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Light Triggered Addition/Annulation of 2-Isocyanobiphenyls toward 6-Trifluoromethyl-Phenanthridines under Photocatalysts-free Conditions

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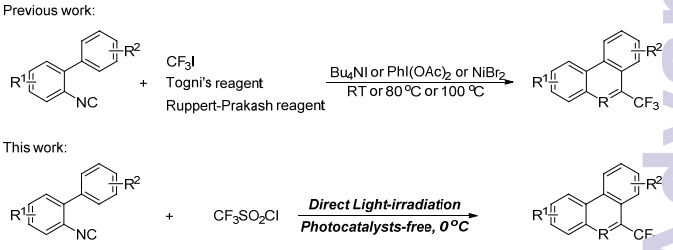
A photocatalyst-free, light promoted sequential radical addition/annulation of 2-isocyanobiphenyls to 6-trifluoromethyl phenanthridines is presented. Wide substrate scopes and scale-up experiment demonstrate the promising efficiency and utility of this strategy.

Isonitriles are privileged motifs in organic synthesis because of their vast potential for the synthetic versatility, which have aroused intensive and continuing interest from the chemists.[1] Recently, the radical addition/intramolecular homolytic aromatic substitution (HAS) reactions of 2-isocyanobiphenyls have been extensively investigated. By such a cascade procedure, a rapid assembly of 6-substituted phenanthridine frameworks is accomplished with high synthetic efficiency and atomic economy. To date, various types of radical precursors including boronic acids,[2] halides,[3] aldehydes,[4] diphenylphosphine oxide,[5] *a*-oxocarboxylic acids,[6] silanes,[7] peroxide,[8] amides,[9] ethers,[10] alkanes,[11] hydrazines,[12] and carbazates[13] have been gradually explored for such transformations and a myriad of phenanthridine scaffolds with diverse functional groups have been successfully produced. Due to the wide applications in pharmaceutical science and drug research of phenanthridines and their derivatives,[14] the synthesis 6-trifluoromethyl phenanthridines may be of great significance, and their potential biological activities may be profoundly enhanced to some extent. Because the incorporation of trifluoromethyl group can alert the physicochemical properties of the parent compounds, such as electronegativity, hydrophobicity, metabolic stability, and bioavailability compared to their non-trifluoromethylated counterparts.[15] Although various methods leading to 6-trifluoromethyl phenanthridines have emerged,[16] there are only three examples involving the somophilic isocyanide insertion by the electrophilic CF_3 radical (Scheme 1). In 2013, Studer and co-workers reported a novel Bu_4NI-catalyzed formation of 6-trifluoromethyl phenanthridines by using Togni's reagent as the precursor of CF_3 radical in 1,4-dioxane at 80 °C.[17] Almost simultaneously, Zhou *et al.* described a $PhI(OAc)_2$-mediated oxidative cyclization of 2-isocyanobiphenyls with CF_3SiMe_3 in combination with BQ as the additives.[18] In 2014, the Studer group again made an advance on finding a $NiBr_2$ assisted and CF_3I participated facile synthesis of 6-trifluoromethyl phenanthridines.[19] Despite of the high effectiveness for the above strategies, some disadvantages are existed, such as the use of expensive CF_3 reagents and environmentally unfriendly oxidants, the relatively high temperature, which restrict their practical applications to some extent. Therefore, the development of a mild, economic, environmentally benign and efficient alternative to realize the direct construction of 6-trifluoromethyl phenanthridines via the radical addition followed by HAS process is still an urgent need.



Scheme 1 The cascade addition/annulation of 2-isocyanobiphenyls to 6-trifluoromethyl phenanthridines.

Typically, the CF_3 radical can be generated in three ways: the reduction, oxidation and homolysis of CF_3 reagents using reducing agents, oxidizing agents and light-excitation or at high temperature or using initiators. The former two are preferred methods for the trifluoromethylation of a wide range of interesting scaffolds,[20] while the reactions involving the homolysis of CF_3 reagents are still

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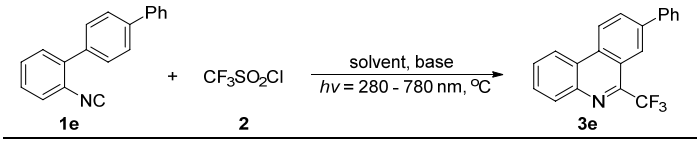
limited.[21] Recently, we have investigated the light-induced BiOBr nanosheets accelerated trifluoromethylation/arylation of *N*-aryl acrylamide substrates employing CF_3SO_2Cl as CF_3 source.[22] It is observed that small amount of the product is still formed without the addition of BiOBr photosensitizer. We reason this result may be ascribed to the homolysis of CF_3SO_2Cl under light-excitation generating the CF_3 radical that engages this tandem transformation. This speculation inspired us to take advantage of these unexpected CF_3 radicals, produced from the direct light-irradiation of CF_3SO_2Cl, for the trifluoromethylation of organic molecules. Isonitriles are isoelectronic with carbon monoxide, which can act as well-established C-radical acceptors. Herein, we wish to disclose our recent endeavor in light-mediated sequential arylation/trifluoromethylation of 2-isocyanobiphenyls to synthesize 6-trifluoromethyl-phenanthridines. Compared with the aforementioned protocols on the synthesis of 6-trifluoromethyl-phenanthridines, this procedure own three unique features: (1) the use of easily available and high active CF_3SO_2Cl as trifluoromethylating reagent; (2) direct light-induced transformation under photocatalyst-free conditions; (3) gram-scale synthesis. This promising and green chemical transformation constitutes an important complementary to the current light-driven organic reactions.[23]

Table 1 Optimization of reaction conditions.[a]



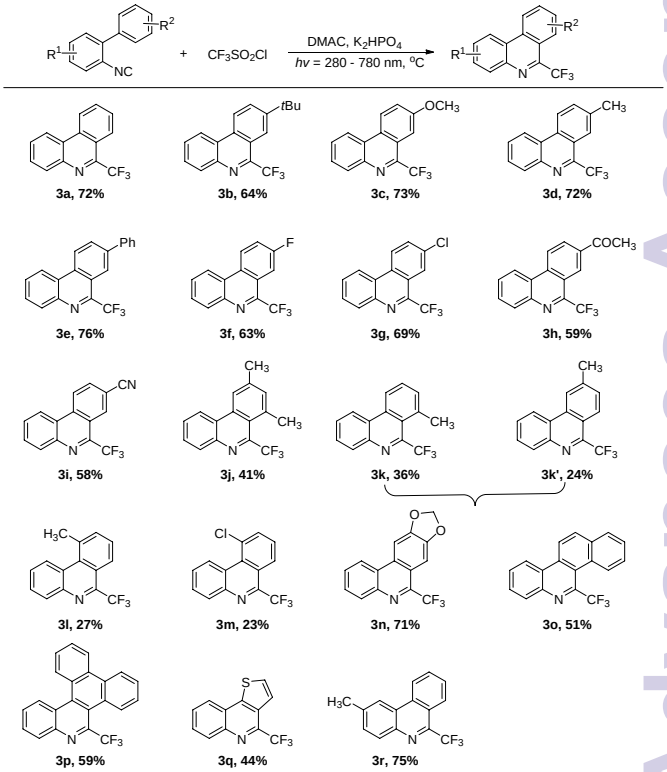
1e 2 3e

Entry	Solvent	Base	Yield (%)[b]
1	DMF	K_2HPO_4	33
2	DMAC	K_2HPO_4	76
3	DMSO	K_2HPO_4	N.R.[c]
4	CH_3CN	K_2HPO_4	23
5	NMP	K_2HPO_4	N.R.[c]
6	EtOAc	K_2HPO_4	43
7	CH_3OH	K_2HPO_4	N.R.[c]
8	THF	K_2HPO_4	N.R.[c]
9	CH_2Cl_2	K_2HPO_4	Trace
10	Toluene	K_2HPO_4	N.R.[c]
11	DMAC	K_2CO_3	68
12	DMAC	K_3PO_4	57
13	DMAC	KOH	67
14	DMAC	*t*BuOK	49
15	DMAC	Et_3N	34
16	DMAC	-	30
17[d]	DMAC	K_2HPO_4	N.R.[c]

[a] reaction conditions: **1a** (0.2 mmol), **2** CF_3SO_2Cl (0.6 mmol), solvent (1.0 mL), base (0.6 mmol), 300 W Xe lamp, 0 °C. [b] isolated yield is reported. [c] no reaction. [d] in the dark.

The light-induced isonitriles insertion reaction is initiated by treating 2-isocyanobiphenyl (**1e**) and CF_3SO_2Cl (**2**) with the addition of K_2HPO_4 in DMF under light irradiation (280-780 nm) for 6 h. As expected, this reaction indeed occur, and 33% yield of the desired product is obtained (Table 1, entry 1). Delightedly, the yield of **3e** dramatically increased to 76% when the reaction proceeds in DMAC (Table 1, entry 2). Encouraged by this exciting result, we further screen the other solvents including the polar/non-polar and protonic/non-protonic solvents, and observe that the others are inferior to DMAC, or even useless (Table 1, entries 3-10), which indicates a significant solvent effect of this radical addition/annulation of 2-isocyanobiphenyls. Additionally, examination of bases reveals that this one-pot tandem reaction proceeds more effectively in the presence of inorganic base, and K_2HPO_4 is the optimal one (Table 1, entries 11-15). However, the reaction efficiency is remarkablely decreased when K_2HPO_4 is removed from the reaction media, demonstrates that the base plays a stimulative rol for the creation of trifluoromethylated phenanthridines (Table 1, entry 16). In order to identify the effective wavelength range for this reaction clearly, the UV-visible absorption test of CF_3SO_2Cl in DMAC is conducted (Fig. S1 in ESI). From the spectrogram we can see that the optimal wavelength absorption range of CF_3SO_2Cl is approximately between 270-350 nm. It means that only this wavelength range is effective for the activation of CF_3SO_2Cl, and the rest section that from 350 nm to 780 nm is almost useless. Noting that, the reaction does not occur in the dark, showing that the CF_3 radical is indeed generated by light illumination, and that is the light that triggers this cascade transformation (Table 1, entry 17).

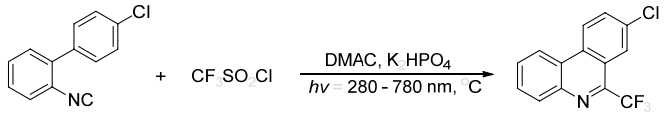
Table 2 Substrate scope of the light triggered direct arylation/trifluoromethylation of 2-aryl Isonitriles.[a]



[a] reaction conditions: **1a** (0.2 mmol), **2** CF_3SO_2Cl (0.6 mmol), DMAC (1.0 mL), K_2HPO_4 (0.6 mmol), 300 W Xe arclamp, 0 °C.

With the optimized reaction conditions in hand, the substrate scope is evaluated to test the universality, as listed in Table 2. Firstly, we investigate the influence of R^2 on the nonisonitriles phenyl rings on the reaction efficiency. The substrates bearing the electron-rich or electron-deficient groups at *para*-position could all work

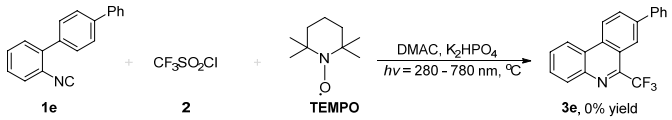
smoothly to deliver the corresponding products in middle to good yields (Table 2, **3a**-**i**). Noting that, even the potentially sensitive functional groups acetyl and cyano are both tolerated, and 59% and 58% yields of the phenanthridines derivatives are obtained (Table, **3h** and **3i**). The existence of acetyl and cyano gives an oppotunity for further transformations by transition-metal catalyzed strategies or other technologies. In contrast, the *ortho*- and *meta*-substituted 2-isocyanobiphenyls suppress the reaction and a relatively low yields of the products are formed (Table 2, **3j**-**m**). When *meta*-methyl substituted substrate is used, two regioisomers **3k** and **3k'** are formed with a ratio of 1.5: 1. The dioxy heterocyclic group functionalized isocyanide is also a good candidate, and under the present reaction conditions we mainly get one isomer compound as the final product with the isolated yield of 71% (Table 2, **3n**). The obtained molecule **3n** containing a trisphaeridine framwork, which can function as DNA intercalator.[14a,c] According to the significant characteristics of the trifluoromethyl-containing compounds, the biological activity of the synthesized trifluoromethylated trisphaeridine may be obviously improved. When the aromatic ring is replaced with 2-naphthyl, 9-phenanthrene and electron-deficient 2-thienyl moiety, this radical addition/cyclization reaction still occur, giving rise to the desired products **3o**-**q** in acceptable yields. Next, the substrate with a methyl group on the aromatic ring of the isocyano group is amenable to the selected reaction conditions and 75% yield of the corresponding product is fabricated (Table 2, **3r**).



1g (1.2 mmol) **2** (3.6 mmol) **3g** (219.2 mg, 65%)

Scheme 2 Scaled-up experiment.

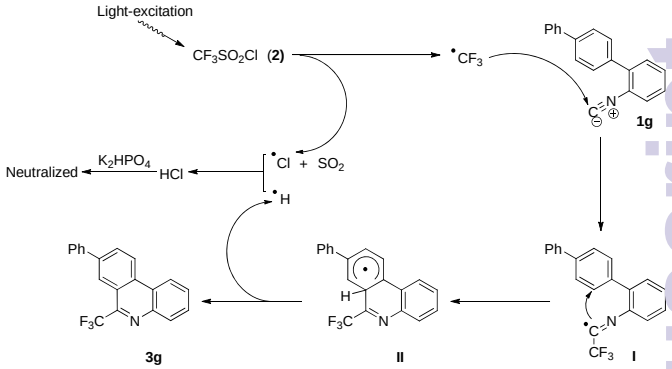
To highlight the utility of this photochemical protocol, an amplifying reaction of 1.2 mmol of **1g** with 3.6 mmol CF_3SO_2Cl is conducted under the standard conditions (Scheme 2). A comparable yield of the product **3g** with that of 0.2 mmol scale experiment is afforded, hints the potential applicability of our method.



1e **2** **TEMPO** **3e**, 0% yield

Scheme 3 Control experiment.

To confirm that this tandem transformation is originated from the addition of CF_3 radical to the terminal carbon of isocyanide, the control experiment is carried out. Adding radical trapping agent, 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO), to the reaction system under otherwise identical conditions leads to no desired product, which supports that a radical pathway is involved in this radical addition/annulation of 2-isocyanobiphenyls (Scheme 3). This process is distinguished from Yu's work that discloses a concise and efficient synthetic approach to 6-(trifluoromethyl)phenanthridine through the ionic isocyanide insertion with no use of catalyst.[24]



Scheme 4 Proposed mechanism for the radical isocyanide insertion of 2-aryl isonitriles with CF_3SO_2Cl.

Based on the above observations and the previous reports,[2-13] a plausible mechanism is presented (Scheme 4). Homolysis of CF_3SO_2Cl by photoexcitation generates CF_3 radical with the release of SO_2 and Cl radical. Subsequent addition of CF_3 radical to the terminal carbon of isocyanide affords the imidoyl radical **I**, which experiences a intramolecular homolytic aromatic substitution to give the cyclized radical intermediate **II**. The product is finally created with the leave of H radical, which is abstracted by Cl radical to get HCl. The formed HCl can be neutralized by K_2HPO_4 at the end.

In conclusion, we have developed a direct light-initiated one-pot consecutive radical addition/annulation of 2-isocyanobiphenyls using cheap and high active CF_3SO_2Cl as the precursor of CF_3 radical under photocatalysts-free conditions. With this method, a variety of the corresponding 6-trifluoromethyl-phenanthridines derivatives bear diverse functional groups are constructed in acceptable to good yields. The scale-up experiment illustrates the potential applicability of our methodology. The simple, economic and environmentally friendly procedure may be applied to the future industrial processes. Further mechanistic researches and more light-induced cascade reactions for molecules with structural complexity are underway in our lab.

Acknomledgement

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