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Visible-light photoredox-catalyzed dual C–C bond cleavage: synthesis of 2-cyanoalkylsulfonylated 3,4-dihydronaphthalenes through the insertion of sulfur dioxide†

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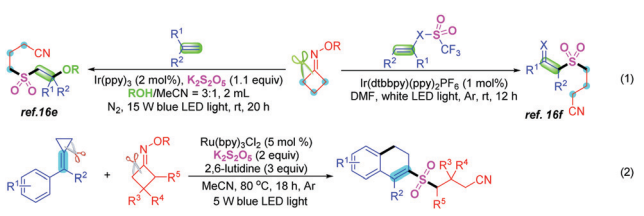
An efficient novel visible-light photoredox-catalyzed dual carbon–carbon bond cleavage of methylenecyclopropanes and cycloketone oximes for the synthesis of 2-cyanoalkylsulfonated 3,4-dihydronaphthalenes through the insertion of sulfur dioxide is established. This dual cleavage of carbon–carbon bonds involves a radical pathway and goes through a sequence of iminyl radical formation, carbon–carbon bond cleavage, sulfur dioxide insertion, sulfonyl radical addition, another carbon–carbon bond cleavage, and intramolecular cyclization.

Sulfur-containing derivatives are important synthons in organic synthesis and drug synthesis.[1,2] Sulfones, a class of useful organosulfur derivatives, have already been widely used in the chemical industry, agrochemicals, pharmaceuticals, organic synthesis and inflaming retarding materials.[3,4] Due to the extensive application of sulfones, the approaches for the construction of sulfones have attracted organic chemists' attention and many novel strategies have been established.[5,6] Among them, the insertion of sulfur dioxide was the most convenient way for the synthesis of sulfones. DABSO [DABCO·$(SO_2)_2$],[7] sodium sulfites[8a–d] and thiourea dioxide[8e,f] were usually used as sulfur dioxide sources to prepare sulfones.

Alkylnitriles are an important class of organic skeletons encountered in many nitrile-containing pharmaceuticals and natural products.[9] Moreover, cyanoalkyl groups could easily be transformed into other functional groups.[10] Thus, the development of efficient and convenient ways to access alkylnitriles has become interesting. Typical strategies to synthesize alkylnitriles include dehydrogenation of amines,[11a–c] dehydration of aldoximes[11d–g] or amides,[12a–c] and cyanation of alkyl halides.[12d–g] Recently, the functionalization of α-C–H bonds in inert alkylnitriles has also been developed.[13] The cleavage of carbon–carbon σ-bonds in cyclobutanone oximes has also emerged as a convenient route to import cyanoalkyl groups, which bear longer aliphatic chains, into organic molecules.[14–16] In 1991, Zard *et al.*[14a] developed the first radical carbon–carbon bond cleavage of cyclobutanone sulphenylimines or carboxymethyl oximes to prepare alkylnitriles. Afterwards, many organic chemists, for example, Uemura's,[14b,c] Selander's,[14d,e] Xiao's,[14f–j] Guo's,[15a–c] Yu's,[15d,e] Wu's,[15f,g] Li's[15h] and other groups,[16] have reported similar ring-opening reactions of cyclobutanone oxime derivatives. Most of these methods employed cyanoalkyl radicals, which came from ring-opening of cyclobutanone oximes, to directly react with other reaction partners. However, the methods which used cyanoalkyl radicals to capture SO_2[16e,f] and then formed sulfonyl radicals were very few (eqn (1), Scheme 1).

MCPs (methylenecyclopropanes) were usually used to react with diverse free radicals,[17] such as CF_3,[17a] SCF_3,[17b] alkyl,[17c] acyl,[17d] α-carbonyl[17e] and sulfonyl[17f–h]-containing radicals. In the reaction between MCPs and sulfonyl radicals, the sulfonyl radicals usually came from sulfonyl chlorides and sodium sulfinates. However, strategies for ring-opening and cyclization of MCPs with cyanoalkylsulfonyl radicals, which formed from cyclobutanone oximes and sulfur dioxide sources, are lacking.

Visible-light-catalysis, as a convenient and simple tool, is widely used in organic synthesis because of its safety, mild reaction conditions, availability, and high efficiency.[18] Herein, we develop a visible-light photoredox-catalyzed difunctionalization



Scheme 1 *In situ* SO_2-capture reactions of cycloketone oxime esters.

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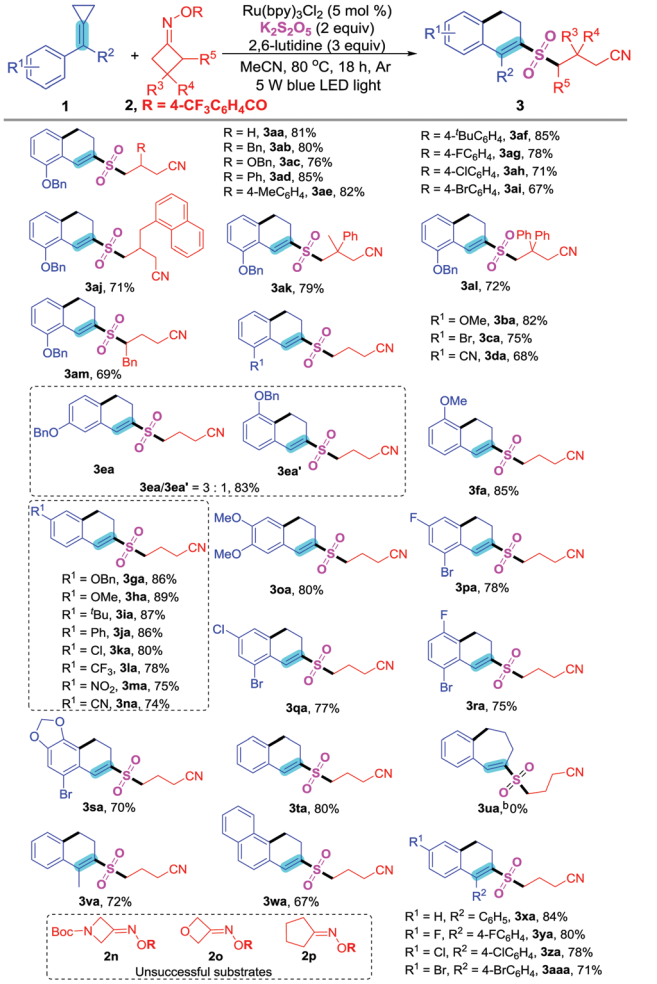
of carbon–carbon σ-bonds to construct 2-cyanopropylsulfonyl-substituted 3,4-dihydronaphthalenes from MCPs, cyclobutanone oximes and $K_2S_2O_5$, which features the cleavage of two carbon–carbon σ-bonds and the formation of one new carbon–carbon bond and two carbon-sulfur bonds in one-pot (eqn (2), Scheme 1).

We chose 1-(benzyloxy)-2-(cyclopropylidenemethyl)benzene **1a** and cyclobutylidenemethyl 4-(trifluoromethyl)benzoate **2a** as model substrates to clarify the standard reaction conditions. To our surprise, the reaction employing $Ru(bpy)_3Cl_2$ as a photocatalyst, $K_2S_2O_5$ as a sulfur dioxide source, 2,6-lutidine as base, and CH_3CN as solvent at 80 °C and irradiated with a 5 W blue LED light source for 18 h could afford the product 4-((8-(benzyloxy)-3,4-dihydronaphthalen-2-yl)sulfonyl)butanenitrile **3aa** in 81% yield. Various sulfur dioxide sources, cyclobutanone *O*-benzoyl oximes, photocatalysts, solvents, visible-light sources, temperatures and reaction times were successively examined, and the optimized conditions were as follows: 1-(benzyloxy)-2-(cyclopropylidenemethyl)benzene **1a** (0.2 mmol), cyclobutylidenemethyl 4-(trifluoromethyl)benzoate **2a** (0.3 mmol, 1.5 equiv.), $Ru(bpy)_3Cl_2$ (0.01 mmol, 5 mol%), 2,6-lutidine (0.6 mmol, 3 equiv.), $K_2S_2O_5$ (0.4 mmol, 2 equiv.), CH_3CN (2 mL) at 80 °C and irradiated with a 5 W blue LED light source for 18 h (see Table S1 in the ESI†).

Based on the optimal conditions, we initially investigated the scope of MCPs **1** and cycloketone oxime esters **2** (Table 1). A series of substituted cycloketone oxime esters **2** were examined in the presence of MCPs **1a** under the standard conditions. The results revealed that 3-substituted cyclobutanone oximes **2b–j** bearing benzyl, benzyloxy, aryl and naphthalen-1-ylmethyl groups were all suitable substrates. The corresponding products **3ab–aj** could be obtained in moderate yields. Employing 3,3-disubstituted cyclobutanone oximes **1k** and **1l** in the reaction could provide the sulfonated products **3ak** and **3al** in 79% and 72% yields, respectively. The 2-substituted cycloketone oxime ester **2m** was examined and the target product **3am** could be obtained in 69% yield. Then, our attention was turned to research the scope of MCPs **1** in the presence of cycloketone oxime esters **2a**, $Ru(bpy)_3Cl_2$, $K_2S_2O_5$, 2,6-lutidine and CH_3CN. A variety of mono-substituted MCPs, which bear one substituent at the *ortho*-, *meta*- or *para*-position on the aryl ring, were examined. All of them could undergo the dual carbon–carbon bond cleavage/sulfonylation reaction smoothly and afforded the corresponding 2-cyanoalkylsulfonylated 3,4-dihydronaphthalenes **3** in moderate yields (**3aa–na**). The reaction yields indicated that both the hindrance and electronic effect of the substituents had obvious influences on the transformation: the reactivity order is *ortho*- < *meta*- < *para*- and electron-withdrawing < electron-donating. However, the *meta*-substituted MCP **1e** gave products **3ea** and **3ea′** (3 : 1) in 83% yield. Subsequently, di-substituted MCPs **1o–r** and poly-substituted MCP **1s** were also explored and all of them were suitable for this reaction (products **3oa–sa**). Halogen substituted MCPs **1p–s** were suitable for this transformation, which provided opportunities for further modification of the products (products **3pa–sa**). Additionally, phenyl-substituted MCP **1t** was also a good candidate for this reaction. However, the four-membered substrate **1u** could not install the target product **3ua**. Substrate **1v**, which connected a phenyl group and a methyl,

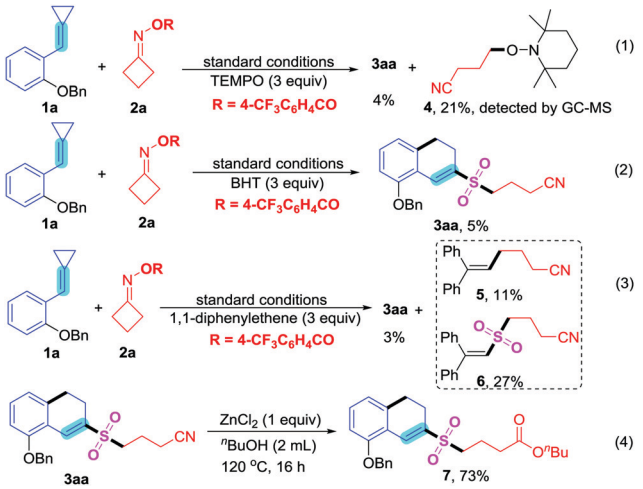
Table 1 Scope of cycloketone oxime esters (**2**) and MCPs (**1**)[a]



[a] Reaction conditions: **1** (0.2 mmol), **2a** (0.3 mmol, 1.5 equiv.), $K_2S_2O_5$ (0.4 mmol, 2 equiv.), $Ru(bpy)_3Cl_2$ (5 mol%), 2,6-lutidine (0.6 mmol, 3 equiv.), CH_3CN (2 mL) at 80 °C under an argon atmosphere and 5 W blue LED irradiation for 18 h. [b] Most of the substrate **1** was decomposed.

could react well with **2a** to deliver the sulfonylated product **3va** in 72% yield. The naphthyl-substituted MCP **1w** could also deliver product **3wa** under the standard conditions (product **3wa**). Finally, a series of MCPs **1x–aa** connecting two aryl rings at the terminal of the carbon–carbon double bond also worked well and afforded the ring-opening and cyclization products **3xa–3aaa** in good yields. However, 1-Cbz-3-azetidinone, oxetan-3-one and cyclopentanone derived *O*-acyl oximes **2n**, **2o** and **2p** could not give the target products **3** under the standard conditions.

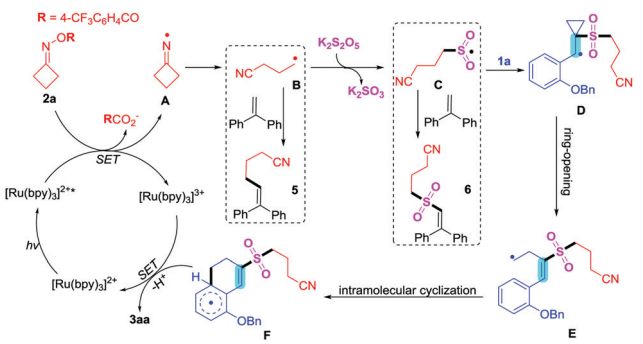
To gain further understanding of the reaction mechanism, three radical trapping experiments were carried out. The yields of MCPs **1a** with cycloketone oxime esters **2a** and $K_2S_2O_5$ were very low when these reactions were carried out by using TEMPO, hydroquinone and 1,1-diphenylethene as radical inhibitors (eqn (1)–(3), respectively, Scheme 2). Additionally, using TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl) in the transformation could afford 4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)butanenitrile



Scheme 2 Control experiments.

4 (eqn (1), Scheme 2). The trapping product **4** could be detected by GC-MS. Using 1,1-diphenylethene in the sulfonylation reaction could give cyanoalkyl group-trapping product 6,6-diphenylhex-5-enenitrile **5** and cyanoalkylsulfonyl group-trapping product 4-((2,2-diphenylvinyl)sulfonyl)butanenitrile **6** in 11% and 27% yields, respectively (eqn (3), Scheme 2). These experimental results suggested that this transformation definitely contained a radical process. Additionally, the cyano group could be transformed into a series of other functional groups, such as carboxyl,[14g,15b,16a] amide,[16a] ester,[16a] and tetrazole[15b] groups. The cyano group in the sulfonylated product **3aa** could be converted into an ester group in the presence of $ZnCl_2$ and *n*-butyl alcohol (eqn (4), Scheme 2).

According to the literature[14–18] and above results, we proposed a mechanism for the visible-light photoredox-catalyzed dual cleavage of C–C bonds (Scheme 3). Initially, $[Ru(bpy)_3]^{2+}$ was transformed into $[Ru(bpy)_3]^{2+*}$ photoexcited under irradiation with visible-light.[17f,18c] Subsequently, the reduction of cycloketone oxime **2a** by $[Ru(bpy)_3]^{2+*}$ provided iminyl radical **A**, which underwent cleavage of the carbon–carbon bond to deliver cyanoalkyl radical **B**. Next, intermediate **B** trapped sulfur dioxide, which came from $K_2S_2O_5$, to give cyanoalkylsulfonyl radical **C**. Both the cyanoalkyl radical **B** and cyanoalkylsulfonyl radical **C** could be trapped by



Scheme 3 Possible reaction mechanism.

1,1-diphenylethene to give products **5** and **6**, respectively. Then, the addition of cyanoalkylsulfonyl radical **C** to the carbon–carbon double bond in substrate **1a** afforded radical **D**, which went through another cleavage of the carbon–carbon bond to install radical **E**. The intramolecular cyclization of intermediate **E** assembled radical **F**. Finally, radical **F** underwent SET by $[Ru(bpy)_3]^{3+}$ and deprotonation by 2,6-lutidine to provide the target product **3aa**, and $[Ru(bpy)_3]^{3+}$ was reverted to ground-state $[Ru(bpy)_3]^{2+}$.

In summary, we have developed a facile and convenient route to diverse 2-cyanoalkylsulfonated 3,4-dihydronaphthalenes *via* visible-light photoredox-catalyzed dual C–C bond cleavage of methylenecyclopropanes and cycloketone oxime esters by the insertion of sulfur dioxide. This reaction follows a radical pathway and goes through a sequence of iminyl radical formation, C–C bond cleavage, sulfur dioxide insertion, sulfonyl radical addition, another C–C bond cleavage, and cyclization. Both the cyanoalkyl radical and cyanoalkylsulfonyl radical were trapped by radical inhibitors. Further research and application of this dual C–C bond cleavage reaction are currently underway in our laboratory.

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Conflicts of interest

There are no conflicts to declare.

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