃ for 12 h.

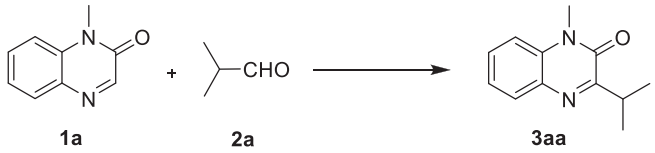
With the optimized conditions in hand, we turn our attention to embark on the scope of quinoxaline-2(1*H*)-ones for this transformation. The results are summarized in Table 2. Firstly, the effect of protecting groups on the amide nitrogen atom of quinoxaline-2(1*H*)-ones was examined. As expected, the corresponding products were isolated in moderate to high yields for all the tested substrates. *N*-Methyl and *N*-ethyl substrates generated the corresponding products in 88% and 85% yields respectively, and free amide gave the yield of 67% **(3aa-3ac)**. The yield of *N*-benzyls ranged from 65% to 77% (**3ad-3ag**), and the ethyl 2-(3-isopropyl-2-oxoquinoxalin-1(2*H*)-yl) acetate offered the corresponding product in 65% yield (**3aj**). It turns out that various *N*-substituents included electron-donating and electron-withdrawing ones have no obvious effects on this reaction. In addition, the product yield of *N*-propyne is nearly 15% higher than that of *N*-allylic (**3ah-3ai**). We further investigated the effects of substituents on benzene ring of quinoxalinone-2(1*H*)-ones. 6,7-Dimethyl-1-methyquinoxalin-2(1*H*)-one generated 3ak in 74% yield. More excitingly, the yield of the bis-chlorine substituted substrate on the benzene ring is the highest, up to 93% (**3al**). Similarly, **1m** and **1n** offered the corresponding products **3am** and 3an in good yields. To our delight, when 6-bromo-1-methylquinoxaline-2(1*H*)-one was employed, the reaction was also tolerated well to provide the product **3ao** and **3ap** with 82% and 85% yields, respectively. Interestingly, the yields of mixed products of methyl substitution on the benzene ring is up to 90% (**3aq**). Subsequently, other heterocycles were tested to react with isobutyraldehyde, such as quinoline, Isoquinoline, pyridine, imidazopyridine, benzothiazole and so on. However, the results were not satisfactory, and only quinoline provided the corresponding product as detected by GC-MS.

To further expand the substrate scope of this transformation, a broad range of aliphatic aldehydes with varied alkyl chains were screened. As shown in Table 3, the yields of acetaldehyde, propionaldehyde, butyraldehyde, isovaleraldehyde, valeraldehyde, Lily aldehyde and 2-Phenylpropionaldehyde were 38%-69% (**3ba-3ha**), In addition, the quinoxaline-2(1*H*)-one substituted by dichloro on benzene ring was used to react with valeraldehyde, and the yield of product rose to 72% **(3ia).** The yield of cyclohexyl aldehyde is as high as 78% (**3ja**). Encouraged by this result, *N*-substituted and aryl ring substituted quinoxalin-2(1*H*)-ones were used to react with cyclohexyl aldehyde, all of these reactions gave good yields of 80%-86% (**3ka-3ma**).

To explore the practical application of this method, a gram-scale experiment was performed. As described in Scheme 2, the reaction of quinoxaline-2(1*H*)-one **1a** (7 mmol, 1.0 g) with **2** was conducted effectively to produce the product **3a** in 76% yield (0.99 g). This result suggested that this method can be applied as a practical protocol for large-scale preparation of 3-alkylquinoxalin-2(1*H*)-ones.

To derive a plausible mechanism for this transformation, a serial of control experiment was carried out. When 1.5 equiv of 2,2,6,6-tetramethy-1-piperidinyloxy (TEMPO) was added to the reaction system under the standard conditions, the alkylation was inhibited completely. At the same time, the reaction was suppressed remarkably in the presence of butylated hydroxytoluene (BHT) and the trapping adduct of BHT with isobutyraldehyde

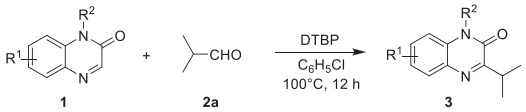
Table 1
Optimization of reaction conditions.[a,b]

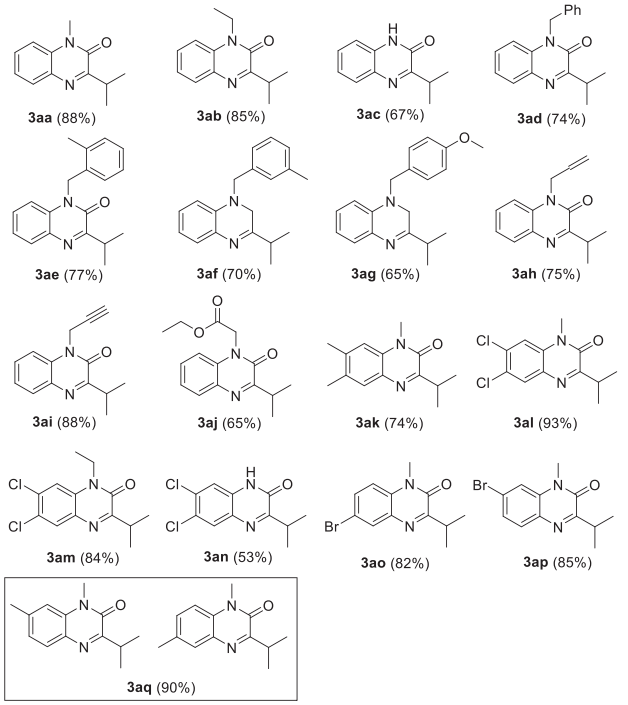


Entry	Oxidant (equiv)	Catalyst (equiv)	Solvents	Yield [b]
1	DTBP (3.0)	–	1,2-DCB	65%
2	DTBP (3.0)	–	DMF	23%
3	DTBP (3.0)	–	EtOH	25%
4	DTBP (3.0)	–	Toluene	Trace
5	DTBP (3.0)	–	CH_3OH	17%
6	DTBP (3.0)	–	C_6H_5Cl	82%
7	TBHP (3.0)	–	C_6H_5Cl	36%
8	H_2O_2 (3.0)	–	C_6H_5Cl	30%
9	DCP	–	C_6H_5Cl	28%
10	CHP	–	C_6H_5Cl	15%
11	$K_2S_2O_8$	–	C_6H_5Cl	Trace
12	DTBP (1.5)	–	C_6H_5Cl	88%
13	DTBP (1.0)	–	C_6H_5Cl	63%
14	DTBP (1.5)	$Ni(CH_3COO)_2{\cdot}4H_2O$	C_6H_5Cl	41%
15	DTBP (1.5)	$CuBr_2$	C_6H_5Cl	Trace
16	DTBP (1.5)	$Mn(OAc)_3{\cdot}2H_2O$	C_6H_5Cl	49%
17	DTBP (1.5)	$Mn(OAc)_2{\cdot}4H_2O$	C_6H_5Cl	43%
18	DTBP (1.5)	$Mn(OAc)_2$	C_6H_5Cl	39%
19 [c]	DTBP (1.5)	–	C_6H_5Cl	66%
20 [d]	DTBP (1.5)	–	C_6H_5Cl	85%
21 [e]	DTBP (1.5)	–	C_6H_5Cl	74%
22 [f]	DTBP (1.5)	–	C_6H_5Cl	83%

[a]Reaction condition: quinoxaline-2(1*H*)-one **1a** (0.2 mmol, 0.032 g), Isobutyraldehyde **2a** (0.4 mmol, 0.018 mL), DTBP (1.5 equiv, 0.055 mL), and solvent (2.0 mL), 100℃, 12 h. [b]Isolateed yield. [c]under 80℃. [d]under 120℃. [e]14 h. [f]10 h. TBHP (70% in water). H_2O_2 (30% in water).

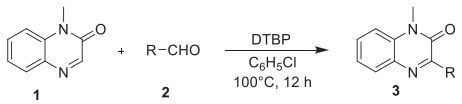
Table 2
Substrate scope with substituted quinoxaline-2(1*H*)-ones.[a,b]

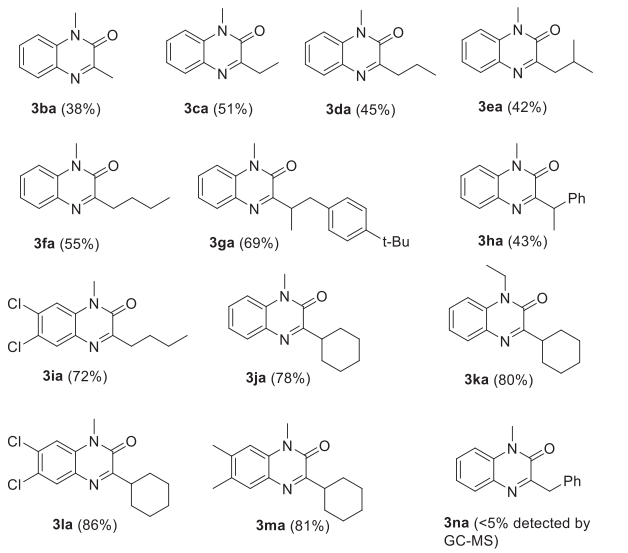




[a]Reaction conditions: quinoxalin-2(1*H*)-ones **1** (0.2mmol), Isobutyraldehyde **2a** (0.4 mmol), DTBP (0.3 mmol) in 2.0 mL C_6H_5Cl solvent at 100°C for 12 h. [b] Isolated yield.

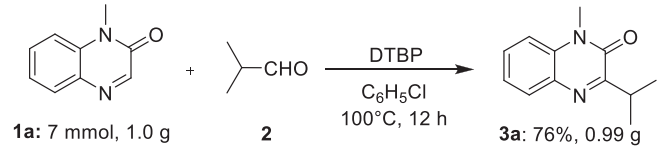
Table 3
Substrated scope with aldehydes.



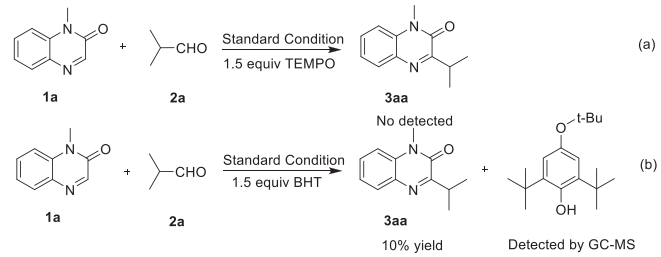


[a] Reaction condition: quinoxalin-2(1*H*)-ones **1** (0.2mmol), Aliphatic aldehydens **2** (0.4 mmol), DTBP (0.3mmol) in 2 mL C_6H_5Cl solvent at 100°C for 12 h. [b]Isolateed yield.

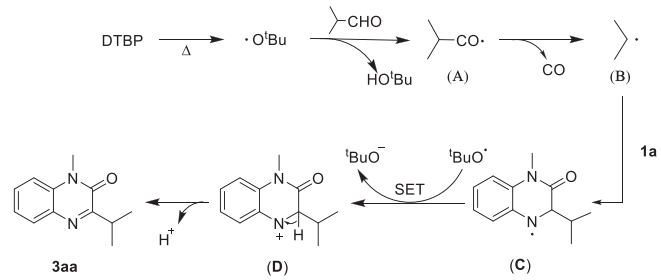
radical could be detected by GC-MS. The results were shown in Scheme 3. In addition, the previous experiment demonstrated that this transformation did not occur without DTBP. These experi-



Scheme 2. Gram-scale reaction.



Scheme 3. Control experiment.



Scheme 4. Proposed reaction mechanism.

ments indicated that a radical mechanism might be involved in the reaction (Scheme 4).

Based on the above results and literature reports [8], a plausible mechanism for the C-3 alkylation of quinoxaline-2(1*H*)-ones was derived. Initially, *Di-tert*-butyl peroxide generates a *tert*-butoxyl radical by heat. Next, *tert*-butoxyl radical abstracts the aldehyde hydrogen atom to give the acyl radical **A**. Then, the acyl radical **A** undergoes decarbonylation to afford alkyl radical **B**. The radical **B** attacks selectively the C-3 position of quinoxaline-2(1*H*)-ones to give the nitrogen radical. Subsequently, a single-electron transfer from **C** would release the intermediate **D**. Finally, the product **3aa** is obtained by the deprotonation of the intermediate **D**.

Conclusions

In summary, In comparison to the previous approaches, this system has good compatibility with quinoxaline-2(1*H*)-ones, regardless of whether the substituents on *N*-heterocycle and benzene ring are electron-withdrawing or electron-donating groups. This protocol will be expectedly used as an alternative approach for derivatization of quinoxaline-2(1*H*)-one motif.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2020.152720.

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