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Communication

Photoinduced reaction of potassium alkyltrifluoroborates, sulfur dioxide and *para*-quinone methides *via* radical 1,6-addition

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ABSTRACT

A photoinduced reaction of potassium alkyltrifluoroborates, sulfur dioxide, and *para*-quinone methides under visible light irradiation at room temperature is developed, giving rise to diarylmethyl alkylsulfones in moderate to good yields. This reaction works well under photocatalysis with a broad substrate scope by using DABCO·$(SO_2)_2$ as the source of sulfur dioxide. Mechanistic study shows that this transformation is initiated by alkyl radicals generated *in situ* from potassium alkyltrifluoroborates in the presence of photocatalyst. The subsequent insertion of sulfur dioxide and radical 1,6-addition of para-quinone methides with alkylsulfonyl radical intermediates afford the corresponding diarylmethyl alkylsulfones.

The introduction of sulfonyl motifs into organic molecules has attracted continuous interest in medicinal chemistry, due to the dramatic improvement in biologically active compounds and pharmaceutical agents [1]. Therefore, enormous efforts have been made for the preparation of diverse sulfonyl compounds [2]. Among them, diarylmethyl sulfones have attracted significant focus recently, and are often used as building blocks in synthetic chemistry [3]. They are valuable synthetic intermediates for the construction of triarylmethane derivatives, which are ubiquitous in the area of medicinal chemistry and materials science [4]. However, only several classical strategies for the preparation of diarylmethyl sulfones have been developed, including nucleophilic addition of aromatic aldehydes [5], nucleophilic substitution of alcohols [6], transition metal-catalyzed arylation of methyl sulfones [7] and nucleophilic 1,6-addition of *para*-quinone methides (*p*-QMs) [8]. We noticed that previous works described above were initiated by sulfinyl anion starting from sodium arylsulfinates or arylsulfonyl hydrazines. Meanwhile, *p*-QMs as versatile Michael acceptors are structurally characterized by the unique assembly of carbonyl and olefinic moieties, which have been applied in the synthesis of natural products and bioactive agents [9]. Compared to the reports on nucleophilic 1,6-addition of *p*-QMs [10], radical 1,6-addition of *p*-QMs is much less, which also provides an important and alternative route for the generation of valuable entities [11]. Despite these significant advances, development of more straightforward methods as well as environmentally friendly conditions to construct diverse diarylmethyl sulfones is still highly desirable.

Using sulfur dioxide surrogates of DABCO·$(SO_2)_2$ or potassium/sodium metabisulfite as the source of sulfur dioxide has become a facile pathway for the preparation of sulfonyl compounds [12,13]. Recently, we accomplished the generation of sulfonyl compounds through radical insertion of sulfur dioxide. Several radical precursors including aryldiazonium tetrafluoroborates [14], potassium alkyltrifluoroborates [15], Katritzky salts [16] and 4-substituted Hantzsch esters [17] have been utilized in the sufonylation process. During the reaction process, sulfonyl radicals formed *in situ* would be the key intermediates. So far, 1,2-addition of sulfonyl radicals with the difunctionalization of olefins has been realized [18]. Prompted by the recent advance of 1,6-addition of *p*-QMs and the radical chemistry of sulfur dioxide, we envisioned that radical 1,6-addition of *p*-QMs with the insertion of sulfur dioxide would be feasible as well, which would give rise to diarylmethyl sulfones. Herein, we report a photoinduced reaction of potassium alkyltrifluoroborates, sulfur dioxide, and *p*-QMs under visible light irradiation at room temperature, leading to diarylmethyl alkylsulfones in moderate to good

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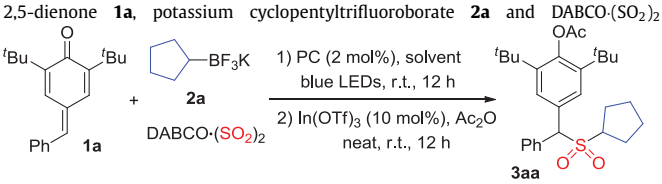
yields. This reaction works well under photocatalysis with a broad substrate scope by using DABCO·$(SO_2)_2$ as the source of sulfur dioxide. Various functional groups are compatible. Mechanistic study shows that this transformation is initiated by an alkyl radical generated in situ from potassium alkyltrifluoroborate in the presence of photocatalyst. The subsequent insertion of sulfur dioxide and radical 1,6-addition of *p*-QMs with alkylsulfonyl radical intermediate afford the corresponding diarylmethyl alkylsulfone.

Guided by the hypothesis as mentioned above, we therefore started to investigate the feasibility of this proposed route. Initial studies were performed for the reaction of 4-benzylidene-2,6-di–*tert*-butylcyclohexa-2,5-dienone **1a**, potassium cyclopentyltrifluoroborate **2a** and DABCO·$(SO_2)_2$. At the beginning, the reaction was carried out in 1,2-dichloroethane (DCE) in the presence of 30 W blue LEDs (465–475 nm) at room temperature. Although we discovered that the starting materials were consumed and the phenol-type diarylmethyl sulfone was produced, the product could not be purified and isolated since the elimination of sulfinic acid might be reversible [19]. Thus, a late-modification by indium(III) triflate catalyzed acylation was introduced [20], and the optimization of reaction conditions for the radical 1,6-addition was provided in Table 1. A brief screening of photocatalysts revealed that 9-mesityl-10-methyl acridinium perchlorate [Mes-Acr]ClO_4 was the best one, and the corresponding diarylmethyl alkylsulfone **3aa** was obtained as expected in 52% yield (Table 1, entry 1). Inferiors results were observed when other photocatalysts were used in the reaction (Table 1, entries 2–6). We further explored the reaction in different solvents, and DCE was found to be the best choice (Table 1, entries 7–13). Next, evaluation of additives was carried out, showing that the presence of 0.5 equiv. of KH_2PO_4 gave rise to compound **3aa** in 86% yield (Table 1, entry 14. For details, see Tables S1 in Supplementary information). Considering the easy availability, sodium metabisulfite or potassium metabisulfite was used as the source of sulfur dioxide. However, no product was detected (Table 1, entry 15). Additionally, no reaction occurred when the reaction was performed in dark (Table 1, entry 16).

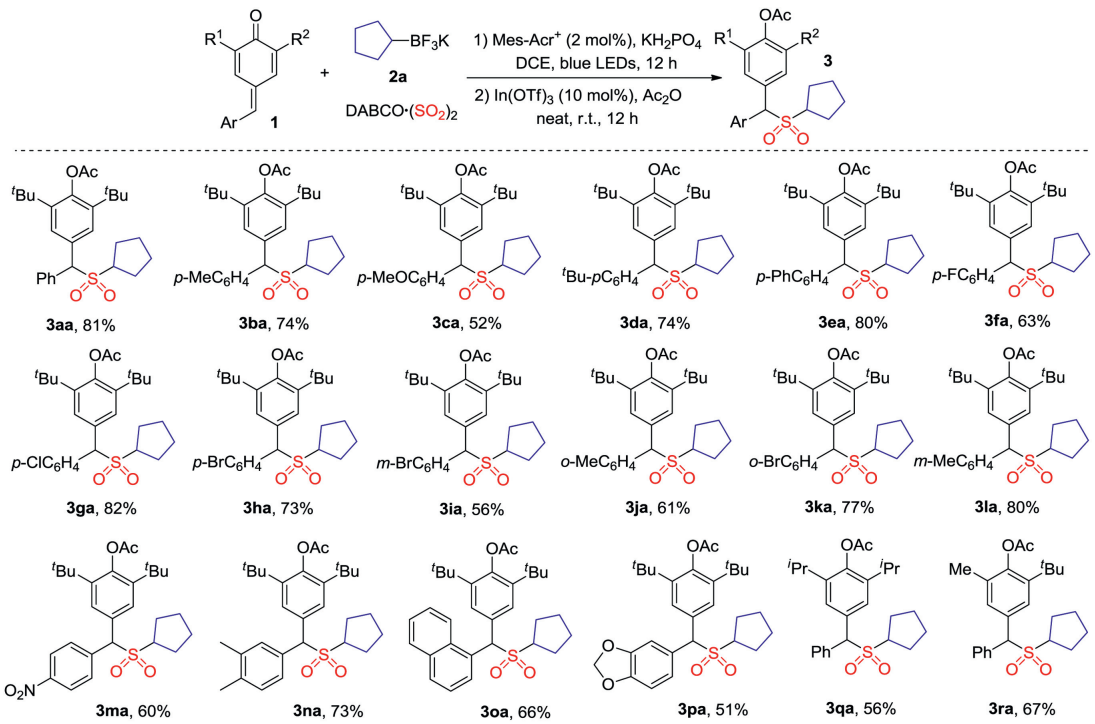
Table 1
Initial studies for the reaction of 4-benzylidene-2,6-di–*tert*-butylcyclohexa-2,5-dienone **1a**, potassium cyclopentyltrifluoroborate **2a** and DABCO·$(SO_2)_2$.[a]



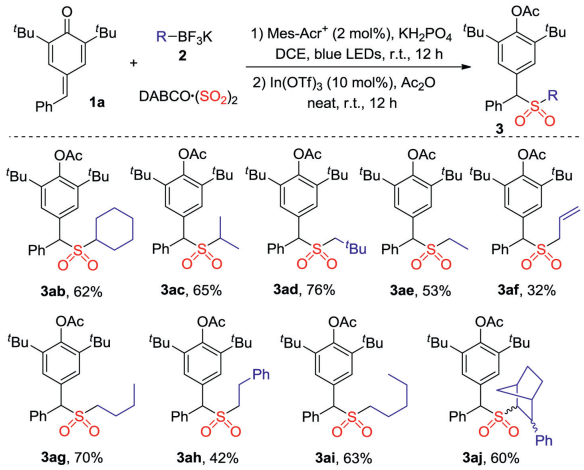
Entry	PC	Solvent	Yield (%) [b]
1	Mes-Acr$^+$	DCE	52
2	Eosin Y	DCE	trace
3	$Ru(bpy)_3Cl_2$	DCE	n.d
4	$Ir(ppy)_3$	DCE	n.d
5	Rhodamine 6G	DCE	trace
6	4-CzIPN	DCE	trace
7	Mes-Acr$^+$	MeCN	51
8	Mes-Acr$^+$	DMF	40
9	Mes-Acr$^+$	DMSO	trace
10	Mes-Acr$^+$	1,4-Dioxane	48
11	Mes-Acr$^+$	Toluene	46
12	Mes-Acr$^+$	MeOH	18
13	Mes-Acr$^+$	THF	50
14 [c]	Mes-Acr$^+$	DCE	86 (81)
15 [d]	Mes-Acr$^+$	DCE	n.d
16 [e]	Mes-Acr$^+$	DCE	n.r

n.d = no desired product; n.r = no reaction.
[a] Reaction conditions: 4-benzylidene-2,6-di–*tert*-butylcyclohexa-2,5-dienone **1a** (0.1 mmol), potassium cyclopentyltrifluoroborate **2a** (0.15 mmol), DABCO·$(SO_2)_2$ (0.1 mmol), photocatalyst (2 mol%), solvent (1.0 mL), N_2, blue LEDs (30 W, 465–475 nm), r.t. for 12 h; then workup without purification, $In(OTf)_3$ (10 mol%) and Ac_2O (0.2 mL), neat, r.t. for 12 h.
[b] ^1H NMR yield using 1,3,5-trimethoxybenzene as an internal standard (isolated yield in parentheses).
[c] In the presence of compound **2a** (0.12 mmol) and KH_2PO_4 (0.05 mmol).
[d] $Na_2S_2O_5$ or $K_2S_2O_5$ was used instead of DABCO·$(SO_2)_2$.
[e] In dark.

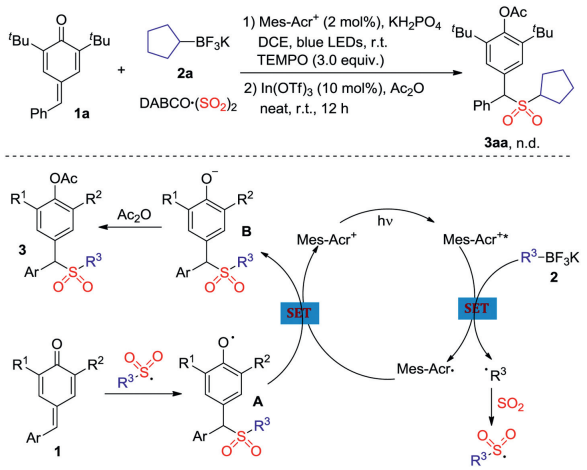
After obtaining the optimized conditions, we then evaluated the substrate scope of this radical 1,6-addition of *p*-QMs. As illustrated in Scheme 1, a variety of substituted *para*-quinone methides



Scheme 1. Reaction of substituted *para*-quinone methides **1**, potassium cyclopentyltrifluoroborate **2a** and DABCO·$(SO_2)_2$. Isolated yield based on *para*-quinone methides **1**.



Scheme 2. Reaction of *para*-quinone methide **1a**, potassium alkyltrifluoroborate **2** and DABCO·$(SO_2)_2$. Isolated yield based on *para*-quinone methide **1a**.



Scheme 3. Control experiment and plausible mechanism.

1 could react smoothly with potassium cyclopentyltrifluoroborate **2a** and DABCO·$(SO_2)_2$ under the standard conditions, providing the desired diarylmethyl alkylsulfones in moderate to excellent yields.

From Scheme 1, it was found that *para*-quinone methides bearing electron-donating or electron-withdrawing groups located at the *para*-position of aromatic ring exhibited good reaction efficiency and diverse functional groups including methyl, methoxyl, *tert*–butyl, phenyl, fluoro, chloro, bromo and nitro were well tolerated under the optimal conditions, leading to the desired products **3ba-3 ha** and **3ma** in moderate to good yields (52%–82% yields). Additionally, the efficiency of this protocol was not impeded by the *ortho*- or *meta*-substituents on the aromatic ring, which gave rise to the corresponding products **3ia-3la** in 56%–80% yields. Interestingly, muti-substituted *para*-quinone methides **1n-1p** could also take part in this transformation. Notably, the *para*-quinone methides **1q** and **1r** could react with compound **2a** smoothly, affording the desired products **3qa** and **3ra** in 56% and 67% yield, respectively.

In order to further verify the applicability of the strategy, a range of potassium alkyltrifluoroborates **2** were examined in the reaction of 4-benzylidene-2,6-di–*tert*-butylcyclohexa-2,5-dienone **1a** and DABCO·$(SO_2)_2$ (Scheme 2). As expected, all reactions worked well, affording the corresponding diarylmethyl alkylsulfones in moderate to good yields. The alkyl groups including cyclohexyl, isopropyl, neopentyl, *n*-pentyl, *n*–butyl and ethyl groups were compatible under the conditions. Compound **3aj** with diastereoisomers was furnished in 60% yield. In this transformation, the substrate scope was limited since only *para*-quinone methides as 1,6-acceptor with 2,6-dialkyl groups could be employed. It was reported that the presence of 2,6-dialkyl groups could not be avoided for the successful conversion, as they provided stability to *para*-quinone methides [21]. Additionally, the bulky *tert*–butyl or isopropyl groups could block the C-4 position of *para*-quinone methides, thus directing the *para*-quinone methides to undergo 1,6-conjugate addition over 1,4-conjugate addition in a highly regioselective manner.

Since a radical 1,6-addition was hypothesized, a control experiment was subsequently performed with the addition of 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) as a radical scavenge under the standard conditions (Scheme 3). As expected, the reaction was completely hampered, and no desired product **3aa** was detected. Thus, combined with our previous work and related reports [12], a plausible mechanism was proposed (Scheme 3). We reasoned that under visible light irradiation, the photocatalyst Mes-Acr$^+$ would convert to the excited state of Mes-Acr^{+*}, which would promote the transformation from potassium alkyltrifluoroborate **2** to alkyl radical intermediate and the reduced species Mes-Acr$^{\cdot}$. Subsequently, alkyl radical would be trapped by sulfur dioxide, leading to the formation of alkylsulfonyl radical. Then, radical 1,6-addition process would occur between alkylsulfonyl radical and *para*-quinone methides **1** giving rise to radical intermediate **A**. Radical intermediate **A** would be reduced by Mes-Acr$^{\cdot}$ to provide anionic intermediate **B** along with the regeneration of photocatalyst Mes-Acr$^+$. Further acylation would produce the corresponding diarylmethyl alkylsulfone **3**.

In conclusion, we have described a photoinduced reaction of potassium alkyltrifluoroborates, sulfur dioxide, and *para*-quinone methides under visible light irradiation at room temperature. Diverse diarylmethyl alkylsulfones are produced in moderate to good yields. This reaction works well under photocatalysis with a broad substrate scope by using DABCO·$(SO_2)_2$ as the source of sulfur dioxide. Mechanistic study shows that this transformation is initiated by alkyl radicals generated *in situ* from potassium alkyltrifluoroborates in the presence of photocatalyst. The subsequent insertion of sulfur dioxide and radical 1,6-addition of *para*-quinone methides with alkylsulfonyl radical intermediates afford the corresponding diarylmethyl alkylsulfones.

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.

Acknowledgments

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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.2021.05.007.

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