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[3 + 2]-Cycloaddition of 2*H*-Azirines with Nitrosoarenes: Visible-Light-Promoted Synthesis of 2,5-Dihydro-1,2,4-oxadiazoles

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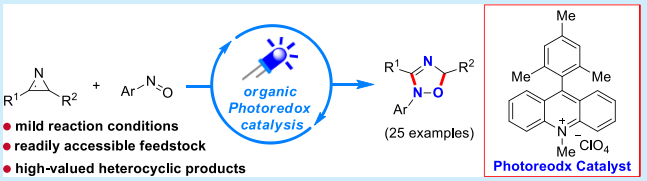
Supporting Information

ABSTRACT: A formal [3 + 2]-cycloaddition reaction of 2*H*-azirines with nitrosoarenes has been achieved under irradiation by visible light with the assistance of organic dye photoredox catalyst. This method utilizes nitrosoarenes as efficient radical acceptors and provides a green and powerful method for a series of biologically important 1,2,4-oxadiazole derivatives in moderate to good yields.



The oxadiazole skeleton is one of the most important five-membered nitrogen-containing heterocyclic compounds.[1] In the oxadiazole family, molecules containing 1,2,4-oxadiazole moieties not only are frequently found in many natural and artificial products showing interesting physiological and biological activities but also apply as the functionalized organic materials (Figure 1).[2] As representative examples shown in

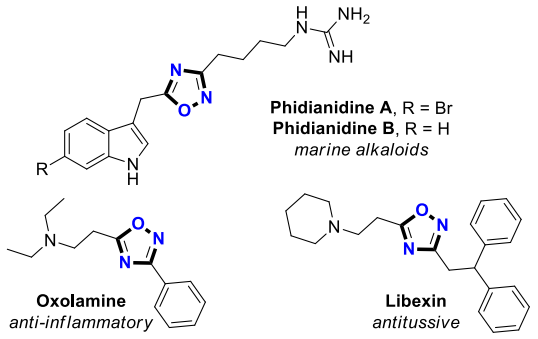


Figure 1. Some biologically important structures containing 1,2,4-oxadiazole motifs.

Figure 1, phidianidines A and B were isolated from marine opisthobranch mollusk *Phidiana militaris*, which exhibits cytotoxicity against tumor and nontumor mammalian cell lines in in vitro assays.[3] Drugs containing a 1,2,4-oxadiazole core, such as libexin, exhibit antitussive activity, while oxolamine has anti-inflammatory activity.[4] Driven by their rich biological activities, the development of efficient and practical methods for the synthesis 1,2,4-oxadiazole is of widespread interest in synthetic organic chemistry. Traditionally, 1,2,4-oxadiazole derivatives are accessed by 1,3-dipolar cycloaddition of nitriles to nitrile oxides or thermally promoted cyclization of in situ formed amidoxime derivatives.[5] Despite these elegant methods, further exploration of new approaches for highly efficient preparation of structurally diverse 1,2,4-oxadiazoles under green and benign reaction conditions is still highly desirable.

The cycloaddition reaction is a powerful strategy to build molecular complexity into heterocycles.[6] In this regard, nitrosoarenes have been extensively investigated due to their high reactivity profile in the preparation of highly functionalized heterocyclic compounds.[7] The pioneering work toward the [3 + 2]-cycloaddition of nitrosoarene as dienophiles was demonstrated by the Studer group in 2011, in which allylstannanes were used as the formal 1,3-dipoles.[8] Shortly after this discovery, [3 + 2]-cycloaddition of nitrosoarene with donor−acceptor cyclopropanes (DAC) was also realized by the same group.[9] Compared with those well-established ionic cycloaddition processes where nitrosoarenes are employed as electrophiles, reactions of nitrosoarenes as radical acceptors have been considerably overlooked.[10] In another arena, chemical reactions initiated by visible-light-induced photoredox catalysis have attracted much attention in the past several years due to its environmental compatibility and versatility in character.[11] However, to the best of our knowledge, visible-light-promoted transformation of nitrosoarenes, especially in [3 + 2]-cycloadditions toward the construction of heterocycles, has not yet been disclosed.

In 2017, Sheikh, Leonori, and co-workers reported a visible-light-promoted decarboxylative radical addition/protonation of carboxylic acids and nitrosoarenes, where nitrosoarenes serve as suitable radical acceptors to form persistent nitroxyl radical intermediates (Scheme 1a).[12,13] Motivated by these findings

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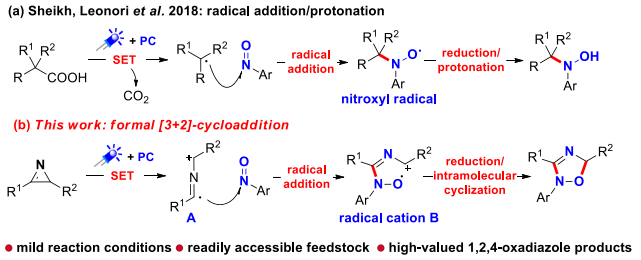
Scheme 1. Nitrosoarenes as Radical Acceptors in Visible-Light-Induced Photoredox Catalysis

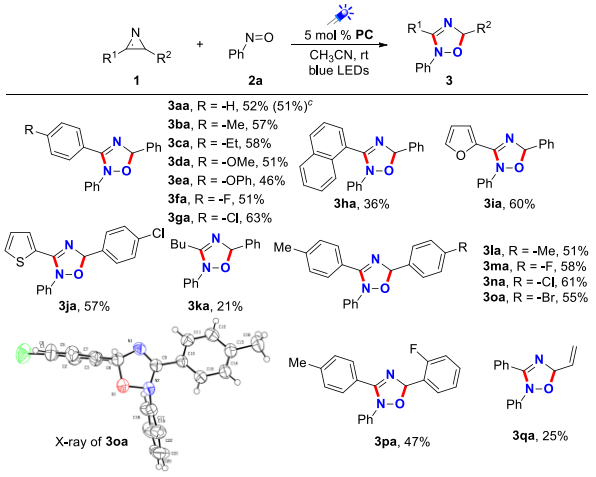


and our ongoing research interest in the development of heterocycle-oriented methodologies,[14] we became interested in further exploring the potential reactivity of nitrosoarenes with other types of radical species, such as radical cation **A**, generated from the ring opening of 2*H*-azirines under visible-light irradiation.[15] We assumed that the formed radical cation **B** might undergo a SET reduction/intramolecular cyclization sequence, thus providing a new avenue to access biologically important 2,5-dihydro-1,2,4-oxadiazoles (Scheme 1b). Herein, we describe the preliminary results of this study.

At the outset, 2,3-diphenyl-2*H*-azirine **1a** and nitrosobenzene **2a** were selected as model substrates to test the feasibility of the designed process. Fortunately, condition optimization revealed that the proposed formal [3 + 2]-cycloaddition reaction did indeed occur with 52% isolated yield in the presence of 5 mol % of 9-mesityl-10-methylacridinium perchlorate[16] as photoredox catalyst in degassed CH_3CN, under irradiation with blue LED for 5 h. Control experiments indicated that the photoredox catalyst, blue LED irradiation, and degassing procedure are all crucial to the process (for details of the optimization and control experiments, see the Supporting Information). To our delight, an almost similar yield was obtained when the reaction was enlarged to 1.0 mmol scale (10 h, 51%).

After identifying the optimal reaction conditions, we set out to explore the scope of this formal [3 + 2]-cycloaddition process by reacting a series of substituted 2*H*-azirines **1** with nitrosobenzene **2a**. As the results in Scheme 2 revealed, the electronic property variation on the R^1-phenyl ring of 2*H*-azirines **1** has no obvious effect on the reaction efficiency. Both electron-rich (−Me, −Et, −OMe, −OPh) and electron-deficient substituents (−F, −Cl) at the *para*-position were suitable for this photocatalytic transformation, providing the corresponding 1,2,4-oxadiazole derivatives **3ba**−**ga** in good yields (46−63%). Note that reactions with 2*H*-azirines bearing R^1-naphthyl (**1h**) and R^1-heterocycles such as furan (**1i**) and thiophene (**1j**) proceeded smoothly. The R^1-aryl substituent could be replaced by an alkyl substituent as documented for the butyl congener, albeit with relative low yield (**3ka**, 21%). Moreover, substituent modification on the R^2-phenyl ring also proved to be successful, giving the [3 + 2]-cycloaddition adducts **3la**−**pa** in average good yield. It is worth noting that the structure of **3oa** was unambiguously confirmed by X-ray diffraction analysis. The 3-phenyl-2-vinyl-2*H*-azirine **3q** is also a suitable substrate for this photocatalytic [3 + 2]-cycloaddition process to give 2,5-dihydro-1,2,4-oxadiazole **3qa** in 25% yield. The aryl groups in 2*H*-azirines **1** are crucial for the high yields of 2,5-dihydro-1,2,4-oxadiazole products, which might be because it can stabilize the formed radical-cation intermediate **B** during the reaction (Scheme 6).[15]

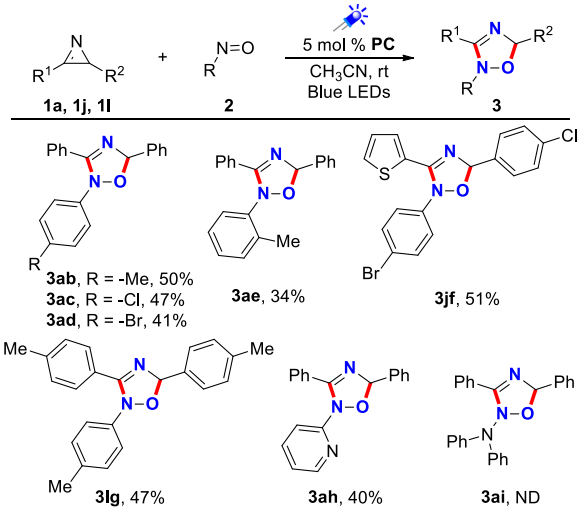
Scheme 2. Substrate Scope of 2*H*-Azirines[a,b]



[a]**1** (0.1 mmol), **2a** (0.2 mmol), and **PC** (0.05 mmol) in degassed CH_3CN (1.0 mL) under irradiation with 24 W blue LEDs. [b]Isolated yield. [c]The reaction was carried out on a 1.0 mmol scale. **PC** = 9-mesityl-10-methylacridinium perchlorate.

We next turned our attention to further investigating the substrate scope with respect to the nitrosoarene component (Scheme 3). Further underscoring the efficiency of this [3 + 2]-cycloaddition process, 2,5-dihydro-1,2,4-oxadiazole products were obtained in reasonably good yields when the *para*- and *ortho*-substituted nitrosobenzenes and 2-nitrosopyridine (**2h**) were treated with 2*H*-azirines under the standard reaction conditions (**3ab**−**ae**, **3jf**, **3lg**, and **3ah**). With respect to reaction efficiency, better results were observed for *para*-substituted nitrosoarenes compared to *ortho*-substituted substrates, which might be due to the steric effect. Note that a nitroso compound bearing electron-rich amine motifs, such as *N*-nitrosodiphenylamine **2i**, did not react at all.

Scheme 3. Substrate Scope: Variation of Nitrosoarenes[a,b]



[a]**1a**, **1j**, or **1l** (0.1 mmol), **2** (0.2 mmol), and **PC** (0.05 mmol) in degassed CH_3CN (1.0 mL) under irradiation with 24 W blue LEDs. [b]Isolated yield. **PC** = 9-mesityl-10-methylacridinium perchlorate.

The preparative utility of this method can be demonstrated by exploring the follow-up chemistry as depicted in Scheme 4.

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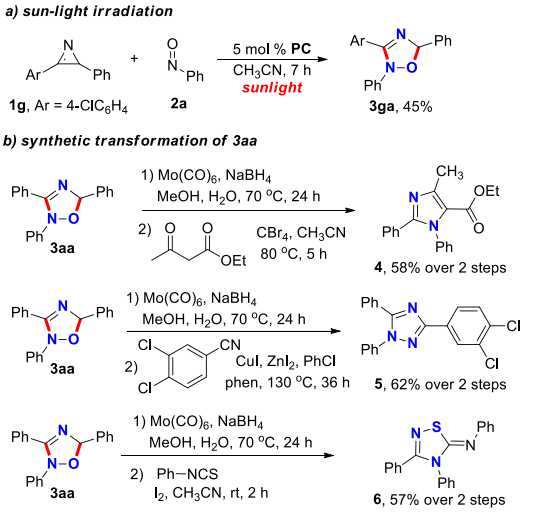
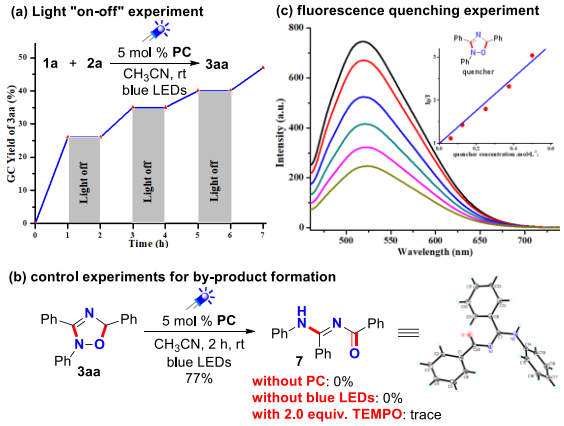
Scheme 4. Follow-up Chemistry



Direct sunlight irradiation of the reaction of **1g** with **2a** provided the desired 2,5-dihydro-1,2,4-oxadiazole **3ga** with moderate yield. It was found that reductive N–O bond cleavage in **3aa** with $Mo(CO)_6/NaBH_4$ afforded *N*-phenylbenzimidamide in 65% yield (see the SI). This provided us a good option to transfer 1,2,4-oxadiazole derivatives to other biologically important heterocycles, such as imidazole **4**,[18a] triazole **5**,[18b] and thiadiazole **6**,[18c] after a two-step simple operation.

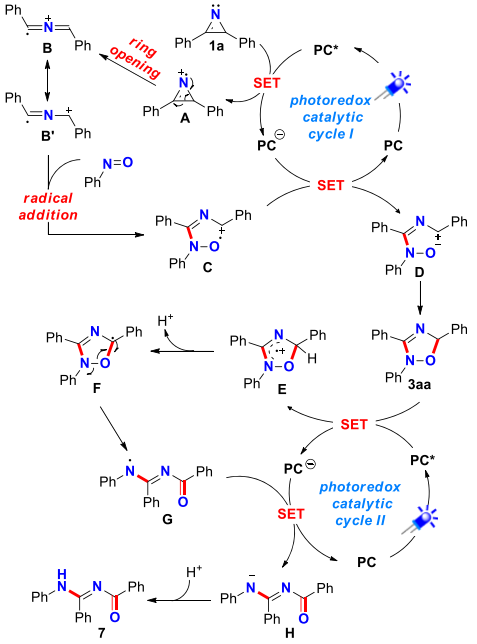
Regarding the mechanism, we found that this [3 + 2]-cycloaddition process required continuous visible-light irradiation based on the results of light "on–off" experiments (Scheme 5a). In the reaction system, we can isolate a ring-opening byproduct, and this is also the major reason for the relatively low yields of the [3 + 2]-cycloaddition products in some cases. To gain insight into the formation of this byproducts, some control experiments were conducted (Scheme 5b). Treatment of **3aa** under the standard reaction conditions provided byproduct **7** in 77% isolated yield after 2 h irradiation. Control experiments revealed that both of photoredox catalyst and light irradiation were essential for the formation of **7** from **3aa**. In addition, only a trace amount of **7** was observed when 2.0 equiv of TEMPO was added to the reaction system. These results clearly revealed that **7** was generated from **3aa** under photoredox catalytic reaction conditions, and this ring-opening reaction included a radical step. Stern–Volmer fluorescence quenching experiments indicated that **3aa** could efficiently quench the photoexcited 9-mesityl-10-methylacridinium perchlorate, where it presumably engaged in a SET process in the reaction (Scheme 5c). Moreover, the oxidative potential of **3aa** in CH_3CN was determined to be +1.21 V vs SCE (see the SI), indicating that direct oxidation of **3aa** by the photoexcited 9-mesityl-10-methylacridinium perchlorate ($E_{1/2}^{red}* = +2.06$ V vs SCE)[16] is thermodynamically favored.

Scheme 5. Mechanistic Experiments



On the basis of the above observations and the literature reports, a plausible reaction mechanism is proposed in Scheme 6. The formation of radical cation intermediate **B** or **B′** from ring-opening of 2*H*-azirines under photocatalytic conditions was the same as in previous reports.[15] Radical addition of **B** or **B′** to nitrosobenzene affords radical cation species **C**, which subsequently undergoes a single-electron reduction/intramolecular cyclization cascade to give the formal [3 + 2]-cycloaddition adduct **3aa** and regenerates the photoredox catalyst to complete the first photocatalytic cycle. As a competing process, the formed 1,2,4-oxadiazole can serve as reductive quencher to reduce the photoexcited 9-mesityl-10-methylacridinium perchlorate and generate a resonance-stabilized radical cation **E**. Deprotonation of **E** afforded the α-amino radical species **F**.[17] The N–O cleavage of **F** provides N-centered radical intermediate **G**, which is reduced by a low-valent photoredox catalyst to complete the second photocatalytic cycle and provides the nitrogen anion **F**. Finally, protonation of **F** gives the ring-opening byproduct.

Scheme 6. Plausible Reaction Mechanism



In conclusion, we have developed a visible-light-promoted [3 + 2]-cycloaddition reaction of 2*H*-azirines with nitrsoarenes. The method utilizes nitrosoarenes as suitable radical acceptors, which provides a green and efficient method to a series of biologically important 1,2,4-oxadiazole derivatives in average good yields. More importantly, the direct sunlight-irradiation process and the easy synthetic transformation of the final 2,5-dihydro-1,2,4-oxadiazoles to other biologically important

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heterocycles further renders the approach attractive and valuable. The further discovery of new cycloaddition reactions by using the nitrosoarenes as radical acceptors is currently underway in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b01416.

Experimental procedures, characterization data, and ^{1}H, and ^{13}C NMR spectra (PDF)

Accession Codes

CCDC 1904024 and 1904026 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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