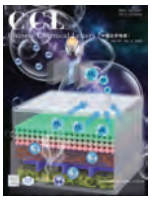
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Decatungstate-photocatalyzed heterogeneous direct benzylation of *N*-heterocycles with benzaldehydes

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ABSTRACT

The first example of heterogeneous direct benzylation of *N*-heterocycles (quinoxalin-2(1*H*)-ones and quinoxalines) with benzaldehydes has been accomplished *via* the sequence of a NaDT-photocatalyzed HAT, a single electron transfer reduction and a proton transfer, a spin-center shift and a back-HAT. Utilizing this strategy, a diverse array of benzylated *N*-heterocycles (29 examples) can be produced with high yields. Importantly, the DT-photocatalyzed direct benzylation with benzaldehydes would be a useful complement to the more extensively studied DT-photocatalyzed acylation and hydroxyalkylation, expanding the scope of DT photocatalysis.

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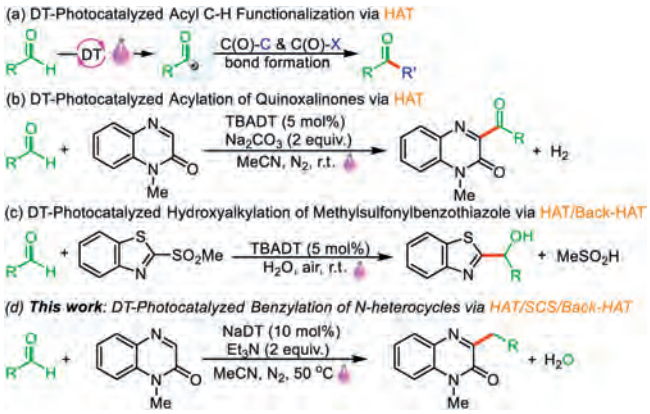
The direct functionalization of C–H bonds in organic molecules is an ideal methodology to produce value-added compounds from easily available feedstocks [1]. Along these lines, hydrogen atom transfer (HAT) as an elementary process in chemical reactions and biological systems, has attracted much attention as an atom- and step-economical strategy to realize catalytic C–H radical functionalization reactions [2,3]. Visible-light photocatalysis [4–13], which harnesses renewable light energy to drive bond-forming reaction under mild reaction conditions, is a versatile synthetic tool in organic synthesis [14–21]. The photocatalytic HAT strategy [22,23], which combines main advantages of the HAT catalysis and visible-light photocatalysis, has emerged as an energy-efficient and eco-friendly platform to achieve C–H functionalization for diverse organic transformations.

Decatungstate anion (DT, $[W_{10}O_{32}]^{4-}$), a type of low cost and readily available metal-oxygen cluster, has been demonstrated to be one of the most efficient and environmentally friendly heterogeneous HAT photocatalyst for promoting the homolytic cleavage of aldehydic C(O)-H bond to realize C(O)-H activation [24,25]. Since the seminal work of Fagnoni in 2007 [26], considerable progress has been made in the conversion of aldehydes into various value-added products *via* DT-photocatalyzed C(O)-C [27–32] and C(O)-X [33–39] bond formations with acyl radical as the key intermediate (Scheme 1a). For example, Yu and co-workers reported the DT-photocatalyzed direct acylation of quinoxalin-2(1*H*)-ones with aldehydes for the synthesis of 3-acylated quinoxalin-2(1*H*)-ones (Scheme 1b) [40]. Despite these achievements, all these reactions are limited to the production of carbonylation products. In 2023, Xu and Deng have made a significant breakthrough in the field of aldehyde conversion (Scheme 1c). They described the construction of 2-hydroxyalkyl benzothiazoles through DT-photocatalyzed direct hydroxyalkylation of 2-methylsulfonyl benzothiazoles with aldehydes, which involves the HAT and back-HAT processes [41].

Benzyl *N*-heterocycles, such as 3-benzyl quinoxalin-2(1*H*)-ones [42–46] and 2-benzylquinoxalines [47], are found in numerous natural products and synthetic pharmaceuticals possessing valuable biological activities [48,49]. In comparison with the DT-photocatalyzed direct acylation and hydroxyalkylation, the DT-photocatalyzed direct benzylation with aromatic aldehydes is scarce and remains a challenge. On the other hand, the spin-center shift (SCS) [50], in which a 1,2-radical shift proceeds with the

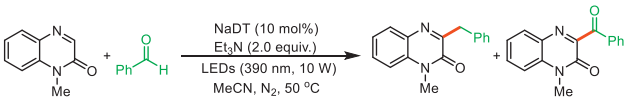
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Scheme 1. DT-photocatalyzed aldehydic C(O)-H functionalization.

Table 1
Optimization of reaction conditions.[a]



1a 2a 3aa 4aa

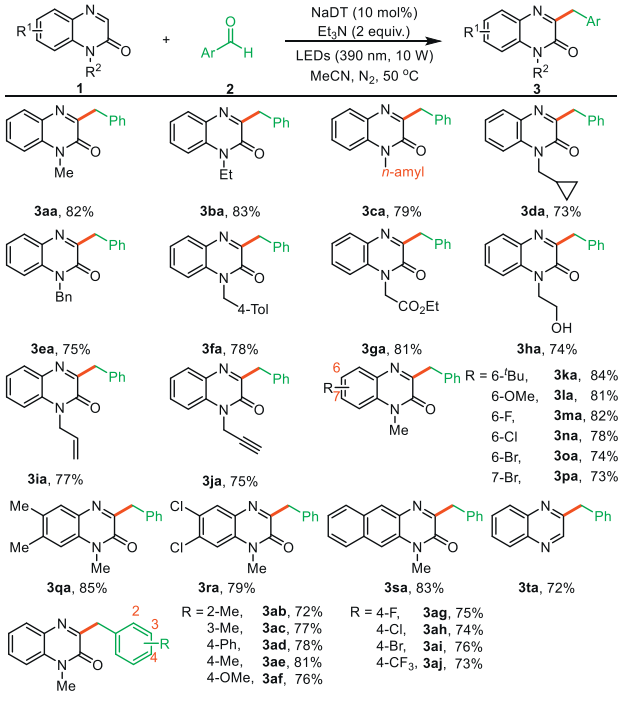
Entry	Variation from the standard conditions	Yield (%)[b]
1	None	84
2	Without NaDT	trace
3	Without light irradiation	N.R.
4	1.5 equiv. of Et_3N was used	73
5	Without Et_3N	trace
6	TBADT, TMADT was used	51, 42
7	Me_3N, DIPEA, $^nPr_3N$ was used	72, 19, trace
8	KH_2PO_3, Na_2CO_3 was used	trace
9	DCE was used	59
10	Acetone, THF, DMF was used	all trace
11	room temperature	38
12	Under open-air atmosphere	N.D.
13	8 W or 12 W LED was used	75, 83

[a] Conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), NaDT (10 mol%), Et_3N (2 equiv.), MeCN (2.5 mL), LED (390 nm, 10 W), N_2, 50 °C, 36 h.
[b] Yield estimated by GC with dodecane as the internal reference.

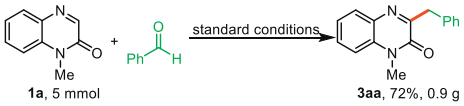
elimination of an oxygen-containing leaving group, has emerged as a powerful strategy for the scission of C–O bond [51–53]. The remarkable breakthrough in such fields was made by MacMillan in 2015, they pioneered the photocatalyzed dehydroxylative alkylation of alcohols *via* a SCS process [54]. We envisioned that if the HAT and the back-HAT could be combined, a tandem aldehydic C(O)-H activation/reduction/dehydroxylation strategy could be realized for the direct benzylation of *N*-heterocycles with benzaldehydes. In continuation of our research on green chemistry [55–64], we herein report the DT-photocatalyzed direct benzylation of *N*-heterocycles with aromatic aldehydes *via* a combination of HAT and SCS. The present strategy not only provides an efficient and sustainable route to benzylated *N*-heterocycles but also offers a complement of classical DT-photocatalyzed acylation, hydroxyalkylation and expands the scope of DT catalysis.

We began our investigations by selecting the benzylation of quinoxalin-2(1*H*)-one (**1a**) with benzaldehyde (**2a**) as a model reaction for optimization (Table 1). After optimizing the reaction parameters, we found that, when **1a** was treated with **2a** (2 equiv.) and NaDT (10 mol%) in MeCN under 10 W LEDs (390 nm) at 50 °C, the benzylation proceeded effectively, thus affording the corresponding product **3aa** in 84% GC yield (Table 1, entry 1). Only a trace amount of **3aa** was detected when the reaction was carried out in the absence of the NaDT (entry 2). Unsurprisingly, no reaction occurred in the absence of light irradiation (entry 3). Decreasing the loading of Et_3N led to a lower yield of **3aa** (entry 4). Without Et_3N but under otherwise optimal conditions, only a trace amount of **3aa** was detected, whereas 3-benzoyl-1-methylquinoxalin-2(1*H*)-one (**4aa**) was formed (entry 5). Conducting the reaction with other DT salt, such as TBADT and TMADT, gave **3aa** in a lower yield (entry 6). Replacing Et_3N with Me_3N, DIPEA or $^nPr_3N$ diminished the reaction yield (entry 7). When inorganic bases were used instead of Et_3N, only a trace amount of **3aa** was observed, indicating that alkylamie might play a key role in this reaction (entry 8). A series of solvents were next investigated, and the replacement of MeCN with DCE gave a decreased yield (entry 9), while only a trace amount of **3aa** was detected when employing acetone, THF or DMF as the solvent (entry 10). Reducing the temperature from 50 °C to room temperature decreased the reaction yield (entry 11), suggesting the reaction temperature had an influence on the reaction. No benzylation reaction was observed when the reaction was performed in air atmosphere, revealing that atmospheric oxygen was not beneficial to this reaction (entry 12). Further efforts in varying the power of LED light did not improve the yield of **3aa** (entry 13).

Following the acquisition of optimized conditions (Table 1, entry 1), we then investigated the scope generality of these photoinduced reactions of *N*-heterocycles **1** with benzaldehydes **2** (Scheme 2). The quinoxalin-2(1*H*)-ones, comprising a range of *N*-substituted methyl, ethyl, *n*-amyl, cyclopropylmethyl, benzyl, 4-methylbenzyl, ester and hydroxyethyl groups, were all compatible to deliver the target products (**3aa**-**3ha**) in good to excellent yields. Substrates containing a C=C double bond or C≡C triple bond, which could capture free radical, participated in this reaction efficiently (**3ia** and **3ja**). In addition, *N*-methylquinoxalin-2(1*H*)-one substrates bearing either an electron-rich groups (-^{t}Bu and -OMe) or electron-deficient substituents (-F, -Cl and -Br) on phenyl ring ran smoothly to deliver the target products (**3ka**-**3pa**) in good yields. 6,7-Disubstituted quinoxalin-2(1*H*)-ones and benzo[*g*]quinoxalin-2(1*H*)-one reacted well with **2a** to afford the corresponding products (**3qa**-**3sa**) in high yields. Furthermore, our protocol was successfully applied to quinoxaline (**3ta**). Next, a se-



Scheme 2. Substrate scope. **1** (0.2 mmol), **2** (0.4 mmol), NaDT (10 mol%), Et_3N (2 equiv.), MeCN (2.5 mL), LED (390 nm, 10 W), N_2, 50 °C.



Scheme 3. Large-scale synthesis of **3aa**.

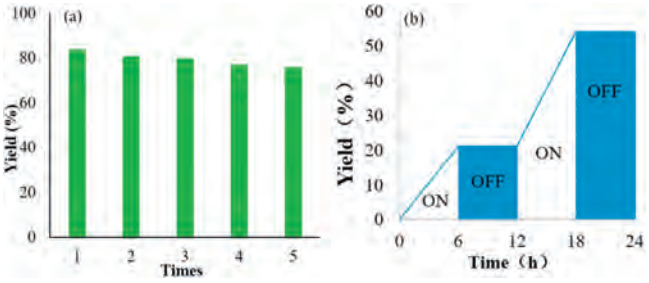


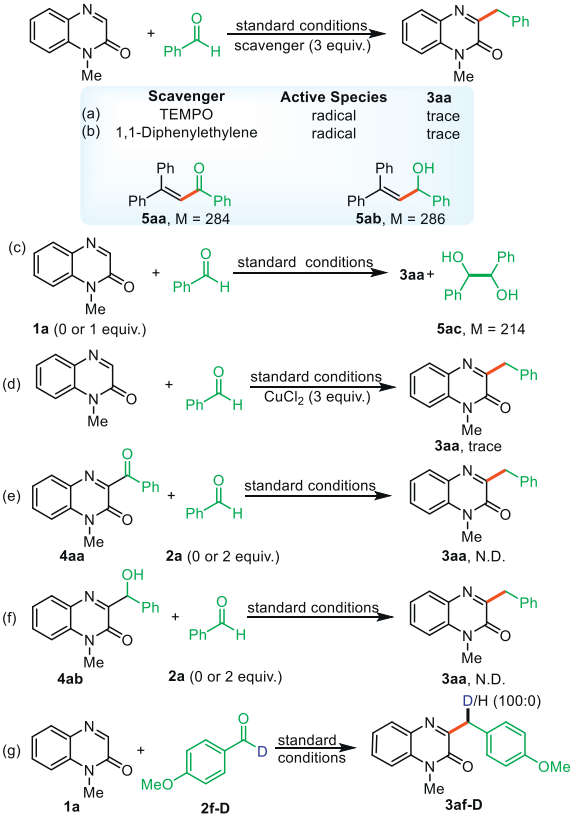
Fig. 1. (a) Reusability of NaDT. (b) Visible-light on/off experiments.

ries of benzaldehydes were investigated. To our delight, no matter whether the phenyl ring of benzaldehyde was modified with either a sterically hindered, electron-rich or electron-deficient group, all of them gave the desired products (**3ab**-**3aj**) in good yields. However, when heteroaromatic aldehydes (4-pyridinecarboxaldehyde or 3-thiophenecarboxaldehyde) were used as the substrates, only a trace amount of the desired product was observed.

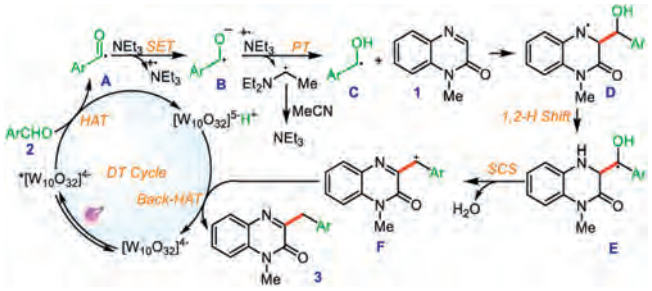
To further depict the practicability of the DT-photocatalyzed aldehyde conversion, both gram-scale synthesis and NaDT recycling tests were presented. With the amplification of **1a** from 0.2 mmol to 5.0 mmol, the benzylation reaction of **1a** produced 0.90 g of **3aa** in 72 % yield without further optimization (Scheme 3). The reusability of NaDT was also investigated using the reaction between **1a** and **2a**. As illustrated in Fig. 1a, the photocatalytic efficiency of NaDT was found to be constantly excellent even after 5 consecutive recycles. Taken together, these results highlighted the synthetic practicability of this DT-photocatalyzed strategy. Considering that this type of deoxygenation processes may involve the H_2O as the by-product, but all efforts failed to detect H_2O.

First, performing the reaction with 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) and 1,1-diphenylethene as a radical scavenger under standard conditions, the formation of **3aa** was suppressed (Schemes 4a and b), and both benzoyl-diphenylethylene adduct **5aa** and ketyl-diphenylethylene adduct **5ab** were detected by MS. When **2a** or the mixture of **1a** and **2a** was subjected to the standard conditions, 1,2-diphenylethane-1,2-diol (**5ac**) was observed (Scheme 4c) [65]. Taken together, these results indicated the involvement of benzoyl and ketyl radical intermediates in this process. The addition of single electron transfer (SET) scavenger $CuCl_2$ to the reaction mixture led to no reaction, revealing a SET process might be involved in this transformation (Scheme 4d). Both **4aa** and 3-(hydroxy(phenyl)methyl)-*N*-methylquinoxalin-2(1*H*)-one (**4ab**) could not be transformed into **3aa** under the standard conditions (Schemes 4e and f), ruling out that they were the key intermediates in this transformation. When deuterated benzaldehyde (**2f-D**) was used as a substrate, the deuterated product **3af-D** was formed (Scheme 4g), suggesting that the benzylic hydrogen atom in product **3** originated from aldehyde, which supported the HAT and back-HAT mechanism. Furthermore, the results of visible light on/off experiments showed that continuous light irradiation was necessary for this reaction (Fig. 1b). Considering that this type of deoxygenation processes may involve the H_2O as the by-product, but all efforts failed to detect H_2O.

Based on the aforementioned experimental results and literature precedents [41,65–68], a tentative mechanism was proposed in Scheme 5. First, the excited-state DT catalyst $^*[W_{10}O_{32}]^{4-}$ would



Scheme 4. Control experiments.



Scheme 5. Proposed reaction mechanism.

abstract a hydrogen atom (HAT) from the aldehyde **2** to produce an acyl radical **A** and $[W_{10}O_{32}]^{5-}H^+$. Next, the produced radical **A** underwent a sequential SET and proton transfer (PT) processes with Et_3N to afford the ketyl radical **B**. The ketyl radical **B** selectively attacked the C=N double bond of quinoxalin-2(1*H*)-one **1** to form a nitrogen-centered radical **D**, which underwent a 1,2-hydrogen shift process to generate a carbon-centered **E**, followed by a SCS process to eliminate H_2O and yield the open-shell benzylic radical **F**. Finally, a back-HAT process between radical **F** and $[W_{10}O_{32}]^{5-}H^+$ delivered the target product **3** and ground-state DT catalyst.

In conclusion, we have developed the first DT-photocatalyzed direct benzylation of *N*-heterocycles (quinoxalin-2(1*H*)-ones and quinoxalines) with benzaldehydes. This novel strategy shows good reaction substrate tolerance, providing efficient and sustainable access to a broad range of benzylated products in a highly concise fashion. In addition, this reaction can be performed on a gram-scale with good efficiency and allowed the NaDT catalyst to be easily recycled. The overall reaction involved a sequence of DT-photocatalyzed production of acyl radical *via* HAT, the reduction of acyl radical into ketyl radical *via* SET and concomitant PT, the

radical addition, the elimination of hydroxyl group *via* SCS and the back-HAT. The deuterium-labeling experimental result manifested that the benzylic hydrogen atom in the product originated from aldehyde, which supported the HAT and back-HAT mechanism. The mild and concise conditions, readily available reactants, wide substrate scope, large-scale synthesis and reusable NaDT catalyst make the present strategy highly attractive in organic and pharmaceutical chemistry. Furthermore, this DT-photocatalyzed direct benzylation would be a useful complement to classical DT-photocatalyzed acylation and hydroxyalkylation, expanding the scope of DT catalysis.

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.

CRediT authorship contribution statement

Yan-Yan Zeng: Methodology. **Jun Jiang:** Supervision. **Yan-Cui Wen:** Methodology. **Chun-Lin Zhuang:** Supervision. **Li-Juan Ou:** Writing – review & editing. **Zi Yang:** Investigation. **Hai-Tao Zhu:** Conceptualization, Formal analysis. **Zu-Li Wang:** Investigation. **Wei-Min He:** Writing – review & editing, Project administration.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.111776.

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