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Transition-metal free trifluoromethylimination of alkenes enabled by direct activation of *N*-unprotected ketimines

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ABSTRACT

A highly site-selective intermolecular trifluoromethylimination of activated and unactivated olefins was reported under transition-metal- and photosensitizer-free conditions. This newly developed strategy provides straightforward and efficient access to diverse value-added vicinal trifluoromethyl amines without resorting to the pre-functionalized reagents. Mechanistic experiments demonstrate that the approach proceeded through CF_3 and iminyl two-radicals process, which were generated directly from commercially available benzophenone imine in a novel electron-donor mode via a SET process activated by the bifunctional hypervalent iodine reagents. The synthetic potential of the protocols was further showcased *via* the condensation/amination sequential cascade, and transformations to access β-CF_3 primary amines.

Nitrogen-containing organic compounds have been widely found in ubiquitous pharmaceuticals, biologically active natural products and synthetic compounds, such as dyes, pesticides, functional materials [1]. The introduction of a CF_3 group leads to changes in the chemical and physical properties of potential drug candidates, owing to its strong electron-withdrawing nature, high lipophilicity, and metabolic stability [2]. During the past several decades, many CF_3-containing pharmaceuticals [3] and agrochemicals, such as Prevacid, Prozac, fluazinam, and norflurazon, have been developed. So far, a number of methods have been developed to incorporate a CF_3 group into organic molecules [4–14], with a particular emphasis on the amine-containing functional molecules. In addition, β-trifluoromethyl primary amines are also the valuable structural skeletons of ubiquitous biologically active molecules [15,16]. Accordingly, the synthetic methods to access vicinal trifluoromethyl amines from simple and easily available raw materials have been of great interest to chemists. In this regard, the trifluoromethylamination of alkenes represents straightforward routes to an array of such useful scaffolds, given the facile accessibility of alkene starting materials and quickly increasing molecular complexity in a single step [17–20]. Among these protocols, several two-components intermolecular methods were independently developed to generate trifluoromethylated aziridines, pyrazolines and pyrrolidines [21–24]. In addition, the existing three-components intermolecular approaches either afforded trifluoromethylated amides and anilines *via* Ritter-type processes through β-trifluoromethyl carbenium ions [25–26], or alternatively gave trifluoromethylated azides [27–34] *via* transition-metal or photoredox catalyzed azidotrifluoromethylation of alkenes (Scheme 1a, top).

In recent years, N-centered radicals have received considerable attention from the synthetic community because these powerful species offer great opportunities for the construction of C–N bonds [35–42]. Among various developed nitrogen-centered radicals, the ambiphilic iminyl radicals have received considerable attention recently, which were generated by the cleavage of N–O bond of oxime derivatives *via* an electron-acceptor heterolytic pathway [43–46] or photoinduced energy-transfer homolytic manner [47–54]. Recently, the Molander group [55] and Yang group [56] disclosed two elegant examples on photochemical alkene trifluoromethylamination using prefunctionalized oxime esters or sulfonamides as linked N- & C-centered radicals precursors, respectively, to generate both CF_3 and iminyl radicals *via* an energy-transfer fragmentation process (Scheme 1a, bottom).

Despite the precedents have been realized in this area, the synthetic utility was often limited due to the need for the expensive transition-metal catalysts or photocatalysts and extra

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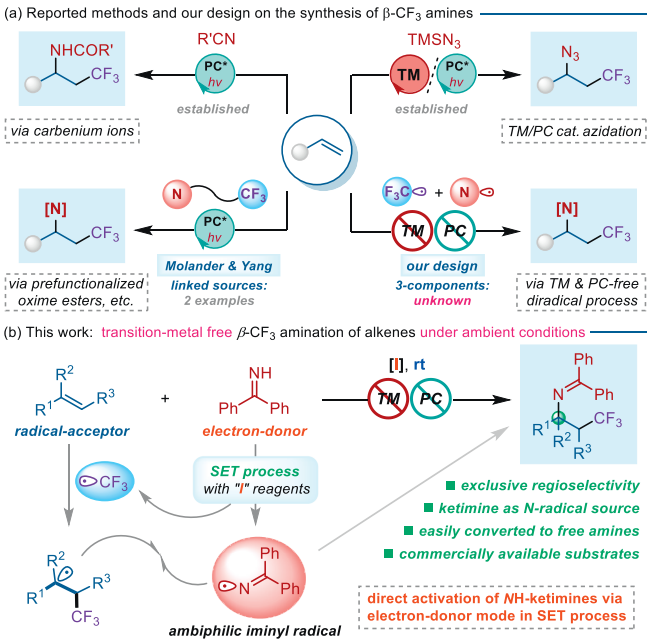
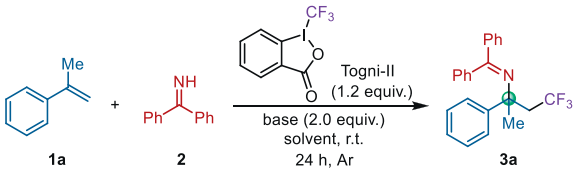


Scheme 1. Trifluoromethylamination of alkenes with benzophenone imine.

photochemical ancillaries, requiring the circumscribed use of the functionalized substrates prior prepared in several steps. As such, new strategies to modularly access vicinal trifluoromethyl amines from commercially available starting materials under transition-metal- and photosensitizer-free conditions remain highly desirable and appears to be practical. Inspired by our ongoing sustainable chemical research on the synthesis of nitrogen-containing compounds [57–60], we envisioned that a three-components site-selective trifluoromethylimination of alkenes could be developed under transition-metal free conditions at room temperature directly from commercially available benzophenone imines without the need for substrate pre-functionalization, which were generally utilized as *C*-electrophiles and *N*-nucleophiles in previous studies [61,62]. Moreover, in terms of radical mechanism, either N–H or *N*-alkyl/aryl substituted benzophenone imines were typically employed to generate ketiminyl radical anions *via* electron-acceptor mode (accepting one electron) [63,64], in this current research, they would be innovatively employed as a nitrogen-radical precursor to generate diphenyliminyl radical *via* an electron-donor mode (donating one electron) (Scheme 1b).

To validate our hypothesis, we set out to explore trifluromethylimination of olefins by employing α-methylstyrene **1a** as the acceptor to capture the CF_3 radical and iminyl radical. As our expectation, using bifunctional CF_3-hypervalent iodine reagent (Togni-II) as the trifluoromethyl radical precursor and the activator of unprotected ketimine to iminyl radical [57], Cs_2CO_3 as base, EtOAc as solvent, the reaction delivered the desired trifluoromethylaminative product **3a** in 23% yield through a robust operation at ambient temperature (Table 1, entry 1). Further investigations were performed on commonly inorganic and organic bases such as LiO*t*Bu, NaO*t*Bu, LiOMe and DBU (entries 2–5). It revealed that the reaction was not only dependent on the basic property, but the lithium salts were also essential for this protocol. Other common solvents, for instance, THF, PhCl, dichloromethane and MeCN, were next examined, it found that EtOAc is the optimal one in terms of the efficiency and sustainability (entries 6–9). Of particular note is that the reaction occurred smoothly at higher temperature or using 1.0 equiv. of LiO*t*Bu, giving similar yield (entries 10 and 11). Other CF_3 radical precursors, such

Table 1
Optimization of reaction conditions.[a]



1a 2 3a

Entry	Solvent	Base	Yield (%)[b]
1	EtOAc	Cs_2CO_3	23
2	EtOAc	LiO*t*Bu	70
3	EtOAc	NaO*t*Bu	15
4	EtOAc	LiOMe	65
5	EtOAc	DBU	Trace
6	THF	LiO*t*Bu	24
7	PhCl	LiO*t*Bu	40
8	CH_2Cl_2	LiO*t*Bu	53
9	MeCN	LiO*t*Bu	55
10	EtOAc	LiO*t*Bu	68[c]
11	EtOAc	LiO*t*Bu	70[d]
12	EtOAc	LiO*t*Bu	n.d.[e]
13	EtOAc	LiO*t*Bu	19[f]

EtOAc = Ethyl acetate; THF = Tetrahydrofuran; DBU = 1,8-Diazabicyclo[5.4.0]undec–7-ene.

[a] Reaction conditions: **1a** (0.1 mmol), **2** (0.15 mmol), Togni-II reagent (1.2 equiv.), base (2.0 equiv.) in corresponding solvent (2.0 mL) at r.t., Ar, 15 h.

[b] Determined by ^{19}F NMR analysis of the crude reaction mixture using $PhCF_3$ as an internal standard.

[c] At 50 °C.

[d] With LiO*t*Bu (1.0 equiv.).

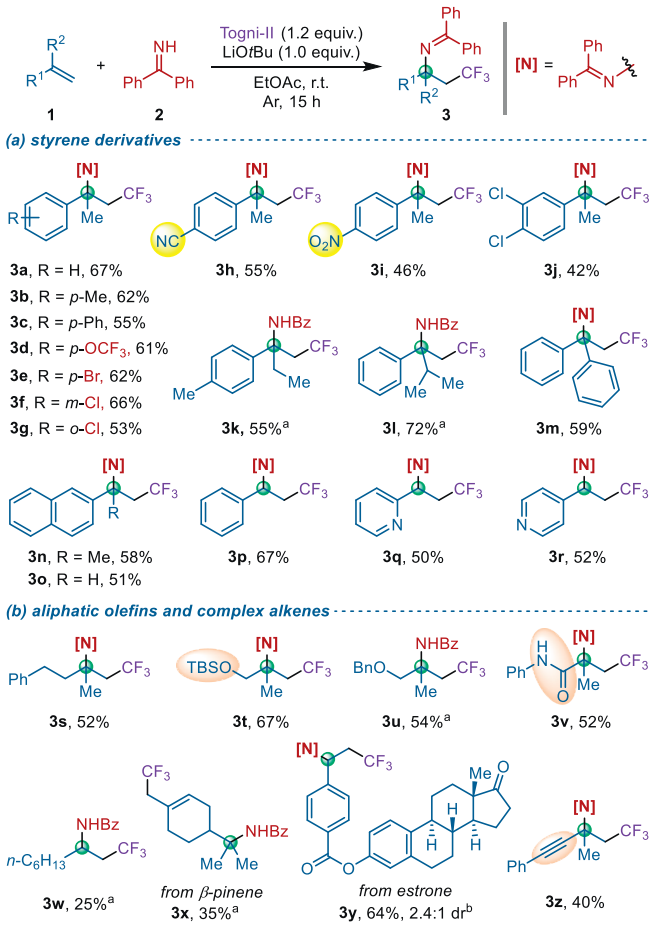
[e] With Togni-I reagent as CF_3 source.

[f] With $TMSCF_3$ (2.0 equiv.), PIDA (2.0 equiv.) and CsF (2.0 equiv.).

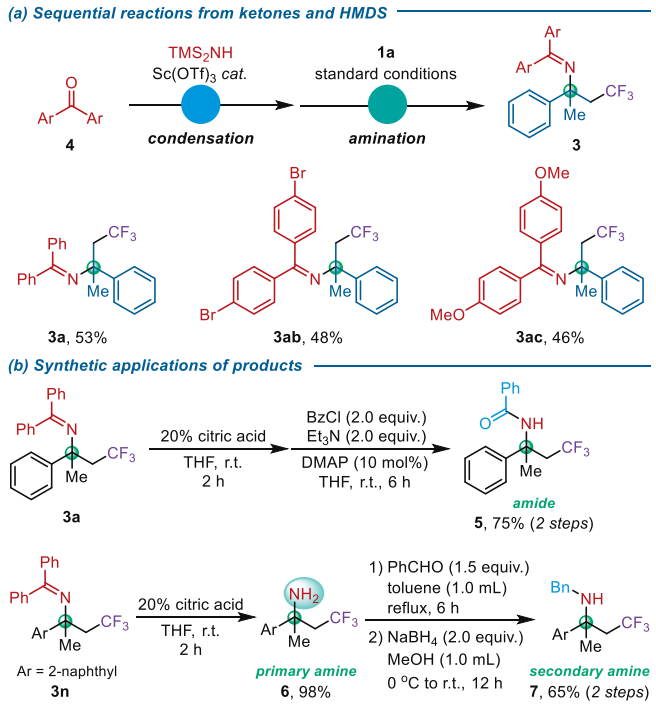
as 1-trifluoromethyl-dimethylbenziodoxole (Togni-I) and (Trifluoromethyl)trimethylsilane ($TMSCF_3$) in combination with (diacetoxyiodo)benzene (PIDA) were further explored, providing no product and 19% yield, respectively (entries 12 and 13).

With the optimal conditions in hand, the generality of this protocol was evaluated with a series of alkenes (Scheme 2). To our delight, a wide range of 1,1-disubstituted styrene derivatives bearing various functional groups were amenable to this transformation. Typically, the regioselectivity of trifluoromethylimination was uniformly exclusive and the desired compounds **3a**-**3j** can be isolated in moderate to good yields, basically independent of any electronic and steric perturbation. It is worth noting that the trifluoromethoxy, chloro, bromo, cyano and nitro groups were tolerated. The styrene bearing α-ethyl, isopropyl and phenyl substituents were viable substrates for the process (**3k**-**3m**). The imine products were relatively sensitive to acidic conditions and readily hydrolyzed to access valuable β-CF_3 primary amines, which could be protected *in-situ* with benzoyl chloride to β-CF_3 amide in high yield (**3k**, **3l**). When 2-isopropenylnaphthalene and monosubstituted (hetero)aryl olefins were used as reaction components, all reactions proceeded smoothly under the mild conditions in good yield varying from 50% to 67% yield (**3n**-**3r**).

To further illustrate the feasibility of this platform, variants of unactivated aliphatic alkenes and several complex alkenes were next surveyed, which proved to be quite productive. A variety of versatile aliphatic units, including long-chain alkyl, *tert*–butyldimethylsilyl (TBS) group and benzyl (Bn) group protected alcohol moieties, even amide unit were compatible in current protocol (**3s**-**3v**). In addition, the reactivity of monosubstituted aliphatic alkene was tested, *e.g.*, with 1-octene as substrate. It also provided the target β-CF_3 amination product **3w** in 25% yield. The relatively low yield may be attributed to the instability of the secondary carbon radical. It is more encouraging to note that the complex olefins derived from natural products, such as β-pinene



Scheme 2. Scope of trifluoromethylamination of olefins. Reaction conditions: **1** (0.2 mmol), **2** (1.5 equiv.) and Togni-II (1.2 equiv.), LiO*t*Bu (1.0 equiv.) in EtOAc (2.0 mL) at r.t., 15 h. [a] The reaction mixture was treated with 20% citric acid (aq., 0.5 mL) at room temperature for 2 h, then the crude free amine was treated with benzoyl chloride (2.0 equiv.) at r.t. for 6 h. [b] Determined by ^{19}F NMR analysis of crude reaction mixture.



Scheme 3. (a) Scope of diverse imines *via* sequential reactions directly from ketones and HMDS. Conditions for condensation: diaryl ketones (0.5 mmol), TMS_2NH (0.6 mmol) and $Sc(OTf)_2$ (5 mol%) in 1,4-dioxane (0.5 mL) at 90 °C, Ar. Standard conditions for the sequential amination with alkene **1a** in 0.2 mmol scale. (b) Synthetic applications of products.

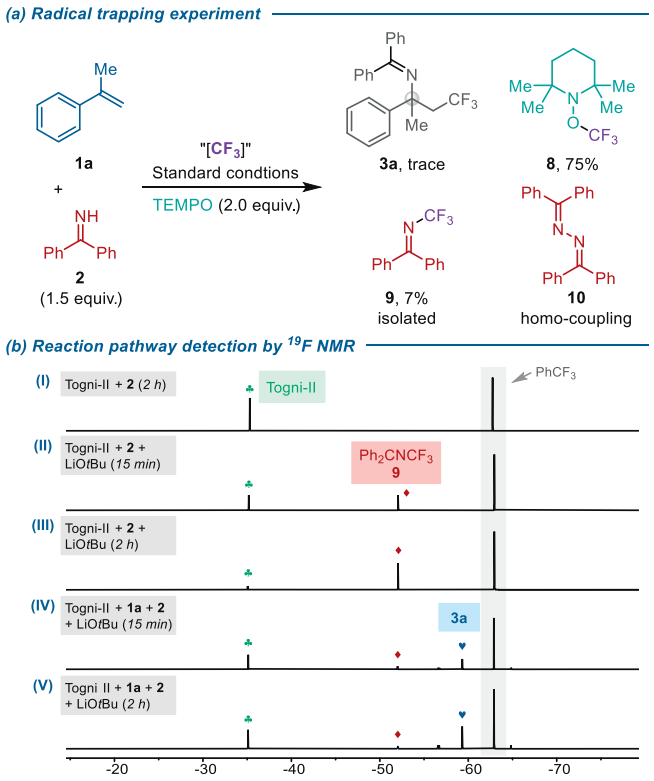
and estrone, reacted effectively (**3x**-**3y**). The ring-opening product **3x** was smoothly formed by the cleavage of 4-member ring of β-pinene, albeit in a slightly diminished yield for the amination/hydrolysis/acylation cascade, demonstrating that a radical pathway is potentially involved in the reaction. When an 1,3-enyne was served as substrate, excellent regioselective addition to the double bond was observed while retaining the triple bond, giving the internal alkyne **3z** in moderate yield.

Referring to previous studies on the preparation of *N*-unprotected ketimines [65,66], we therefore chose the more facilely accessible and relatively stable bis(trimethylsilyl)amine (HMDS) as a nitrogen source to investigate the scope of diverse imines *via* sequential reactions directly from aryl ketones **4** (Scheme 3a). From the point view of reactivity of amination, the *in situ* generated ketimines appears to be an alternative option to be involved in this newly developed approache (as for **3a**: 53% *vs.* 67% in Scheme 2). Other ketimines derived from diaryl ketones bearing various substituents with different electronic properties, were all smoothly compatible and delivered diverse fancy structures **3ab** and **3ac** in moderate yields, which would greatly extend the applicability of this amination method. However, when using benzaldehyde and TMS_2NH as the precursors of aldehyde-derived imine to explore the sequential reaction under the standard conditions, the reaction only gave the target product in 14% NMR yield, which was further detected by gas chromatography-mass spectrometry (see Supporting information).

To demonstrate the synthetic potential of the β-trifluoromethyl imination products, several transformations were carried out (Scheme 3b). To our delight, the imine groups of products was readily hydrolyzed after a simple work-up procedure with citric acid to afford highly value-added β-CF_3 primary amines (for **6** in 98% yield) [67,68], which could further undergo *in-situ* protections to amide **5** by benzoyl chloride and to dialkylated amine **7** *via* reductive amination process in high efficiency.

In order to gain insights into this difunctionalization mechanism, several control experiments were performed. The addition of 2,2,6,6-tetramethylpiperidinooxy (TEMPO) as a free radical scavenger significantly interfered with this process, with no product **3a** being formed, whereas the TEMPO–CF_3 adduct **8** was generated in 75% yield. In this process, radical-radical cross-coupled product, CF_3 substituted benzophenone imine **9**, was evidently isolated, and the byproduct **10** from homo-coupling of iminyl radical was also detected by gas chromatography-mass spectrometry (Scheme 4a). These observations suggest a free radical mechanism seems reasonable, consistent with the previous results obtained *via* the ring-opening of β-pinene.

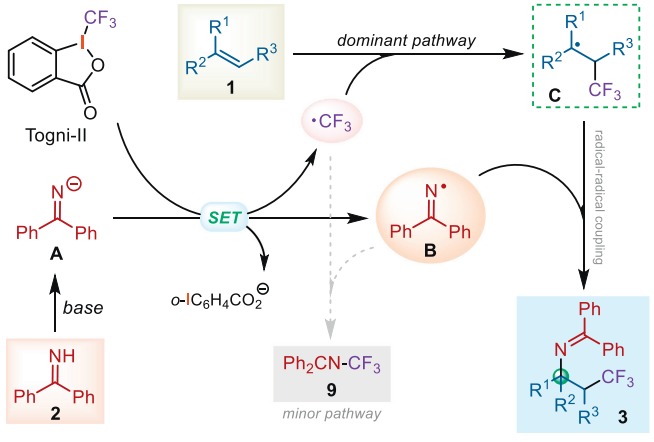
We next aimed at identifying the reaction pathway and intermediates by ^{19}F NMR monitoring *via* the sequential addition of each reaction component to an NMR tube. This approach enabled the detection of the interaction between different compounds that are likely involved in this process. When Togni-II reagent was mixed with benzophenone imine **2** in CD_3CN/DMSO–d_6 (9/1), no reaction occurred (Scheme 4, b-I). However, upon the addition of 1.0 equiv. of LiO*t*Bu to this mixture, a new signal obviously appeared and increased gradually while the Togni-II signal decreased, which elucidates that there exists a subtle correlation between the hypervalent iodine reagent and benzophenone imine anion. The signal at −52.1 ppm corresponds to the compound identified as the



Scheme 4. Mechanistic studies. (a) Radical trapping experiment with additional TEMPO under standard conditions. (b) *In situ* monitoring by ^{19}F NMR *via* sequential addition of all components of the model reaction. Togni-II: green ♣; **3a**: blue ♥; **9** red ♦.

adduct $Ph_2CN–CF_3$ **9** on the basis of isolation (Scheme 4, b-II and b-III). While mixing the standard stoichiometric amounts of olefin **1a**, benzophenone imine **2** and Togni-II at room temperature led to the rapid appearance of two signals at −59.3 and −52.1 ppm, which were attributed to the product **3a** and an accompanied generating two-free-radicals adduct **9**, respectively (Scheme 4, b-IV and b-V).

On the basis of the above mechanistic experiments, a putative mechanism for the reaction is outlined in Scheme 5. Initially, a single-electron-transfer (SET) process occurs between benzophenone imine anion **A** and hypervalent-iodine-CF_3 reagent (path a), generating a CF_3 radical and *o*-$IC_6H_4CO_2H$ anion along with the corresponding ambiphilic diphenyliminyl radical **B**. Followed by the



Scheme 5. Proposed mechanism.

addition of the CF_3 radical to an alkene, the carbo-radical intermediate **C** is formed. Finally, the subsequent selective trapping steered by the "persistent radical effect" [69,70] with the iminyl radical **B** produces the corresponding trifluoromethylimination products **3**.

In summary, we have disclosed a practical method for the trifluoromethylamination of activated and unactivated alkenes under transition-metal free and ambient conditions. This multicomponent reaction can be compatible with broad scope and a wide range of functional groups, excellent regioselectivities, which was enabled by a robust experimental operation. Monitoring of the reactions by NMR spectroscopy, supplemented by byproduct detection shed light on some of the mechanistic subtleties in this study. Notably, the imine unit could be readily converted to unprotected amines thereby facilitating downstream transformations. We believe that the described protocol will prove useful in an array of synthetic contexts and would be potential for finding applications in both academic research and industry.

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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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.109502.

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