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Visible-Light-Mediated Decarboxylative Tandem Carbocyclization of Acrylamide-Attached Alkylidenecyclopropanes: Access to Polycyclic Benzazepine Derivatives

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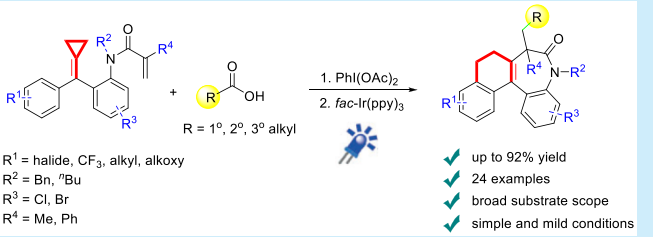
ABSTRACT: A visible-light-mediated decarboxylative tandem carbocyclization of acrylamide-tethered alkylidenecyclopropanes with phenyliodine(III) diacetate and various aliphatic acids has been reported in this paper. An alkyl radical in situ generated from phenyliodine(III) dicarboxylates upon visible-light irradiation catalyzed by *fac*-Ir(ppy)$_3$ adds to the less hindered central carbon of alkylidenecyclopropane to initiate the tandem annulation, generating tetracyclic benzazepine derivatives in moderate to good yields with broad substrate scope under mild conditions.



The azepine skeleton is one of the most ubiquitous heterocycles in a variety of biologically active and medicinally valuable compounds.[1] As its structural units, polyheterocyclic benzazepine motifs built on the azepine backbone exhibit significant biological and pharmaceutical activities (Figure 1).[2] For example, compound **A**, in which the

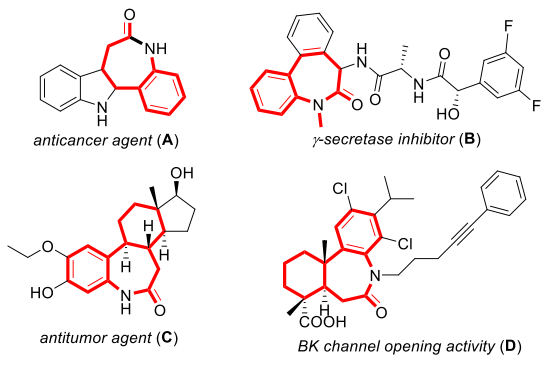


Figure 1. Representative nature products containing benzazepine motifs.

benzazepine fused with indoline, is an anticancer agent. Compound **B** can be used as a γ-secretase inhibitor in the therapy of Alzheimer's disease.[2d] As a metabolite of estrogen, compound **C** is demonstrated as an inhibitor for the proliferation of various cell types and also can be employed as an antiangiogenic and antitumor agent.[2e] Hexahydrodibenzazepinone derivative **D**, exhibiting BK channel opening activities, has potential therapeutic applications in different pathophysiological conditions such as coronary artery spasm, hypertension, progressive deafness, and urinary incontinence.[2f]

Therefore, many efforts have been devoted to explore general and facile protocols for the rapid construction of this kind of pivotal benzazepine motifs in the field of organic synthesis and medicinal chemistry owing to their indispensable applications.

Carboxylic acids, as promising fine chemical feedstock, can be applied to rapidly expand libraries of complex small molecule.[3] Among all of their applications, the decarboxylative reduction of carboxylic acids is a general synthetic transformation.[4] However, the decarboxylative functionalization of carboxylic acids with easily accessible phenyliodine(III) diacetate (DIB) has been rarely reported so far.[5,6] Liu's group employed the organocatalyst to achieve arylalkylation of activated alkenes through decarboxylation of PhI(O$_2$C^{t}Bu)$_2$, affording a variety of oxindoles in good yields (Scheme 1a).[5c] Later on, Zhu and co-workers reported a visible-light-promoted decarboxylative tandem reaction of *N*-methyl-*N*-phenylmethacrylamides with phenyliodine(III) dicarboxylates in the presence of *fac*-Ir(ppy)$_3$ to provide 3,3-disubstituted oxindoles as products in one step in good yields (Scheme 1b).[5d]

During recent years, photoredox catalysis induced by visible light has experienced significant advances due to its special advantages compared with traditional photochemistry such as ease of handling, application safety, natural abundance, and mild conditions.[7] Thus far, our group has been focusing on the exploration of visible-light induced photoredox catalytic

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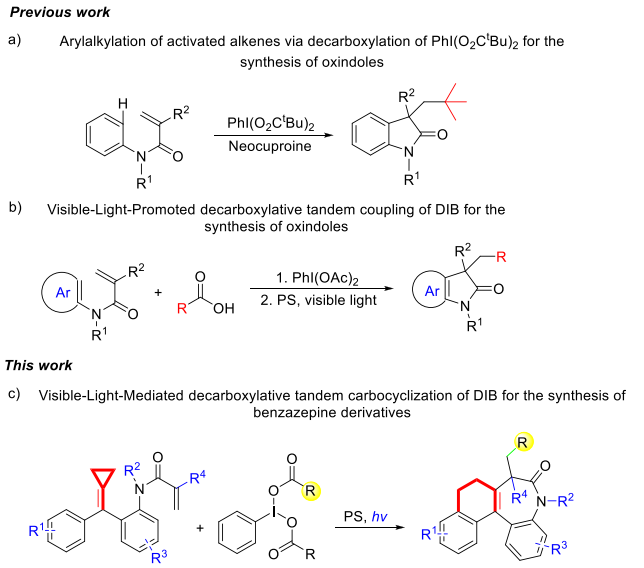
Scheme 1. Decarboxylative Tandem Cyclization Using Phenyliodine(III) Diacetate (DIB) or Its Derivatives

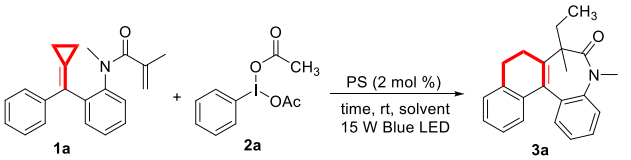


reactions of alkylidenecyclopropanes (ACPs), a highly strained but readily accessible molecule, for the rapid synthesis of complicated molecules.[8] Herein, we demonstrate a novel visible-light-mediated decarboxylative tandem carbocyclization using acrylamide-attached alkylidenecyclopropanes with various in situ generated phenyliodine(III) dicarboxylates for the formation of functionalized seven-membered azepine polycyclic structures (Scheme 1c, this work).

To commence our working hypothesis, *N*-(2-(cyclopropylidene(phenyl)methyl)phenyl)-*N*-methylmethacrylamide **1a** and phenyliodine(III) diacetate (DIB) **2a** (2.0 equiv) were utilized as the model substrates to examine the reaction outcome and subsequently to optimize the reaction conditions, and the results are shown in Table 1. Initially, an array of photosensitizers was screened using DMF as solvent upon irradiation with a 15 W blue LED for 24 h (Table 1, entries 1−4). To our delight, *fac*-Ir(ppy)$_3$ was proven as the best photosensitizer, and the expected tetracyclic benzazepine product **3a** was indeed formed and could be obtained in the yield of 86% (Table 1, entry 4). Using this photocatalyst, we further screened the common solvents and found that the use of other solvents, such as DCM, THF, DMA, PhCl, PhMe, and MeCN, afforded **3a** in moderate yields ranging from 42% to 70% and identified DMF as the most suitable solvent for this reaction (Table 1, entries 5−10). Using 1.0 equiv of **2a** or shortening the reaction time to 12 h resulted in a decreased yield of **3a** to 54% and 45%, respectively (Table 1, entries 11 and 12). Moreover, the control experiments revealed that no reaction occurred when the visible light or catalyst *fac*-Ir(ppy)$_3$ was not employed (Table 1, entries 13 and 14). The X-ray diffraction pattern of **3a**, whose structure had been unambiguously determined is shown in Figure 2. The related CIF data of **3a** are indicated in the Supporting Information.

Having established the optimized reaction conditions, we subsequently investigated the generality of this tandem reaction. The results are shown in Scheme 2. Under the optimal conditions, most substrates underwent the reactions smoothly, generating the products **3b**−**3m** in good yields. Employing substrates **1b** and **1c** bearing different *N*-protecting groups R^2, the yields of desired products **3b** and **3c** were

Table 1. Optimization of the Reaction Conditions for Visible-Light-Mediated Decarboxylative Tandem Carbocyclization Reaction of 1a



entry[a]	PS	solvent	time (h)	yield[b] (%)
1	Ru(bpy)$_3$(PF$_6$)$_2$	DMF	24	28
2	Ru(bpz)$_3$(PF$_6$)$_2$	DMF	24	44
3	Ir(ppy)$_2$(dtbbpy)PF$_6$	DMF	24	36
4	***fac*-Ir(ppy)$_3$**	**DMF**	**24**	**86**[c]
5	*fac*-Ir(ppy)$_3$	DCM	24	62
6	*fac*-Ir(ppy)$_3$	THF	24	59
7	*fac*-Ir(ppy)$_3$	DMA	24	70
8	*fac*-Ir(ppy)$_3$	PhCl	24	66
9	*fac*-Ir(ppy)$_3$	PhCH$_3$	24	45
10	*fac*-Ir(ppy)$_3$	MeCN	24	42
11[d]	*fac*-Ir(ppy)$_3$	DMF	24	54
12	*fac*-Ir(ppy)$_3$	DMF	12	45
13[e]	*fac*-Ir(ppy)$_3$	DMF	24	ND
14[f]		DMF	24	ND

[a]The reaction was run under the following conditions: a solution of **1a** (0.1 mmol), DIB **2a** (0.2 mmol, 2.0 equiv), and photosensitizer (0.002 mmol) in dry solvent (0.5 mL) was stirred at room temperature under nitrogen atmosphere. [b]Yields were determined by ^{1}H NMR spectroscopic using CH_2Br_2 as an internal standard. [c]Isolated yield of **3a** after purification by a flash chromatography. [d]1.0 equiv of DIB **2a**. [e]Reaction was conducted in the absence of light. [f]Reaction was conducted in the absence of PS.

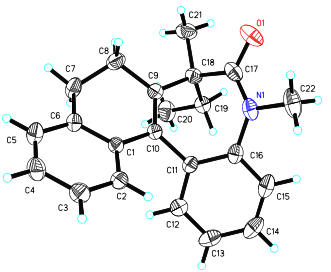


Figure 2. ORTEP drawings of **3a**.

accessed in 92% and 90% yield, respectively. Then the electronic effect of the substrate was examined by changing the substituent R^1 at the *para* position on the benzene ring. When electron-withdrawing substituents were present on the benzene ring (**1d**−**1g**), the desired products **3d**−**3g** were provided in 78−85% yields. For substrate **1h** with an electron-donating substituent on the benzene ring, the corresponding product **3h** was obtained in 87% yield, suggesting that the electronic property of R^1 did not have a significant influence on the product yield. For substrate **1i** having a *meta*-substituted methoxy group on the benzene ring, the corresponding product **3i** was obtained in 82% yield as a single regioisomer. However, the reaction of the *ortho*-substituted substrate **1j** failed to afford the product **3j**, presumably due to steric effects. By introducing a substituent at another benzene ring, for

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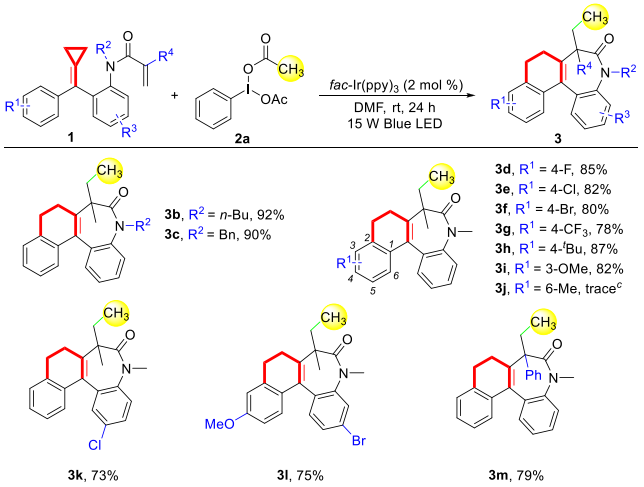
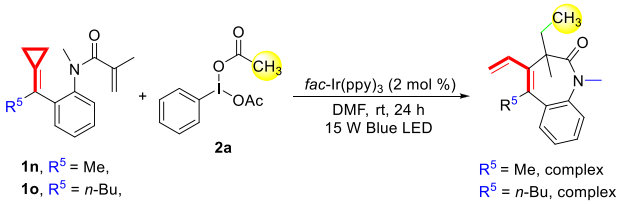
Scheme 2. Substrate scope of acrylamide-tethered alkylidenecyclopropanes 1[a,b]



[a]Unless otherwise specified, all reactions were conducted using **1** (0.2 mmol), **2a** (0.4 mmol, 2.0 equiv), and *fac*-Ir(ppy)$_3$ (2 mol %) in DMF (1.0 mL) at room temperature under nitrogen atmosphere for 24 h. [b]Isolated yields. [c]The desired product was obtained in trace amounts.

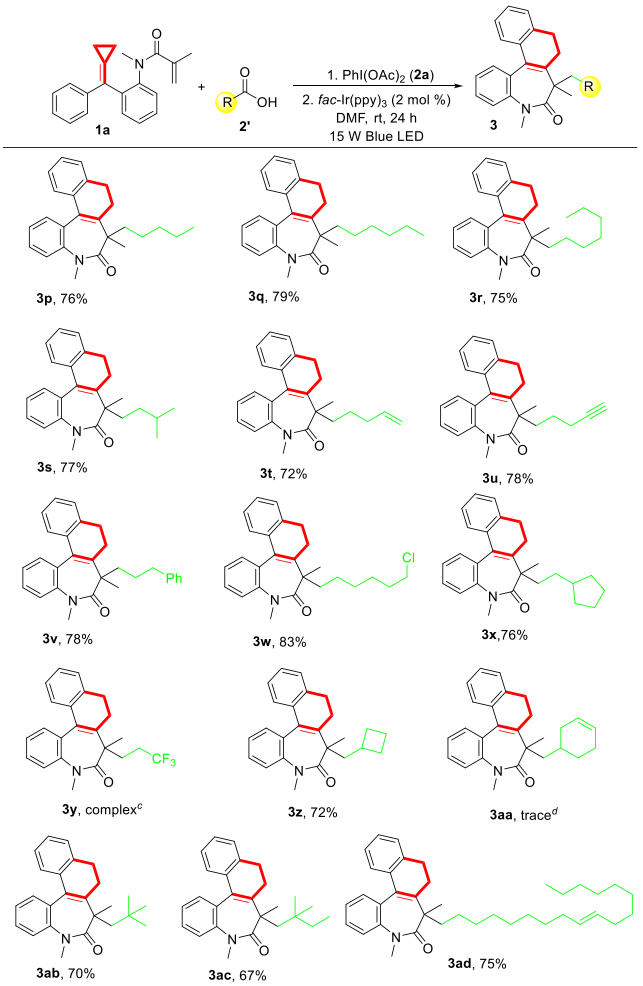
example, when R^3 was a chlorine atom, the desired product **3k** was acquired in 73% yield. Furthermore, substrate **1l** having two substituted benzene rings underwent this reaction, delivering the product **3l** in 75% yield. Using substrate **1m**, in which the methyl group was substituted by a phenyl group, the reaction also proceeded smoothly to give the product **3m** in 79% yield. To further examine the generality of this protocol, we utilized substrates **1n** and **1o**, in which the aromatic moiety was substituted by a methyl or *n*-butyl group. However, only complex mixtures were obtained under the standard conditions (Scheme 3).

Scheme 3. Reactions of Substrates 1n or 1o with 2a



We next focused on examination of the compatibility of the reaction employing a variety of phenyliodine(III) dicarboxylates **2**, and the results are shown in Scheme 4. The ligand metathesis of carboxylic acids **2′** and DIB (**2a**) could easily take place, resulting in the formation of phenyliodine(III) dicarboxylates **2**, which could be used for this reaction without further purification. As shown in Scheme 4, valeric acid, caproic acid found in milk fat,[9] and heptanoic acid were compatible in this reaction, generating the products **3p**, **3q**, and **3r** in the range of 75−79% yields. Moreover, by employing *β*-monosubstituted carboxylic acid, **3s** could be accessed in 77% yield. Notably, primary aliphatic carboxylic acids bearing alkenyl, alkynyl, phenyl, chloro, and cyclopentyl groups were suitable for this reaction, furnishing the corresponding products **3t**−**3x** in good yields ranging from 72% to 83%. The reaction of **1a** with secondary carboxylic acid provided the

Scheme 4. Substrate Scope of Acrylamide-Tethered Alkylidenecyclopropanes 1[a,b]



[a]Unless otherwise specified, all reactions were conducted using **1a** (0.2 mmol), **2′** (4.0 equiv), PhI(OAc)$_2$ (2.0 equiv), and *fac*-Ir(ppy)$_3$ (2 mol %) in DMF (1.0 mL) at room temperature under nitrogen atmosphere for 24 h. [b]Isolated yields. [c]The desired product was obtained in a complex mixture. [d]The desired product was obtained in trace amounts.

targeted product **3z** in 72% yield. However, the use of 3,3,3-trifluoropropionic acid resulted in a complex mixture, probably due to the influence of the electronic effect. For 3-cyclohexene-1-carboxylic acid, the reaction afforded the desired product **3aa** in trace amounts under the optimized reaction conditions. Gratifying, in the case of tertiary carboxylic acids, the reactions proceeded smoothly to deliver the desired products **3ab** and **3ac** in 70% and 67% yields, respectively. The reaction also tolerated naturally occurring carboxylic acids such as oleic acid, delivering the desired product **3ad** in 75% yield.

To demonstrate its synthetic usefulness, a scale-up experiment was carried out with 0.61 g (2.0 mmol) of **1a** and 4-pentynoic acid **2s′** (0.785 g, 8.0 mmol), affording the corresponding product **3u** in 70% yield (497.7 mg) under the standard conditions, testifying this reaction's scalability (Scheme 5). Furthermore, oxidation of **3u** with DDQ furnished the aromatization product **4a** in 98% yield. The copper-catalyzed click reaction of **3u** with tosyl azide afforded the corresponding [3 + 2] cycloaddition product **4b** in 95%

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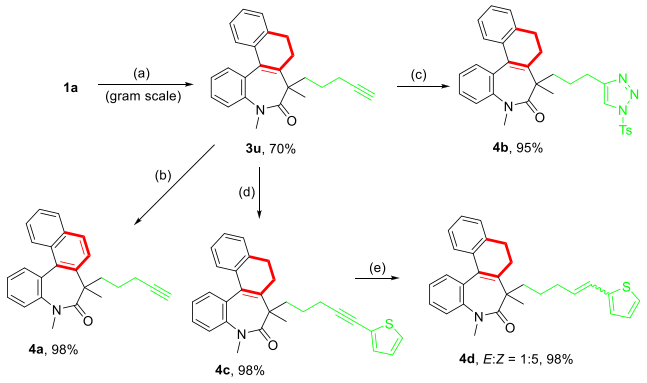
Scheme 5. Scale-up Experiment and Synthetic Usefulness

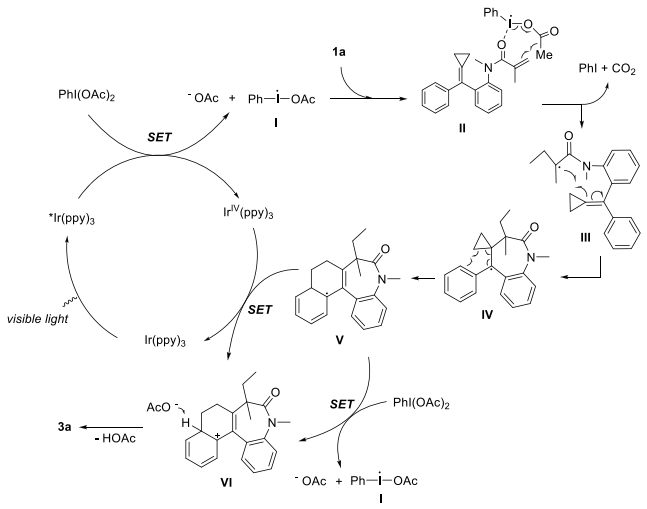


[a]**1a** (2.0 mmol), 4-pentynoic acid **2s′** (8.0 mmol, 4.0 equiv), $PhI(OAc)_2$ (4.0 mmol, 2.0 equiv), *fac*-$Ir(ppy)_3$ (2 mol %), DMF (10 mL), rt, N_2, 24 h, 15 W Blue LED. [b]DDQ, THF, rt, overnight. [c]TsN_3, CuTc, PhMe, rt, 8 h. [d]$Pd(PPh_3)_2Cl_2$, CuI, $^iPr_2NH$, THF, 40 °C, 8 h. [e]Lindlar catalyst, MeOH, rt, H_2.

yield, indicating the potential utility of the resulting alkyne in the medicinal chemistry. Finally, Sonogashira cross-coupling of **3u** with 2-iodothiophene also proceeded smoothly, giving product **4c** in 98% yield, which could be further partially hydrogenated by Lindlar catalyst, affording product **4d** in 98% yield as an E/Z = 1:5 isomeric mixture.

On the basis of previously reported literature and our own examinations,[5d] a plausible mechanism is shown in Scheme 6.

Scheme 6. Plausible Mechanism for the Formation of 3a



Initially, the excited-state $*Ir(ppy)_3$ upon irradiation of $Ir(ppy)_3$ with visible light went through a single-electron transfer (SET) with $PhI(OAc)_2$, generating $Ir^{IV}(ppy)_3$ and the radical intermediate **I**,[5b,10] which interacted with the carbonyl group in **1a** to give an oxygen-coordinated iodine radical species **II**. When CO_2 and PhI were released, a methyl radical was generated. The methyl radical added to **1a** to give radical intermediate **III**, which subsequently added to the less hindered carbon center of alkylidenecyclopropane to generate a radical intermediate **IV**. The ring-opening step took place to furnish a cyclohexadienyl radical intermediate **V**, which underwent another SET with $Ir^{IV}(ppy)_3$ to provide the corresponding cationic intermediate **VI**. Alternatively, the cationic intermediate **VI** could be also formed by a SET of radical intermediate **V** with $PhI(OAc)_2$. Finally, the desired product **3a** was produced upon deprotonation of **VI** with ^-OAc.

In conclusion, we presented a practical and useful protocol for the synthesis of polycyclic benzazepine derivatives by the use of visible-light photoredox catalysis through fast coupling of readily accessible alkylidenecyclopropanes with a series of commercially available aliphatic carboxylic acids in the presence of phenyliodine(III) diacetate. This decarboxylative tandem carbocyclization reaction proceeded effectively through a radical addition initiated cyclopropane ring-opening and cyclization to give the polycyclic products in high yields with broad substrate scope and good functional group tolerance. Moreover, this reaction could be conducted in a gram scale, and the products could be further functionalized to deliver interesting compounds. Efforts are underway to further understand the scope and limitations for this tandem reaction. In addition, the applications of this protocol to biologically interesting compounds are being studied in our lab.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c01856.

Experimental procedures and characterization data for all compounds (PDF)

Accession Codes

CCDC 1937021 contains 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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