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## Synthesis of remote fluoroalkenyl ketones by photo-induced ring-opening addition of cyclic alkoxy radicals to fluorinated alkenes†

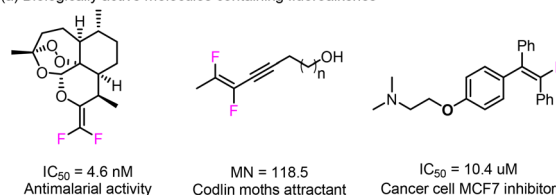
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Fluoroalkenyl moieties are often used as carbonyl mimics in medicine preparation, and thus the development of facile routes for the synthesis of such compounds is of great importance. In this work, we report a photocatalytic ring-opening addition of cyclic alcohols to  $\alpha$ -(trifluoromethyl)styrenes, which underwent a proton-coupled electron transfer and  $\beta$ -scission process, delivering a great variety of remote *gem*-difluoroalkenyl ketone derivatives. This methodology can also be applied in the reaction of *gem*-difluorostyrenes and 1,1,2-trifluorostyrenes to access monofluoro- and 1,2-difluoroalkenyl ketones.

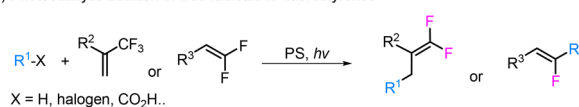
Fluoroalkenes are important building blocks in many pesticides and pharmaceuticals.<sup>1–5</sup> As potential bioisosteres of carbonyl groups, the introduction of fluoroalkenyl units into organic molecules can influence their stabilities, physicochemical properties and biological activities to a great extent, and thus improve their pharmaceutical performance. For example, when the carbonyl group of artemisinin was replaced by a *gem*-difluoroalkenyl substituent, its antimalarial activity ( $IC_{50}$ ) improved from 8.9 nM to 4.6 nM.<sup>6</sup> Also 1,2-difluoroalkenyl codlemone elicited stronger flight attraction of the codling moth (MN: 118.5) than the non-fluorinated codlemone analogue (MN: 82.5).<sup>7</sup> Besides, monofluoroalkenyl-decorated tamoxifen exhibited enhanced growth inhibition activity in the MCF7 cancer cell line (Scheme 1a).<sup>8</sup> Therefore, the development of facile routes for the synthesis of fluoroalkenes has gained increasing attention in recent years and various strategies for the construction of these molecules have been reported.<sup>9–12</sup>

Photo-redox catalysis associated with free radical addition and fluorine anion elimination represents one of the most efficient routes to multifariously structured fluoroalkenes. The  $S_N2'$  type of defluorinative addition–elimination reaction of  $\alpha$ -(trifluoromethyl) styrenes with various free radicals generated under visible light which could lead to *gem*-difluoroalkenes has been well studied in the past decades.<sup>13–21</sup> The *gem*-

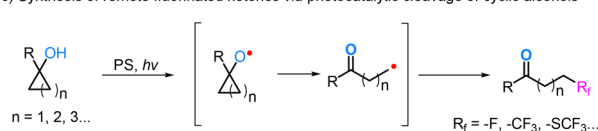
## (a) Biologically active molecules containing fluoroalkenes



## (b) Photocatalytic addition of free radicals to fluorostyrenes

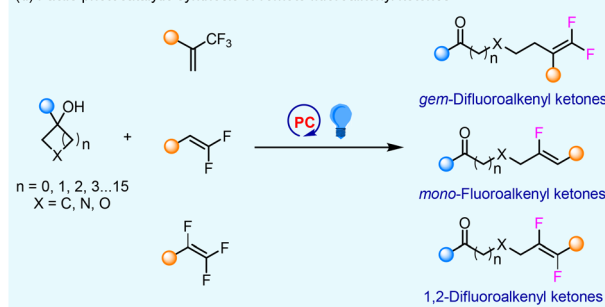


## (c) Synthesis of remote fluorinated ketones via photocatalytic cleavage of cyclic alcohols



## This work

## (d) Facile photocatalytic synthesis of remote fluoroalkenyl ketones



Scheme 1 Visible-light-induced synthetic strategies for fluoroalkenes.

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difluoroalkenes can be further converted to monofluoroalkenes through the same process (Scheme 1b).<sup>22–29</sup>

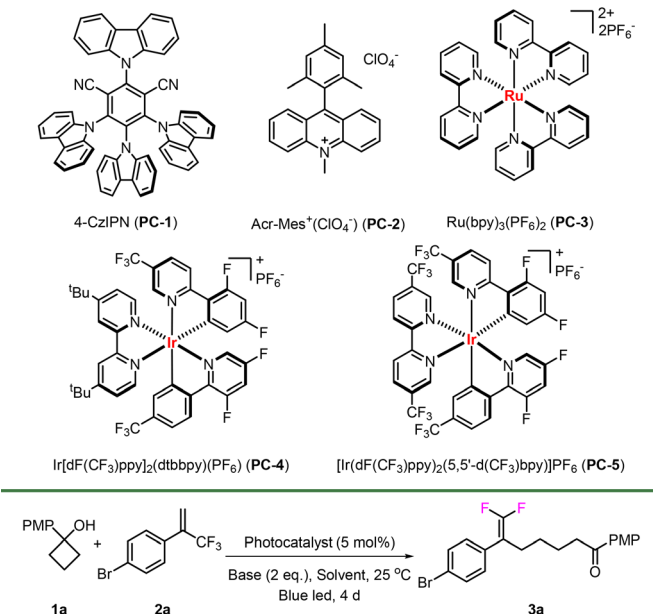
On the other hand, since ketones are common functional groups in natural products, drugs, and agrochemicals, the development of facile methodologies for their elaboration is extremely important. With years of efforts, the synthesis of common ketones has been well established;<sup>30</sup> in contrast, remote functionalized ketone construction still remains a great challenge.<sup>31</sup>

Cyclic alkoxy radicals can undergo cleavage of C–C bonds to furnish remote carbonyl carbon radical species, which act as reactive intermediates in various further transformations.<sup>32,33</sup> Among them, visible-light-induced ring-opening addition of cyclic alcohols has been widely used in the construction of remote functionalized ketones (Scheme 1c).<sup>34–38</sup> To date, various fluorinated ketones with terminal –F, –CF<sub>3</sub>, and –SCF<sub>3</sub> have been synthesized through this strategy;<sup>39,40</sup> however, studies focused on fluoroalkenyl ketones are rare, and in this context, Wang reported a nickel-catalyzed fluoroalkenyl ketone synthesis with cycloalkyl silyl peroxides as alkoxy radical precursors.<sup>41</sup>

Given the importance of diverse fluoroalkenyl-containing carbonyl compounds, we herein report the photocatalytic ring-opening radical addition of cyclic alcohols to  $\alpha$ -(trifluoromethyl) styrenes,<sup>42</sup> *gem*-difluorostyrenes and 1,1,2-trifluorostyrenes, which led to a great variety of remote *gem*-difluoroalkenyl, monofluoroalkenyl and 1,2-difluoroalkenyl ketones in high yields (Scheme 1d).

Initial studies were carried out with cyclopropanol **1a** and  $\alpha$ -(trifluoromethyl)styrene **2a** as model substrates. First, we tried various photosensitizers which are widely used in photoredox catalysis, with 2 eq. collidine as the base and 1,2-dichloroethane (DCE) as the solvent. As shown in Table 1, under 100 W blue LED irradiation, the organic dye 4-CzIPN (**PC-1**) gave the desired product **3a** in 88% yield (Table 1, entry 1). Another organic photocatalyst Acr-Mes<sup>+</sup>ClO<sub>4</sub><sup>–</sup> (**PC-2**), which has been proven to be an efficient photocatalyst for cyclic alkoxy radical generation,<sup>43</sup> showed poor results (13% yield) in this reaction (Table 1, entry 2). Noble-metal-based photoredox catalysts Ru(bpy)<sub>3</sub>(PF<sub>6</sub>)<sub>2</sub> (**PC-3**) and Ir[dF(CF<sub>3</sub>)ppy]<sub>2</sub>(dtpy)(PF<sub>6</sub>) (**PC-4**) did not work in this transformation (Table 1, entries 3 and 4). In the presence of 2% Ir[dF(CF<sub>3</sub>)ppy]<sub>2</sub>(5,5'-d(CF<sub>3</sub>)bpy)]PF<sub>6</sub> (**PC-5**), the ring-opening addition process proceeded and gave **3a** in 56% yield (Table 1, entry 5). Next, various solvents were screened, and dramatically decreased yields were observed in the case of toluene, tetrahydrofuran (THF), and *N,N*-dimethylformamide (DMF), while dimethyl sulfoxide (DMSO) gave 75% yield (Table 1, entries 6–9). Switching collidine with inorganic bases K<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>HPO<sub>4</sub> led to significantly declined yields (21% and 15%) which may be due to their poor solubility in DCE (Table 1, entries 10 and 11). Light sources with shorter wavelengths such as 420 nm and 365 nm gave poor results than a 456 nm blue LED (Table 1, entries 13 and 14). Using lower light power (48 W), a white LED (100 W) gave much lower yields (Table 1, entries 12 and 16). No product was observed in the dark. These results revealed that this reaction

**Table 1** Optimization of the reaction conditions<sup>a,b,c</sup>



Entry	PC	Solvent	Base	Light source	Yield <sup>b</sup> (%)
1	<b>PC-1</b>	DCE	Collidine	456 nm 100 W	88
2	<b>PC-2</b>	DCE	Collidine	456 nm 100 W	13
3 <sup>c</sup>	<b>PC-3</b>	DCE	Collidine	456 nm 100 W	0
4 <sup>c</sup>	<b>PC-4</b>	DCE	Collidine	456 nm 100 W	0
5 <sup>c</sup>	<b>PC-5</b>	DCE	Collidine	456 nm 100 W	56
6	<b>PC-1</b>	Toluene	Collidine	456 nm 100 W	12
7	<b>PC-1</b>	THF	Collidine	456 nm 100 W	5
8	<b>PC-1</b>	DMF	Collidine	456 nm 100 W	21
9	<b>PC-1</b>	DMSO	Collidine	456 nm 100 W	75
10	<b>PC-1</b>	DCE	K <sub>2</sub> CO <sub>3</sub>	456 nm 100 W	21
11	<b>PC-1</b>	DCE	K <sub>2</sub> HPO <sub>4</sub>	456 nm 100 W	15
12	<b>PC-1</b>	DCE	Collidine	456 nm 48 W	25
13	<b>PC-1</b>	DCE	Collidine	420 nm 100 W	32
14	<b>PC-1</b>	DCE	Collidine	365 nm 100 W	19
15	<b>PC-1</b>	DCE	Collidine	White LED 100 W	20
16	<b>PC-1</b>	DCE	Collidine	Dark	0

<sup>a</sup> All reactions were carried out with 0.20 mmol of **1a** and 0.30 mmol of **2a** in 2 mL of solvent under a N<sub>2</sub> atmosphere. <sup>b</sup> Yields are given as isolated yields. <sup>c</sup> 2 mol% photocatalyst was used.

is induced by light and high light power is crucial for the free radical generation step.

Next, under the optimal reaction conditions, we investigated the substrate applicability of this catalytic system, and the results are summarized in Table 2. First, we tested various substituted  $\alpha$ -(trifluoromethyl)styrenes. As shown, various substituted remote *gem*-difluoroalkenyl ketones can be prepared by this method.

Other halogen substituents such as fluoride and chloride were tolerated under these reaction conditions, providing the corresponding products **3b–3d** in good yields (61%, 89%, and 75%). Neutral phenyl product **3e** was also obtained in 70% yield. The steric effect of substituents showed slight influence on the reactivity, and *ortho*- and *para*-Me substituted products **3f** and **3h** were obtained in similar yields (84% and 86%);

Table 2 Substrate scope of cyclic alcohols and  $\alpha$ -(trifluoromethyl)styrenes<sup>a,b,c</sup>

<b>Scope of <math>\alpha</math>-CF<sub>3</sub> styrenes</b>				
<b>Scope of cyclic alcohols</b>				

<sup>a</sup> All reactions were carried out with 0.20 mmol of **1** and 0.30 mmol of **2** in 2 mL of DCE under a N<sub>2</sub> atmosphere. <sup>b</sup> These reactions were carried out in the presence of 5 mol% **PC-1**. <sup>c</sup> These reactions were carried out in the presence of 2 mol% **PC-5**.

however, *meta*-Me and 3,5-diMe substituted products **3g** and **3i** were obtained in low yields (62% and 67%), which may be due to the electron donating effect of the methyl group which could enhance the ability of radical acceptance because of the hyperconjugation effect when it is at the *ortho*- and *para*-positions. Electronic properties affect the reactivity a lot, and substrates bearing the strongly electron donating OMe group at the *para* position gave **3j** in 30% yield. However, product **3k** with two OMe groups at 3,5-positions could be obtained in 74% yield. Then we investigated substrates bearing electron withdrawing substituents, which performed very well in this reaction, for example, **3l** with *para*-CF<sub>3</sub> was obtained in 61% yield, while substrates bearing strongly electron withdrawing groups such as CN and CO<sub>2</sub>Me participated in this transformation and led to products **3m** and **3n** in 79% and 83% yields, respectively. Heterocyclic pyrrole can be used in this reaction, although a protecting group (Boc) is needed, and the desired compound **3o** was produced in 62% yield.

Encouraged by these results, we next investigated the substrate scope of cyclic alcohols, and it was found that 4-CzIPN (**PC-1**) did not perform well in these cases. Delightfully, in the presence of 2 mol% Ir[(dF(CF<sub>3</sub>)ppy)<sub>2</sub>(5,5'-d(CF<sub>3</sub>)bpy)]PF<sub>6</sub> (**PC-5**), all cyclic alcohols reacted smoothly and gave the corresponding products **4a–4f** in moderate to high yields (46%–92%).

Heterocycles also tolerated these reaction conditions, and furyl and thienyl ketones **4g** and **4h** were produced in 75% and 61% yields. Besides, cyclopropanol **1i** was well developed in such a transformation and gave **4i** in good yield (64%). Encouraged by the promising results of cyclobutanes, larger sized cyclic alcohols, which are difficult to undergo ring-opening reactions due to weaker ring tension, were investigated.<sup>44</sup> Cyclohexanol and cycloheptanol can also be used as substrates in this reaction which led to the corresponding products **4j** and **4k** in high yields (84% and 82%). Large cycles such as 12- and 15-membered rings can also be used as good

participants in the reaction to access remote *gem*-difluoroalkenyl ketones **4l** and **4m** in good yields (74% and 87%). Finally, adamantanol was used in this strategy to construct the unique remote bridged fluoroketone **4n** in 53% yield.

Besides carbocyclic alcohols, heterocyclic alcohols are also compatible in this reaction. As summarized in Table 3,<sup>45</sup> five types of heterocyclic alcohols were tested with 2 mol% **PC-5** as the photocatalyst. To our delight, these reactions showed higher reactivity under the same conditions, and all reactions proceeded smoothly and were complete within 2 d. Cyclobutylamino alcohol **1o** performed very well to furnish **4o** in 82% yield. Asymmetrical tetrahydrofuran alcohol **1p** was a good candidate in this reaction and underwent C–C cleavage at the O-site, providing **4p** as the only product. This could be because the O atom could stabilize the O–CH<sub>2</sub>• radical through the conjugate effect. Similar results were obtained in the case of hexahydropyran alcohol **1q**, which gave **4q** in 87% yield and >20:1 regioselectivity. Piperidyl substituted substrates showed the same reactivity to access **4r** and **4s** in 93% and 96% yields. In order to prove the utilization of this methodology, an  $\alpha$ -(trifluoromethyl)styrene-decorated cholesterol was prepared and subjected to the optimal reaction conditions,

giving the corresponding product **4t** in 53% yield.

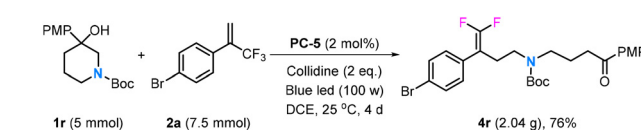
After the investigation of substrate compatibility, we next tested the practicality of this methodology, and a gram-scale synthesis of **4r** was carried out under the optimal conditions (Scheme 2). The reaction of 5 mmol of heterocyclic alcohol **1r** with 7.5 mmol of  $\alpha$ -trifluoromethylstyrene **2a** worked very well and delivered 2.04 g of product **4r** in 76% yield.

In order to prove that the remote carbonyl free radical is a key intermediate in this photocatalytic transformation, a radical capture experiment was conducted with 2 eq. of TEMPO as the free radical scavenger. Under these conditions, the corresponding product **3a** was not observed at all, while the carbonyl alkyl-TEMPO adduct was detected by HRMS (Scheme 3). These results indicated that a remote carbonyl terminal radical was formed through  $\beta$ -scission of the cyclic alkoxy radical. On the basis of these results and previous investigations,<sup>46</sup> a catalytic mechanism is proposed in Scheme 4. First, photocatalyst **PC** was excited by visible light to form excited state **PC\***, and then single electron transfer (SET) occurred between **PC\*** and cyclic alcohol **1a** to generate alkoxy radical **Int.I** and **PC<sup>•-</sup>** with low valence. Then, the alkoxy

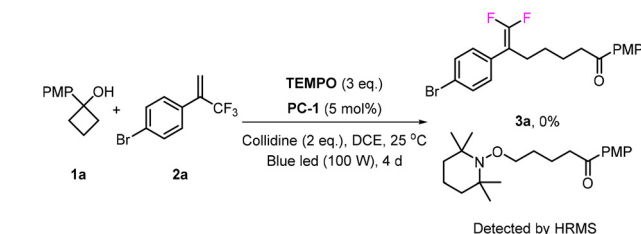
**Table 3** Ring opening addition of heterocyclic alcohols<sup>a,b</sup>

Entry	Substrates	Products
1		
2		
3		
4		
5		
6		

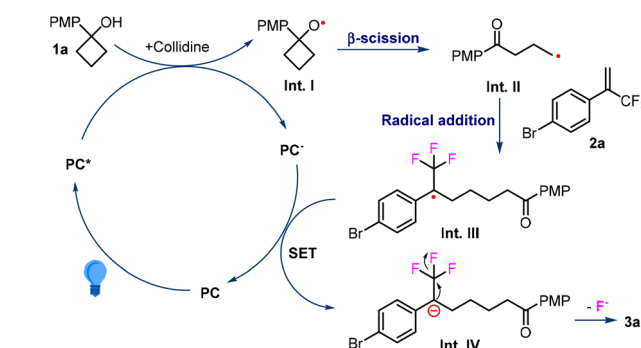
<sup>a</sup> All reactions were carried out with 0.20 mmol of **1** and 0.30 mmol of **2a** in 2 mL of DCE for 2 days. <sup>b</sup> Yields are given as isolated yields.



**Scheme 2** Gram scale reaction.



**Scheme 3** Radical trapping experiment.



**Scheme 4** Proposed catalytic mechanism.

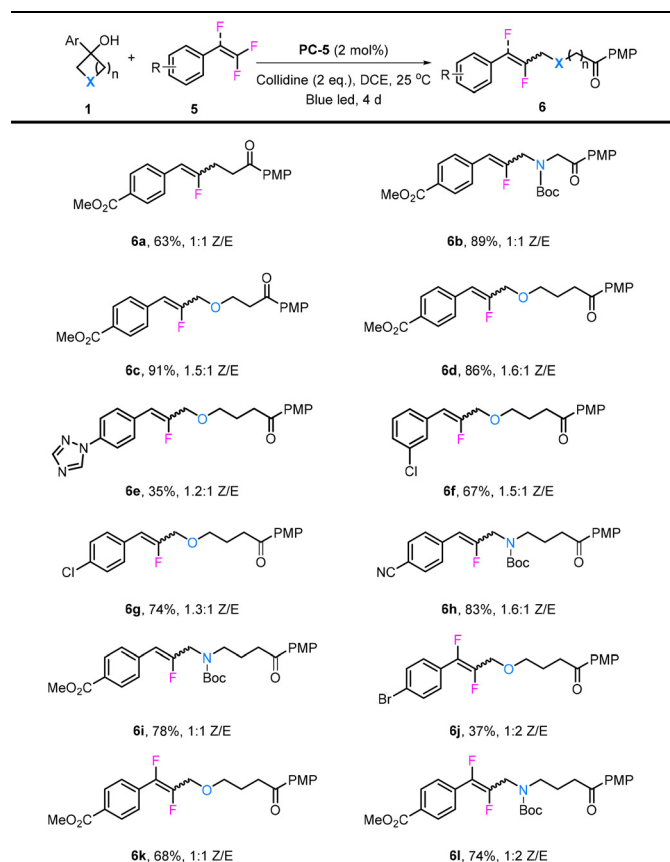
radical converted to the remote carbonyl terminal radical species **Int.II** through  $\beta$ -scission, which could add to  $\alpha$ -trifluoromethylstyrene **2a** to furnish the radical adduct **Int.III**. Finally, the intermediate got one electron from **PC<sup>-</sup>** to furnish **Int.IV** and then  $F^-$  elimination occurred to give the final product **3a**.

Besides  $\alpha$ -trifluoromethylstyrenes, *gem*-difluoro-styrenes and 1,1,2-trifluorostyrenes could also participate in various photocatalytic radical transformations to access monofluorostyrene and 1,2-difluorostyrene derivatives. In our recent work, we achieved an asymmetric 1,3-dipolar cycloaddition of azomethine ylides with *gem*-difluorostyrenes and 1,1,2-trifluorostyrenes, which led to a series of chiral antifungal bioactive fluoro tetrahydropyrroles.<sup>47</sup> On the basis of this work, we applied the methodology to the synthesis of remote monofluoro- and 1,2-difluoroalkenyl ketones, which are also important building blocks in bioactive compounds. The results are shown in Table 4. Under the standard reaction conditions, various *gem*-difluorostyrenes were tested in this reaction. Fluorostyrenes bearing electron-withdrawing-groups such as  $-CO_2Me$  and  $-CN$  showed high reactivity, giving the desired products **6a**, **6b**, **6c**, **6d**, **6h**, and **6i** in high yields (63%–91%) although with subequal quantum of *Z* and *E* isomers

(1 : 1–1.6 : 1). The reaction of hexahydropyranyl alcohol **1q** with *para*-1,2,4-triazole-*gem*-difluorostyrene exhibited poor reactivity, leading to the corresponding monofluoroalkenyl ketone **6e** in 35% yield and with 1.2 : 1 *Z/E*. Additionally, monofluoroalkenyl ketone **6f** and **6g** bearing a chloride atom at the *meta*- and *para*-positions could be produced in 67% and 74% yields. This strategy is also suitable for the reaction of 1,1,2-trifluorostyrenes and the results are similar to those of *gem*-difluorostyrenes. Remote 1,2-difluoroalkenyl ketones **6k–6l** bearing  $-CO_2Me$  at the *para*-position were produced in high yields (68%–74%) with a 1 : 1–1.2 : 2 *Z/E* ratio, while the bromo-substituted product **6j** was obtained in only 37% yield.

In conclusion, we have developed a visible light induced ring opening of cyclic alcohols and radical addition to  $\alpha$ -trifluoromethylstyrenes, *gem*-difluoro-styrenes and 1,1,2-trifluorostyrenes, delivering a variety of remote fluoroalkenyl ketones, which are important building blocks in biologically active natural compounds such as pharmaceuticals and pesticides. This methodology features mild conditions, high efficiency, easily accessible substrates, wide substrate scope, and excellent functional group tolerance, and thus could serve as a facile route for the synthesis of unique remote fluoroalkenyl ketones.

**Table 4** Synthesis of monofluoro- and 1,2-difluoroalkenyl ketones<sup>a,b</sup>



<sup>a</sup> All reactions were carried out with 0.20 mmol of **1** and 0.30 mmol of **5** in 2 mL of DCE for 4 days. <sup>b</sup> Yields were given as isolated yields.

## Conflicts of interest

There are no conflicts to declare.

## Acknowledgements

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