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A facile access for multisubstituted trifluoromethyl olefins by visible light catalysis

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ABSTRACT

Induced by visible light, a green approach for the highly substituted trifluoromethyl olefins has been developed by using alkyl boronic acid as a carbon radical precursor and the Boc-modified trifluoromethylated Baylis-Hillman adduct (BMTBHA) as an acceptor. This method starts from 1,1-disubstituted olefins without the external stoichiometric oxidant.

Introduction

The introduction of fluorine atoms into the molecular skeleton can greatly change the physical and chemical properties of organic compounds and polymers [1]. The fluorinated molecules occupy an increasingly important position in drug molecules due to their excellent chemical stability and lipophilicity [2]. However, it's rare to find fluorinated molecules in natural products, and most of fluorine-containing drugs must be synthesized artificially. Trifluoromethyl olefins are not only a class of important drug structural units, but also important substrates for C-F activation [3]. In the past 10 years, the synthesis of trifluoromethyl olefins has aroused great interest. Normally, cross-coupling is a major way for the construction of vinyl-CF_3 [4], which heavily relies on the use of prefunctionalized olefins. And the commonly used trifluoromethyl reagents such as Umemoto's reagent, Togni's reagent, CF_3 surrogates and etc. have high price with poor atom economy [5].

Recently, some works have been focused on bifunctionalization of alkynes and direct trifluoromethylation of olefinic C—H bonds to form highly substituted trifluoromethyl olefins [6], in which both transition metal catalysts and stoichiometric strong oxidants are necessary. Debien et al. proposed the synthesis of multisubstituted trifluoromethyl olefins by the carbon–oxygen bond homolysis in 2012 [7], however, the preparation of highly functional substrate was troublesome and bulky leaving group led low atom economy, which strongly limits its application in organic synthesis (Scheme 1). Concerning the importance of trifluoromethyl olefins, developing greener and more easily operable method for accessing such compound is still in urgent need.

Visible-light mediated photocatalysis has emerged as a powerful synthetic tool in organic chemistry for the last decade [8]. Its unique performance has realized a variety of chemical transformations, and plays an irreplaceable role in the construction of special organic compounds which could not be accomplished by traditional methods. The types of visible-light-catalyzed reactions include amine oxidation [9], organic boron oxidation [10], reduction dehalogenation [11], double bond reduction [12], ATRP reaction [13], and etc. Especially, the formation of α-amino carbon radical by visible light catalytic oxidation and the formation of C—C and C—B bond by the visible light catalytic oxidation of organic boron reagent are typical representatives. Recently we published several results about the applications of Baylis-Hillman adducts in the field of visible light catalysis [14]. With the continuous interest in the photocatalysis and the importance of trifluoromethyl olefins, the Boc-modified trifluoromethylated Baylis-Hillman adduct **1** (BMTBHA) was prepared and subjected for study. BMTBHA could be obtained easily from a stable and commonly used industrial feedstock 2-bromo-3,3,3-trifluoroprop-1-ene (BTP) and aldehyde promoted by zinc powder [15] and then protected by $(Boc)_2O$. Considering the particularity of trifluoromethyl group and its stronger electron withdrawing capacity, we proposed that **1** is more vulnerable to be competitive nucleophilic attacked between leaving group and coupling partner than traditional Baylis-Hillman adducts. And the selectivity of defluorination and

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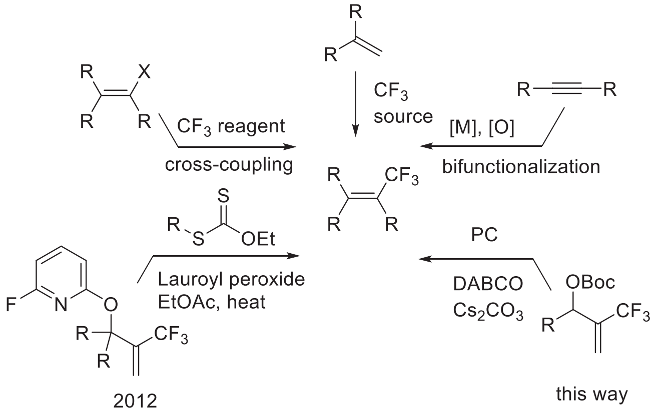
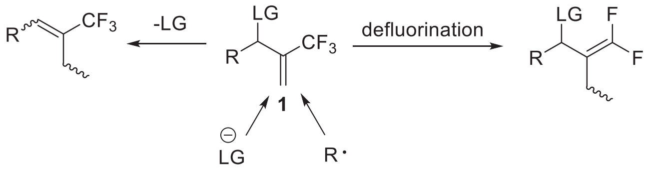


Scheme 1. General ways for trifluoromethyl olefins.



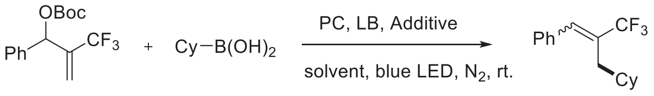
Scheme 2. Two possible reaction paths of **1**.

leaving group's removal is unclear because of the formation of α-trifluoromethyl carbanion [16]. Recently, our group has explored the coupling α-trifluoromethyl aryl olefins with boronic acid through photocatalysis/Lewis base dual catalysis [17]. Upon checking the literatures, there were few researches [18] on the synthesis of trifluoromethyl olefins via double bond transfer, and there have no literatures reporting the synthesis of highly substituted trifluoromethyl olefins by photocatalysis (Scheme 2).

Result and discussion

In order to realize the viability of the reaction, a model reaction employing *tert*-butyl (1-phenyl-2-(trifluoromethyl)allyl) carbonate (**1a**, 0.5 mmol), cyclohexylboronic acid (**2a**, 0.75 mmol), DABCO (0.125 mmol), PC1 (1.0 mol %), and DCE (6 mL) was carried out in a Schlenk tube with 7 W blue LED. Delightfully, the desired **3a** was isolated in 49% yield within 5 h (Table 1, entry 1). Inspired by this result, photocatalyst, solvent, and Lewis base were then investigated. Only activated alkyl boronic acid could be oxidized by excited PC1. The solvents such as DCM, THF, DMF, NMP, and DMSO were tried and the results were negative, compared with that of DCE (entries 4–9). A brief screen of bases revealed that DMAP, DBU, DBN, PPh_3 (entries 10–13) are not as effective as that of DABCO, although there was still more than 39% of **1a** left (entry 1). Considering that boronic acid could be transformed to borate ether or borate ester in methanol [19], which were easily combined with Lewis base, different volumes of methanol were added into

Table 1
Optimization of the reaction conditions.[a]



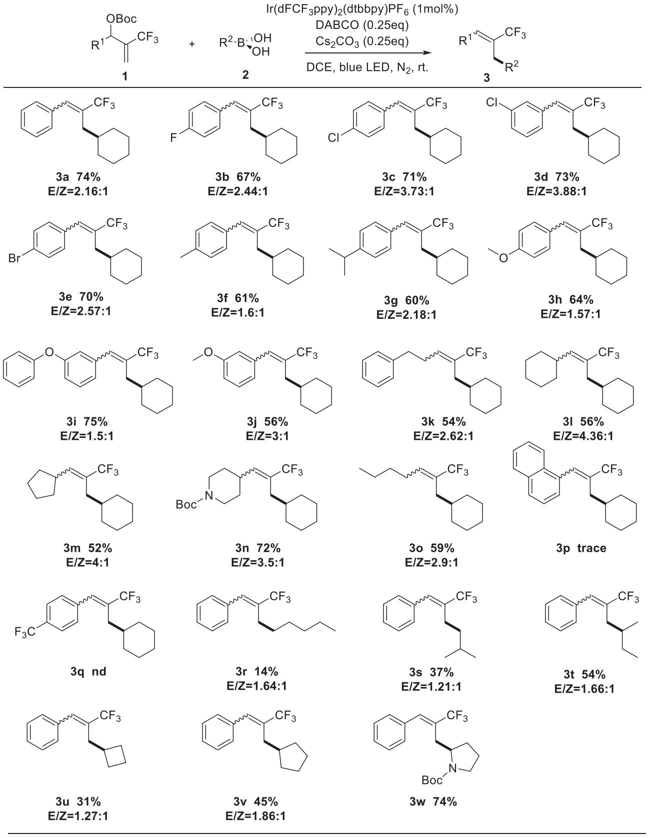
1a **2a** **3a**

Entry	Catalyst	Lewis base	Additive	Solvent	Yield(%)[a]
1	PC1	DABCO	–	DCE	49(30:19)
2	PC2	DABCO	–	DCE	0
3	PC3	DABCO	–	DCE	0
4	PC1	DABCO	–	DCM	24(15:19)
5	PC1	DABCO	–	THF	25(16:9)
6	PC1	DABCO	–	MeCN	7(5:2)
7	PC1	DABCO	–	NMP	23(13:10)
8	PC1	DABCO	–	DMF	11(7:4)
9	PC1	DABCO	–	DMSO	19(13:6)
10	PC1	DMAP	–	DCE	0
11	PC1	DBU	–	DCE	17(10:7)
12	PC1	DBN	–	DCE	14(8:6)
13	PC1	PPh_3	–	DCE	0
14	PC1	DABCO	–	DCE:MeOH(1:1)	Trace
15	PC1	DABCO	–	DCE:MeOH(4:1)	Trace
16	PC1	DABCO	–	DCE:MeOH(9:1)	Trace
17[b]	PC1	DABCO	Na_2CO_3	DCE	50(31:19)
18[b]	PC1	DABCO	K_2CO_3	DCE	73(45:28)
19[b]	PC1	DABCO	Cs_2CO_3	DCE	82(54:28)(74)[c](2.16:1)
20	–	DABCO	–	DCE	0
21	PC1	–	–	DCE	0
22[d]	PC1	DABCO	–	DCE	0

[a] General condition: 1a (0.5 mmol), 2a (0.75 mmol), PC1(1 mol%), Lewis base (0.125 mmol), solvent (6 mL), 7 W blue LED, N_2 atmosphere, PC1 = $Ir(dFCF_3ppy)_2(dtbbpy)PF_6$, PC2 = $Ir(ppy)_2(dtbbpy)PF_6$ PC3 = $Ir(ppy)_3$, the E/Z ratio was determined by GC.
[b] 0.125 mmol (0.25 eq) of additive.
[c] Isolated yield.
[d] In the dark.

Table 2

Scope of the present photocatalytic method for multisubstituted trifluoromethyl olefins.[a]

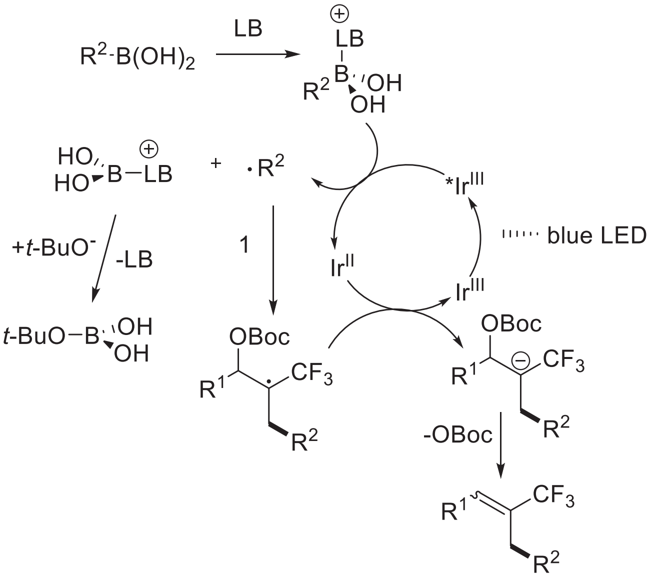


3a 74% E/Z=2.16:1
3b 67% E/Z=2.44:1
3c 71% E/Z=3.73:1
3d 73% E/Z=3.88:1
3e 70% E/Z=2.57:1
3f 61% E/Z=1.6:1
3g 60% E/Z=2.18:1
3h 64% E/Z=1.57:1
3i 75% E/Z=1.5:1
3j 56% E/Z=3:1
3k 54% E/Z=2.62:1
3l 56% E/Z=4.36:1
3m 52% E/Z=4:1
3n 72% E/Z=3.5:1
3o 59% E/Z=2.9:1
3p trace
3q nd
3r 14% E/Z=1.64:1
3s 37% E/Z=1.21:1
3t 54% E/Z=1.66:1
3u 31% E/Z=1.27:1
3v 45% E/Z=1.86:1
3w 74%

[a] General condition: 1a (0.5 mmol), 2a (0.75 mmol), PC (1 mol%), Lewis base (0.125 mmol), solvent (6 mL), 7 W blue LED, N2 atmosphere, the E/Z ratio was determined by NMR analysis of the product.

DCE. Unexpectedly, the reaction was inhibited by even a very small amount of methanol (entries 14–16), which was quite different from the literature's report. We inferred the possibility is that *tert*-butoxide anion on the one hand combined with boronic acid to regenerate DABCO, and on the other hand was protonated by the residual water in the system to form *tert*-butyl alcohol after CO_2 removal from BocO$^-$. No further improvement was achieved with increased equivalent of DABCO and boronic acid. Furthermore, the inorganic bases carbonates were surveyed. **1a** could be totally transformed to **3a** in 74% yield when 0.25 eq Cs_2CO_3 was added. since Cs_2CO_3 is almost insoluble in DCE, the reactions with Cs_2CO_3 in other solvents were studied and it can be concluded that the reaction results in larger polar solvents such as DMSO, DMF, NMP, MeCN, acetone were not as good as it in DCE. The presence of photocatalyst, visible light and Lewis base was essential to the reaction (entries 20–22). The by-product of defluorination or the competitive nucleophilic attack of *tert*-butoxide anion was not observed. The acetyl instead of Boc-modified trifluoromethylated Baylis-Hillman adduct was not effective in this reaction. Based on this and the previous reports, [14] it can conclude that BMTBHA is completely different from conventional Baylis-Hillman adducts from the reaction characteristics and activity. The Z/E configuration was determined by 2D NOE spectrum (See the Supporting Information for details).

Under the optimized conditions, the generality and scope of substrates were subjected to this reaction. Various Structurally different BMTBHA could react smoothly with cyclohexyl boronic acid to afford the target products (**3b**-**3e**) in high yields (Table 2). However, BMTBHA bearing *p*-CF_3 group on the aromatic ring (**3q**) was unreactive under the standard condition. The presence of electron-donating groups on the aromatic ring of BMTBHA were benefit for the reaction and gave moderate yields. Only trace amount of product was obtained from the substrate containing naphthyl (**3p**). The yield of aliphatic BMTBHA substrate (**3o**) was moderate. Then different substituted boronic acids were explored. The yields were slight lower when primary alkyl boronic acids were selected as candidates (**3r**-**3s**), which has great relationship with the stability of the primary carbon radical. When the cyclic alkyl boronic acids such as cyclobutyl, cyclopentyl, cyclohexyl boronic acid (**3a**,**3u**-**3v**) were used, the yields of corresponding products were increased. Tetrahydropyrrole 2-borate protected by *N*-Boc (**3w**) was suitable for the reaction and only *E*-isomer was produced, which could be the result of steric effect and the stabilization effect of nitrogen atom on ortho carbon radicals. Unfortunately, aromatic boronic acid was unreactive under the standard conditions, and the addition of methanol also could not change the result.



Scheme 3. A plausible reaction mechanism.

In order to further understand the reaction mechanism, 3 eq of Tempo was added into the reaction system. No product was detected by TLC, and the peak of tempo-cyclohexyl couldn't be found in GC–MS, which may be contributed to the inhibition of catalytic cycle in the presence of excessive Tempo. Based on the literatures [10,19] and our above experiments, the reaction mechanism is hypothesized. Firstly, boronic acid is activated by Lewis base and oxidized by the excited PC1 under the irradiation of blue LEDs. Then the generated free radical goes through the nucleophilic addition to BMTBHA, and the radical intermediate is further reduced by the PC1. Finally, the product generates after leaving the OBoc group (Scheme 3).

Conclusions

In conclusion, we have developed a facile method for the synthesis of trifluoromethyl olefins by visible light catalysis. Compared with other methods, this method has the following advantages: (1) it can react at normal temperature and pressure with good substrate tolerance, and the driving power of reaction is green and easily available visible light. (2) Only small amounts of Lewis base and inorganic base are needed.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data (copies of ^{1}H NMR, ^{13}C NMR spectra of all products) to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.152829.

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