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Communication

Visible-light-promoted oxidative decarboxylation of arylacetic acids in air: Metal-free synthesis of aldehydes and ketones at room temperature

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ABSTRACT

A metal-free photocatalytic oxidative decarboxylation reaction at room temperature was developed for the synthesis of aromatic aldehydes and ketones from the corresponding arylacetic acids. The reaction was realized under blue-light irradiation by adding 1 mol% of 4CzIPN as photocatalyst and air as oxidant. This reaction represents a novel decarboxylation of a sp^3-hybridized carboxylic acids without traditional heating, additional oxidants, and metal reagents under mild conditions.

Due to their easy availability, carboxylic acids have been widely applied as inexpensive starting materials and building blocks for organic synthesis. Decarboxylation reaction is one of the most useful transformations of carboxylic acids [1–5]. Over the past few decades, a myriad of strategies have been developed for decarboxylation of carboxylic acids to synthesize new compounds [6–14]. Among them, transition-metal-catalyzed decarboxylation of sp-hybridized and sp^2-hybridized carboxylic acids has been extensively explored in previous reports [15–17]. However, the decarboxylation of saturated sp^3-hybridized carboxylic acids is still a daunting challenge due to the inert nature of the C—C σ bond [18–20]. Moreover, the requirements of transition-metal catalysts, stoichiometric toxic oxidant, and high temperature in those previous reported systems have limited their applications.

Aromatic aldehydes and ketones are important compounds, displaying a wide range of applications in organic synthesis. One of the most straightforward strategies for the synthesis of aromatic aldehydes and ketones is oxidative decarboxylation of corresponding arylacetic acids [21–25]. For example, Mirkhani and co-workers reported a series of transition-metal-catalyzed procedures (*e.g.*, Mn [26] and Fe [27]) for the oxidative decarboxylation of arylacetic acids in the presence of complex ligands and additional strong oxidants. In 2014, the Song's group developed an elegant Cu(OAc)$_2$-catalyzed aerobic oxidative decarboxylation of phenylacetic acid to synthesize the aldehydes and ketones in sealed tubes under 120 °C (Scheme 1a) [25]. Recently, Bhat and co-workers achieved the same oxidative decarboxylation reaction of arylacetic acid with $K_2S_2O_8$ as oxidant. Notably, the excess amount of $K_2S_2O_8$ (2 equiv.) and high temperature (90 °C) were required (Scheme 1b) [21]. Although remarkable advances have been made, previous methods required stoichiometric oxidants and high temperature, which may limit their applications. From the point view of green chemistry, the exploration of a metal-free catalytic system with green oxidants (ideally atmosphere pressure of oxygen or air) under mild conditions is highly desired [28–30].

As a rapidly developing field, visible-light-promoted reactions have drawn huge attention in recent years [31–51]. Particularly, 1,2,3,5-tetrakis(carbazol-9-yl)-4,6-dicyanobenzene (4CzIPN) has proven to be an efficient metal-free photocatalyst for the transformations of carboxylic acids in organic synthesis, medicine and materials [52–57]. In the course of our continuing efforts on green chemistry [58–61], we herein disclosed a 4CzIPN-catalyzed transition-metal-free procedure to achieve the oxidative

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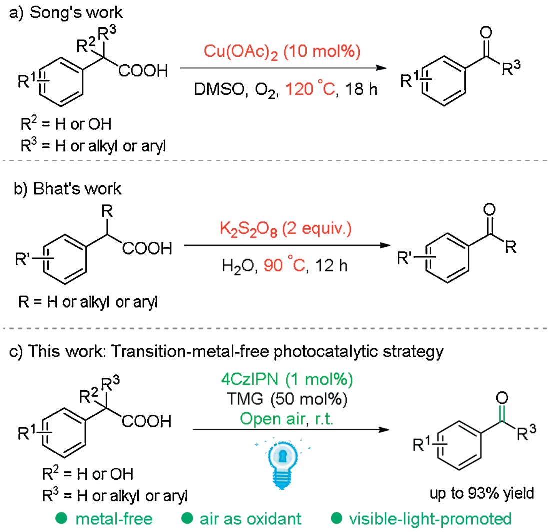
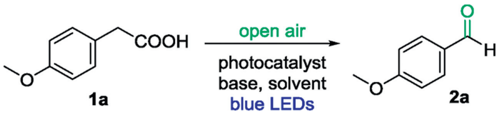


Scheme 1. Oxidative decarboxylation of phenylacetic acids.

decarboxylation of arylacetic acids toward the corresponding aromatic aldehydes and ketones at room temperature under visible-light illumination (Scheme 1c). Importantly, in this procedure air is the sole oxidant, which eliminate the need for large quantities of oxidants. This strategy circumvents the use of transition-metal catalysts and traditional heating.

We started our study using 4-methoxyphenylacetic acid (**1a**) as the model substrate in the presence of 4CzIPN and Cs_2CO_3 under visible-light (Table 1). To our delight, the reaction with 4CzIPN (5 mol%) as photocatalyst and Cs_2CO_3 (50 mol%) as additive in air under the irradiation of blue light gave the desired aldehyde **2a** in 66% yield (entry 1). While the same reaction under nitrogen atmosphere only delivered the product **2a** in 9% yield (entry 2). Such a finding suggested that the oxidative decarboxylation of **1a** with 4CzIPN as photocatalyst and air as the sole oxidant is possible. Subsequently, various solvents including DMSO, CH_3CN, THF, H_2O, 1,4-dioxane, toluene, acetone and PEG_{400} were surveyed in the presence of 4CzIPN/Cs_2CO_3 (entries 3–10). It was found that the reaction in CH_3CN gave a higher yield of **2a** up to 75%, indicating CH_3CN is the optimal solvent for this transformation (entry 4). Next, the effects of different bases on the reaction were investigated. The inorganic bases like K_2CO_3, K_3PO_4, KOH, and the organic bases such as DABCO (1,4-diazabicyclo[2.2.2]octane), DBU (1,8-diazabicyclo[5.4.0]undec-7-ene), TMEDA (tetramethylethylenediamine), Et_3N, DMAP (dimethylaminopyridine), TMG (1,1,3,3-tetramethylguanidine), 2,6-lutidine, and TBD (1,5,7-triazabicyclo[4.4.0]dec-5-ene) were screened respectively with 4CzIPN (5 mol%) as photocatalyst in CH_3CN under blue light irradiation (entries 11–21). The results indicated that TMG was superior to the others and the yield was as high as 91% (entry 19). Then, some organic photocatalysts such as Eosin B, Eosin Y, Rose Bengal were applied as catalysts in the presence of TMG (50 mol%) in CH_3CN for 6 h under air at room temperature, respectively (entries 22–24). However, the oxidative decarboxylation reactions almost did not work. Furthermore, it is delighted to find that even

Table 1
Optimization of reaction conditions.[a]



Entry	Photocatalyst	Solvent	Base	Yield (%)[b]
1	4CzIPN	DMF	Cs_2CO_3	66
2[c]	4CzIPN	DMF	Cs_2CO_3	9
3	4CzIPN	DMSO	Cs_2CO_3	47
4	4CzIPN	CH_3CN	Cs_2CO_3	75
5	4CzIPN	THF	Cs_2CO_3	28
6	4CzIPN	H_2O	Cs_2CO_3	6
7	4CzIPN	1,4-Dioxane	Cs_2CO_3	34
8	4CzIPN	Toluene	Cs_2CO_3	trace
9	4CzIPN	Acetone	Cs_2CO_3	54
10	4CzIPN	PEG_{400}	Cs_2CO_3	15
11	4CzIPN	CH_3CN	K_2CO_3	69
12	4CzIPN	CH_3CN	K_3PO_4	70
13	4CzIPN	CH_3CN	KOH	70
14	4CzIPN	CH_3CN	DABCO	23
15	4CzIPN	CH_3CN	DBU	77
16	4CzIPN	CH_3CN	TMEDA	30
17	4CzIPN	CH_3CN	Et_3N	31
18	4CzIPN	CH_3CN	DMAP	56
19	4CzIPN	CH_3CN	TMG	91
20	4CzIPN	CH_3CN	2,6-Lutidine	18
21	4CzIPN	CH_3CN	TBD	58
22	Eosin B	CH_3CN	TMG	trace
23	Eosin Y	CH_3CN	TMG	trace
24	Rose Bengal	CH_3CN	TMG	trace
25[d]	4CzIPN	CH_3CN	TMG	98 (92)
26	–	CH_3CN	TMG	trace

[a] Reaction conditions: *p*-Methoxyphenylacetic acid (0.2 mmol), 4CzIPN (5 mol%), base (50 mol%), in CH_3CN (1.5 mL) under air at room temperature for 6 h with the irradiation of 25 W blue LEDs.

[b] Yields were determined by ^1H NMR using 1,1,2,2-tetrachloroethane as the internal standard (isolated yield in parentheses).

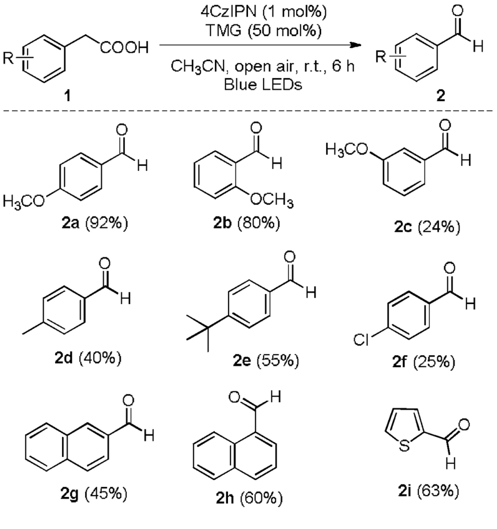
[c] Reaction performed under a nitrogen atmosphere.

[d] 1 mol% of 4CzIPN as photocatalyst.

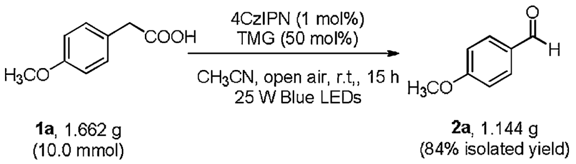
the catalyst loading of 4CzIPN was reduced to 1 mol%, the reaction performance was almost maintained giving the product **2a** in 92% of isolated yield (entry 25). Additionally, the control experiment without any photocatalyst only gave trace amount of product **2a** (entry 26). These results suggested the application of 4CzIPN as a photocatalyst and the irradiation of blue light were critical for such an efficient transformation. Finally, the optimal conditions were established as follows: **1a** (0.2 mmol), 4CzIPN (1 mol%), TMG (50 mol%), CH_3CN (1.5 mL) under air at room temperature for 6 h with the irradiation of 25 W blue LEDs.

With the optimum conditions in hand, we further explored the scope of this visible-light-induced 4CzIPN-catalyzed oxidative decarboxylation reaction *via* the reaction of some commercially available arylacetic acids **1** (Scheme 2). When the phenylacetic acids bearing methoxy group at *ortho*- or *para*-positions, 92% and 80% yields of corresponding products **2a** or **2b** were obtained, respectively. However, the phenylacetic acid with *meta*-substituted methoxy group gave relatively poor yield of the aldehydes **2c**. These results suggested that the position of the substituents on the aromatic rings had a significant effect on the reactivity. When the phenylacetic acids with methyl or *tert*-butyl group at *para*-position, the oxidative decarboxylation reaction also showed good reactivities, giving the corresponding aldehydes **2d** and **2e** in moderate yields (40% and 55%). Unfortunately, only 25% yield of **2f** was observed when the -Cl containing phenylacetic acid was employed as substrate. In the cases of **2c** and **2f**, no obvious improvements were observed even with higher catalyst loading (2 mol%) and longer reaction time (12 h). Notably, no byproducts were observed in those cases, suggesting that the low yields were probably due to the low reactivity of those substrates. Interestingly, 1-naphthylacetic acid and 2-naphthylacetic acid were also applicable in this procedure, affording the desired aldehydes **2g** and **2 h** in 45% and 60% yields. Moreover, the heteroaromatic acid such as 2-(thiophen-2-yl)acetic acid was also suitable in this reaction delivering the thiophene-2-carbaldehyde **2i** in 63% yield under standard conditions.

Additionally, a scale-up reaction (10 mmol-scale) for the model reaction was conducted under the identical conditions. Delightfully, a great yield (84%) of the desired product **2a** was isolated after prolonging the reaction time to 15 h, demonstrating this simple photocatalytic system is practical and potentially useful (Scheme 3).



Scheme 2. Oxidative decarboxylation of arylacetic acids to aldehydes. Reaction conditions: Arylacetic acid (0.2 mmol), 4CzIPN (1 mol%), TMG (50 mol%), in CH_3CN (1.5 mL) in air at room temperature for 6 h with the irradiation of 25 W blue LEDs. Isolated yields were given.



Scheme 3. The gram-scale synthesis of **2a.**

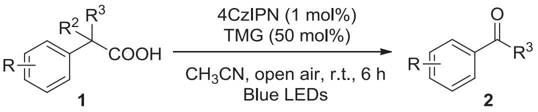
In order to expand the scope of this reaction under standard conditions, we then explored the reaction with α-substituted aromatic acetic acids as substates under optimal reaction conditions (Table 2). To our delight, when α-hydroxy-*p*-methoxyphenylacetic acid **1j** was employed as reactant, the corresponding aldehyde **2j** was obtained in excellent yield of 93%. Next, we used 2-hydroxy-2,2-diphenylacetic acid **1k** and 2-cyclohexyl-2-hydroxy-2-phenylacetic acid **1l** as substrates, the corresponding products, benzophenone **2k** and cyclohexyl(phenyl)methanone **2l** were obtained in good yields of 70% and 82%, respectively. On this basis, a series of ketones (**2m**-**2q**) were synthesized from the α-substituted phenyl acetic acids (**1m**-**1q**) under the standard reaction conditions in moderate to good yields (51%–78%). Moreover, the aliphatic substrate, *e.g.*, cyclohexanecarboxylic acid did not give any product, indicating that aliphatic carboxylic acids are not suitable in this protocol.

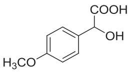
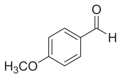
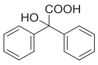
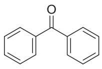
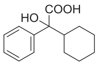
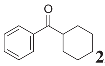
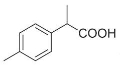
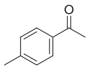
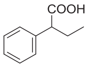
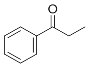
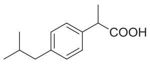
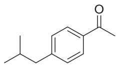
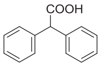
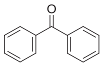
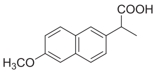
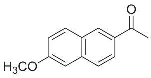
To gain a deeper insight into the mechanism of this photocatalysis reaction, the control experiments were conducted as described. When the model reaction was treated with 5 equiv. of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO), the reaction was totally suppressed and no product **2a** was detected (Scheme 4). This result revealed that a radical pathway was probably involved in the reaction process.

Furthermore, the Stern-Volmer quenching experiments was carried out by separately adding various components of the reaction system to the degassed anhydrous CH_3CN solution containing 4CzIPN. However, none of the reactants had obvious fluorescence quenching effect on the photocatalyst (Supporting information). Considering that it is an aerobic reaction, the experiment of oxygen quenching was carried out later. As expected, molecular oxygen showed a signifcant fluorescence quenching effect on the photocatalyst. The linear relationship between I_0/I (I_0 and I are the fluorescence intensities before and after the O_2 was bubbled into the solution, respectively) and the time for oxygen bubbling indicates that O_2 may quench 4CzIPN (Supporting information). When the superoxide radical ($O_2^{\bullet -}$) scavenger 4-benzoquinone (BQ) was added to the model reaction [62], the yield of **2a** was reduced to 18%, which suggesting the generation of superoxide radical ($O_2^{\bullet -}$) in the oxidative decarboxylation reaction, which was further confirmed by EPR spectroscopy [63] (Supporting Information).

Based on the above results and previous reports [64], a plausible reaction mechanism was proposed as shown in Scheme 5. Under blue-light irradiation, 4CzIPN was excited to produce the corresponding excited-state 4CzIPN*, which is sufficiently reductive ($E_{1/2}(P^{\bullet +}/P^*) = -1.04$ V *vs.* SCE [52]) to donate an electron to O_2 generating the superoxide radical $O_2^{\bullet -}$ ($E_{red} = -0.86$ V *vs.* SCE [65]) along with the release highly oxidative 4CzIPN$^{\bullet +}$. In the presence of base, carboxylic acid **1** was deprotonated to give the carboxylate anion **A**. The highly oxidative 4CzIPN$^{\bullet +}$ ($E_{ox}(P^{\bullet +}/P) = +1.52$ V *vs.* SCE [52]) was able to oxidize the carboxylate anion **A** ($E_{ox} = +1 \sim +1.25$ V *vs.* SCE [66]) producing the radical **B** and regenerating the photocatalyst 4CzIPN. Subsequently, the elimination of CO_2 from radical **B** gave the benzylic radical **C**, which then combined with superoxide radical ($O_2^{\bullet -}$) to give the anion **D**. Finally, the anion **D** was protonated to deliver the

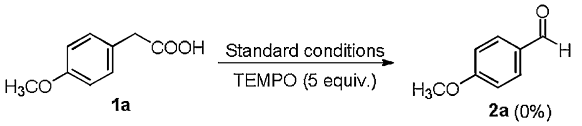
Table 2
Oxidative decarboxylation of α-substituted aromatic acetic acid to aldehydes and ketones.[a]



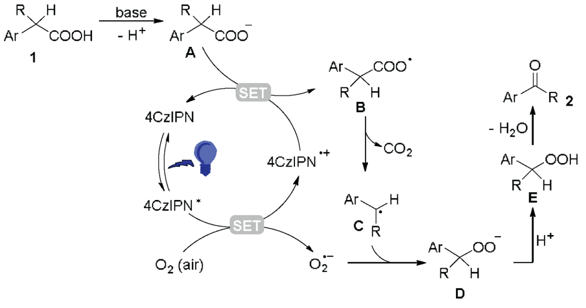
Entry	Substrate	Product	Yield (%)[b]
1	 **1j**	 **2j**	93
2	 **1k**	 **2k**	70
3	 **1l**	 **2l**	82
4	 **1m**	 **2m**	64
5	 **1n**	 **2n**	56
6	 **1o**	 **2o**	51
7	 **1p**	 **2p**	78
8	 **1q**	 **2q**	72

[a] Reaction conditions: α-Substituted aromatic acetic acid (0.2 mmol), 4CzIPN (1 mol%), TMG (50 mol%), in CH_3CN (1.5 mL) in air at room temperature for 6 h with the irradiation of 25 W blue LEDs.

[b] Isolated yields were given.



Scheme 4. Control experiments.



Scheme 5. The Proposed mechanism.

intermediate **E**, followed by dehydration to produce the desired product **2**.

In conclusion, we have developed a photocatalytic protocol for the oxidative decarboxylation of phenylacetic acid derivatives to synthesize corresponding aromatic aldehydes or ketones. This reaction can be carried out in air at room temperature by using 1 mol% of 4CzIPN as photocatalyst. This transition-metal-free procedure avoids the use of excess amount of inorganic/organic oxidants and high temperature. The employment of air as green oxidant eliminate the generation of waste from traditional oxidants. Further research for the application of such a oxidative carboxylation strategy is underway in our research group.

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 material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.cclet.2019.12.031.

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