Photoredox-catalyzed sulfonylation of alkyl iodides, sulfur dioxide, and electron-deficient alkenes†

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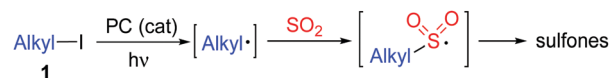
A photoredox-catalyzed sulfonylation of alkyl iodides, sulfur dioxide, and electron-deficient alkenes under mild conditions is achieved. This reaction proceeds through alkyl radicals formed *in situ* from alkyl iodides under visible light irradiation in the presence of a photoredox catalyst. The alkyl radical intermediates would react with sulfur dioxide leading to alkylsulfonyl radicals, which would be trapped by electron-deficient alkenes giving rise to alkyl sulfones. Various functional groups including nitro, halo, acetyl, sufonyl, and pyridinyl are all tolerated under the photoredox conditions.

Transition metal-catalyzed coupling reactions of aryl/alkyl halides have been utilized broadly, due to aryl/alkyl halides being easily available and cheap.[1] Recently, reactions of aryl/alky halides under photoredox catalysis have attracted much attention. It is well recognized that the photoinduced C–X bond dissociation of aryl/alkyl halides would produce the corresponding carbon radicals *via* electron transfer.[2] Therefore, the possibility of the β-H elimination of alkyl halides under transition metal catalysis may be avoided under photoinduced conditions. So far, progress of photoinduced C–X bond dissociation has been witnessed. For example, Peters and Fu reported photoinduced Ullmann C–N coupling by using a stoichiometric or catalytic amount of copper salt under visible light irradiation at room temperature.[2a]

In the past decade, synthesis of sulfonyl compounds through the insertion of sulfur dioxide has developed rapidly,[3] which avoids the utilization of pre-installed sulfonyl precursors.[4] Currently, the sulfur dioxide surrogates including

DABCO·(SO$_2$)$_2$ (1,4-diazabicyclo[2.2.2]octane-sulfur dioxide)[5–7] and inorganic sulfites[8] have been used broadly in the sulfonylation process. As part of a program for the generation of sulfonyl compounds, we are interested in the radical process with the insertion of sulfur dioxide.[7] So far, various radical precursors have been employed in the sulfonylation reaction including aryldiazonium tetrafluoroborates,[7] aryl/alkyl halides,[9] diaryliodonium salts,[10] and potassium alkyltrifluoroborates.[11] Among these precursors, aryl/alkyl halides are especially attractive, as they are easily available and cheap. For instance, aryl/alkyl halides could react with sulfur dioxide and hydrazines under ultraviolet irradiation.[8a] Although this transformation was efficient, the reaction could not be extended to other partners due to the ultraviolet irradiation. Thus, method development for the sulfonylation of aryl/alkyl halides with the insertion of sulfur dioxide under mild conditions, especially in the presence of visible light, would be highly desirable. Herein, we report a photoredox-catalyzed sulfonylation of alkyl iodides, sulfur dioxide, and electron-deficient alkenes under mild conditions. This reaction proceeds through alkyl radicals formed *in situ* from alkyl iodides under visible light irradiation in the presence of a photoredox catalyst, leading to diverse sulfones. Various functional groups including nitro, halo, acetyl, sufonyl, and pyridinyl are all tolerated under the photoredox conditions (Scheme 1).

At the outset, a model reaction of cyclohexyl iodide **1a**, DABCO·(SO$_2$)$_2$, and (*E*)-chalcone **2a** in the presence of TTMS and photocatalyst (5 mol%) was explored. The results are summarized in Table 1. Initially, the reaction was catalyzed by [Ir(dfCF$_3$ppy)$_2$(dtbbpy)]PF$_6$ in 1,2-dichloroethane irradiated by a 15 W blue LED (Table 1, entry 1). To our delight, the corresponding sulfone **3a** was obtained in 31% yield. Inferior results

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Scheme 1 Generation of sulfones under photoredox catalysis with the insertion of sulfur dioxide.

Table 1 Initial studies for the reaction of cyclohexyl iodide **1a**, DABCO·(SO$_2$)$_2$, and (E)-chalcone **2a**[a]

| Entry	PC	Additive	Base	Solvent	Yield[b] (%)
1	[Ir][c]	—	—	DCE	31
2	[Ir][c]	—	—	Dioxane	23
3	[Ir][c]	—	—	MeOH	Trace
4	[Ir][c]	—	—	MeCN	18
5	Rhodamine B	—	—	DCE	0
6	Mes-Acr$^+$	—	—	DCE	35
7	Mes-Acr$^+$	—	K$_2$CO$_3$	DCE	22
8	Mes-Acr$^+$	—	Na$_2$CO$_3$	DCE	24
9	Mes-Acr$^+$	—	NaHCO$_3$	DCE	37
10	Mes-Acr$^+$	—	NaOAc	DCE	42
11	Mes-Acr$^+$	—	Na$_2$HPO$_4$	DCE	35
12	Mes-Acr$^+$	NaBr	NaOAc	DCE	44
13	Mes-Acr$^+$	NaI	NaOAc	DCE	66
14	Mes-Acr$^+$	I$_2$	NaOAc	DCE	71
15[d]	Mes-Acr$^+$	I$_2$	NaOAc	DCE	72
16[e]	Mes-Acr$^+$	I$_2$	NaOAc	DCE	63
17[f]	—	I$_2$	NaOAc	DCE	0
18[g]	Mes-Acr$^+$	I$_2$	NaOAc	DCE	Trace

[a] Reaction conditions: chalcone **1a** (0.2 mmol), iodocyclohexane **2a** (2.0 equiv. 0.4 mmol), DABSO (0.8 equiv.), TTMS (1.0 equiv., 0.2 mmol), additives (10 mol%), base (1.5 equiv. 0.3 mmol), photocatalyst (5 mol%), solvent (3.0 mL), N$_2$, rt, under blue LED irradiation (15 W) for 48 h. [b] Isolated yield based on **1a**. [c] [Ir(dfCF$_3$ppy)$_2$(dtbbpy)]PF$_6$. [d] In the presence of base (2.0 equiv. 0.4 mmol). [e] In the presence of TTMS (1.2 equiv. 0.24 mmol). [f] In the absence of photocatalyst. [g] In the dark.

were observed when the solvent was changed to 1,2-dioxane, MeCN, or MeOH (Table 1, entries 2–4). No reaction occurred when the photocatalyst was replaced by rhodamine B (Table 1, entry 5). A slightly higher yield was afforded when 9-Mes-10-methyl acridinium perchlorate was used as the photocatalyst (Table 1, entry 6). Several inorganic bases were then screened (Table 1, entries 7–11), and it was found that the transformation worked well when sodium acetate was used leading to the expected product **3a** in 42% yield (Table 1, entry 11). Since the presence of halide would promote the conversion, the reaction was examined with the addition of sodium bromide or sodium iodide (Table 1, entries 12 and 13). Gratifyingly, the reaction with the addition of sodium iodide provided the desired product **3a** in 66% isolated yield (Table 1, entry 13). Further exploration revealed that the yield was enhanced to 71% when iodine was employed instead (Table 1, entry 14). No change was observed when the amount of base was increased (Table 1, entry 15). The yield could not be improved by changing the amount of TTMS (Table 1, entry 16). No reaction occurred in the absence of the photocatalyst (Table 1, entry 17). Only a trace
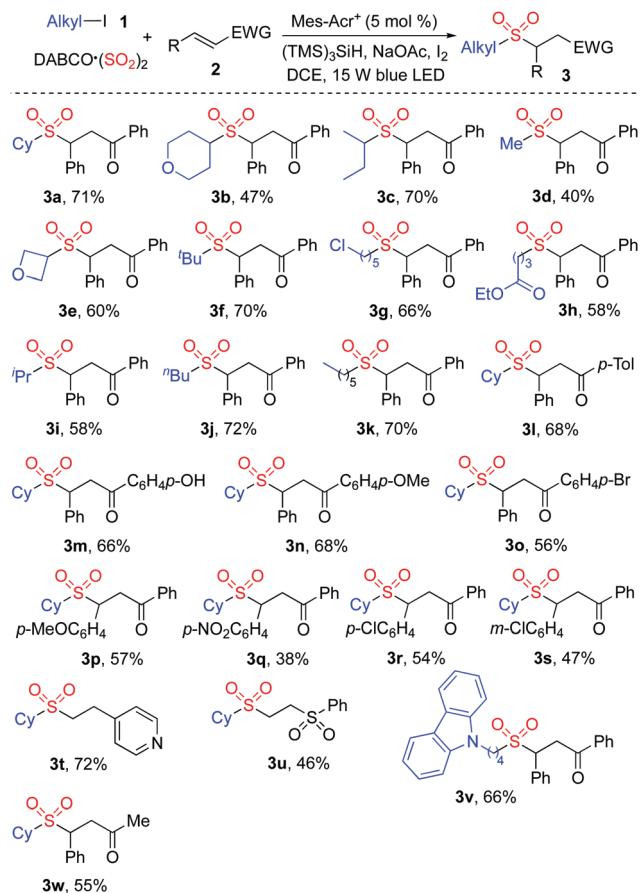
Scheme 2 Scope investigation for the photoredox-catalyzed sulfonylation of alkyl iodides **1**, sulfur dioxide, and electron-deficient alkenes **2**.

amount of the product was obtained when the reaction was performed in the dark (Table 1, entry 18).

The generality of the reaction scope was then investigated under the above optimized conditions. The result is shown in Scheme 2. It was found that this transformation with a range of alkyl iodides was efficient, and proceeded smoothly giving rise to the corresponding sulfones **3** as expected. Not only 4-iodotetrahydro-2H-pyran but also 3-iodooxetane was a suitable substrate in the reaction of DABCO·(SO$_2$)$_2$ and (E)-chalcone **2a**, leading to the desired product **3b** and **3e**, respectively. It was noteworthy that only alkyl iodide was effective in this transformation, and alkyl chloride was inert under these conditions. For example, compound **3g** was produced in 66% yield, and the chloro group was retained. The reaction of an ester-containing substrate also worked well, affording the desired product **3h** in 58% yield. Other substituted chalcones were examined subsequently, and various functional groups including methoxy, nitro, and chloro were all tolerated. Further exploration revealed that cyclohexyl iodide **1a** reacted with DABCO·(SO$_2$)$_2$ and 4-vinylpyridine giving rise to the corresponding product **3t** in 72% yield. Compound **3u** was generated in 46% yield when (vinylsulfonyl)benzene was employed as the substrate. However, the reactions failed to produce the corresponding products when other acrylates, styrenes or Michael
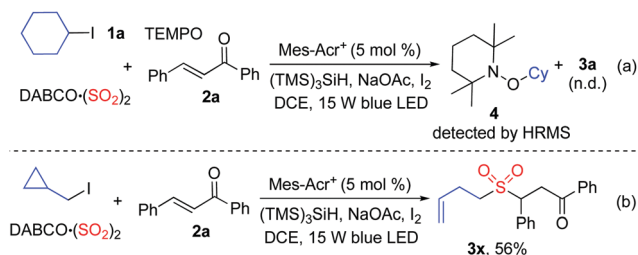
Scheme 3 Control experiments.

acceptor systems holding a β-alkyl substituent were used as substrates.

As mentioned above, we proposed that under photoredox catalysis, alkyl radical intermediates would be produced to initiate the reaction. Therefore, several control experiments were performed, as presented in Scheme 3. As expected, no reaction occurred with the addition of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) in the mixture of cyclohexyl iodide **1a**, DABCO·(SO$_2$)$_2$, and (*E*)-chalcone **2a** under the standard conditions, and compound **4** was detected by HRMS (Scheme 3, eqn (a)). Additionally, (iodomethyl)cyclopropane was employed in the reaction of DABCO·(SO$_2$)$_2$ with (*E*)-chalcone **2a**, resulting in the formation of compound **3x** in 56% yield (Scheme 3, eqn (b)). This result demonstrated that a cyclopropylmethyl radical might be generated during the reaction process. These results indicated that a radical process might be involved, as proposed in Scheme 4.

On the basis of the above results and previous reports,[11] we postulated that under visible light irradiation, the excited state of the photocatalyst would assist the formation of a trimethyl-silylsilyl radical (Scheme 4). Thus, the trimethylsilylsilyl radical would react with alkyl iodide **1** leading to an alkyl radical intermediate, which would be captured by sulfur dioxide to provide an alkylsulfonyl radical. Then, the alkylsulfonyl radical would attack the double bond of electron-deficient alkene **2**, giving rise to radical intermediate **A**. Subsequently, two possible pathways might occur. In path a, radical intermediate **A** would undergo a reductive single electron transfer (SET) to produce anion intermediate **B**. Further protonation would afford the corresponding sulfone **3**. Alternatively, radical **A** would go through
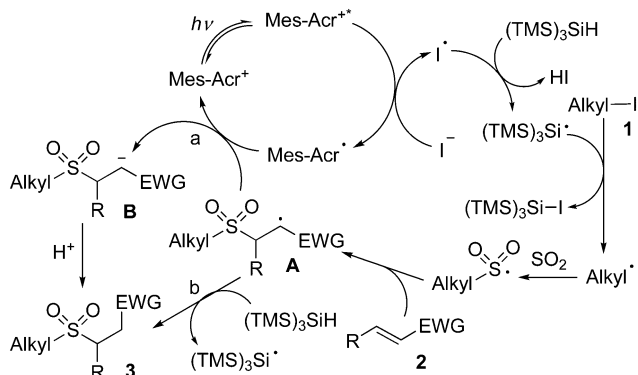
proton abstraction from TTMS, giving rise to the desired product **3** (path b).

In summary, we have described a photoredox-catalyzed sulfonylation of alkyl iodides, sulfur dioxide, and electron-deficient alkenes under mild conditions. This reaction proceeds through alkyl radicals formed *in situ* from alkyl iodides under visible light irradiation in the presence of a photoredox catalyst. The alkyl radical intermediates would react with sulfur dioxide leading to alkylsulfonyl radicals, which would be trapped by electron-deficient alkenes giving rise to alkyl sulfones. Various functional groups including nitro, halo, acetyl, sufonyl, and pyridinyl are all tolerated under the photoredox conditions.

Conflicts of interest

There are no conflicts to declare.

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