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COMMUNICATION

Simple generation of various α-monofluoroalkyl radicals by organic photoredox catalysis: modular synthesis of β-monofluoroketones†

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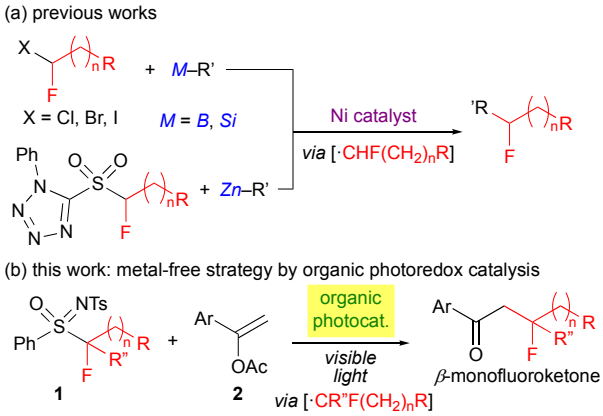
A metal-free and operationally simple strategy for generation of various α-monofluoroalkyl radicals has been developed. A combination of 1,4-bis(diarylamino)naphthalene photocatalyst and sulfoximine-based fluoroalkylating reagents is a key to success. The protocol can be applied to modular synthesis of β-monofluoroketones through radical monofluoroalkylation of alkenyl acetates.

In recent years, importance of organofluorine compounds has continued to increase in fields of pharmaceuticals, agrochemicals and organic functional materials.[1] Therefore, development of efficient and operationally simple synthetic methods for fluorination and introduction of fluorinated organic units has become a pivotal topic in organic synthetic chemistry. Fluorination, which is the simplest method for introduction of a fluorine atom into organic skeletons, has been actively studied so far.[2] On the other hand, while α-monofluoroalkylation can be regarded as a simple method for introduction of organic units bearing a stereogenic carbon center bound to a fluorine atom, introduction of $-CHF(CH_2)_nR$ ($n \geq 1$) scaffolds has still been underdeveloped.[3] Recently, several studies on catalytic α-monofluoroalkylation through radical processes have been reported (Scheme 1a).[4,5] The Gandelman group developed enantioselective synthesis of secondary alkyl fluorides *via* Ni-catalyzed reductive cross-coupling of 1-fluoro-1-haloalkanes and organoboron compounds.[4] The work was followed by Ni-catalyzed cross-coupling reactions with other organometallic reagents such as organosilicon and organozinc reagents.[5a,c] The latter works[5] suggested that α-monofluoroalkyl radical species are involved in the reaction



Scheme 1 Catalytic introduction of $-CR''F(CH_2)_nR$ ($n \geq 1$) groups *via* radical processes.

systems. More recently, the Baran group showed that α-monofluoroalkyl sulfone reagents serve as α-monofluoroalkyl radical sources.[5c] On the other hand, single-electron-transfer (SET) processes by photoredox catalysis have become useful strategies for generation of a variety of radical species under operationally easy conditions, *i.e.*, visible light irradiation below room temperature.[6] For the past several years, organic photoredox catalysis has attracted more attention from the viewpoint of sustainable development goals (SDGs).[7] However, versatile protocols for generation of various α-monofluoroalkyl radicals have not been reported yet. More recently, our group reported that highly reducing organic photoredox catalysts, 1,4-bis(diarylamino)naphthalene derivatives (BDNs), promote radical monofluoromethylation with *N*-tosyl-*S*-monofluoromethyl-*S*-phenylsulfoximine (**1a** in Scheme 2),[8] which is a bench-stable chemical. Herein we describe simple preparation of various *N*-tosyl-*S*-monofluoroalkyl-*S*-phenylsulfoximines (**1**) from **1a** and generation of novel α-monofluoroalkyl radicals from easy-to-handle reagents **1** by organic photoredox catalysis (Scheme 1b). Furthermore, the generated α-monofluoroalkyl radicals can be applied to the reaction with alkenyl acetates (**2**), leading to modular synthesis

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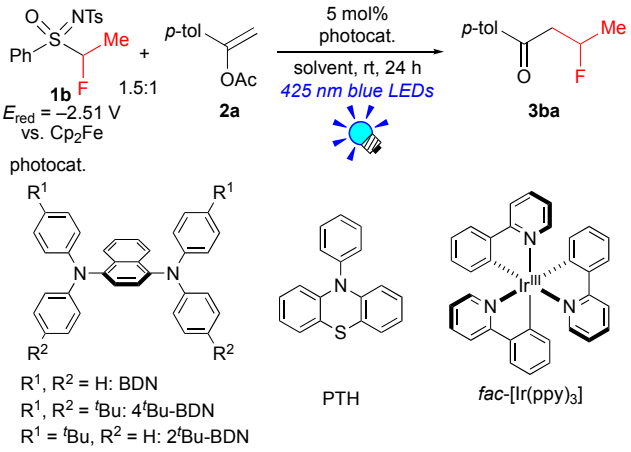
† Electronic Supplementary Information (ESI) available. CCDC 2047104–2047107. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/x0xx00000x

of synthetically valuable β-monofluoroketones, which are not easily accessible.[9] In addition, we have disclosed newly developed 1,4-bis(*N*-*p*-^{t}Bu-phenyl-*N*-phenylamino)naphthalene (2^{t}Bu-BDN) serves as a good organic photoredox catalyst in spite of the short-lived character (10 ns at 20°C) of the electronically excited state.

N-Tosyl-*S*-monofluoroalkyl-*S*-phenylsulfoximines (**1**) were prepared according to the modified procedures with reference to the literature reported previously by Hu.[10] Deprotonation of **1a** by *n*-butyllithium followed by treatment with the corresponding electrophiles (R(CH$_2$)$_n$–L) afforded bench-stable **1** in moderate yields (15–50%: Yields are for mixtures of diastereomers, which could not be separated by column chromatography except for **1b–d, g**)(Scheme 2). Single-crystal X-ray analyses of **1b** and **1d** confirmed their structures (See the ESI).‡ It should be noted that preparation of **1** bearing functionalities such as heteroaromatics and unsaturated C–C bonds are feasible through the present synthetic scheme.

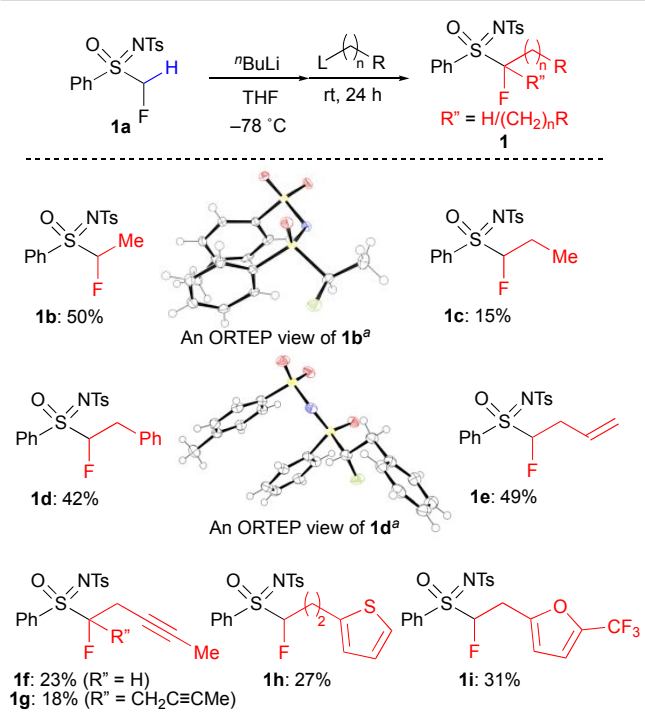
We initially studied photocatalytic reaction of alkenyl acetate derivative **2a** with **1b** (E_{red} = –2.51 V vs Cp_2Fe in acetone) in acetone-d_6 in the presence of strongly reducing 1,4-bis(diphenylamino)naphthalene (BDN) catalyst under visible light irradiation (425 nm blue LEDs). As a result, 3-fluoro-1-(*p*-tolyl)butan-1-one (**3ba**) was obtained in a 65% NMR yield (entry 1 in Table 1). Choice of solvent turned out to be crucial (entries 2–8). Furthermore, addition of a small amount of water to acetone-d_6 increased the yield (73%, entry 5). Next, BDN derivatives, 4^{t}Bu-BDN[8b] and newly developed 2^{t}Bu-BDN,‡,§ were tested as a photocatalyst (entries 9 and 10). Interestingly, 2^{t}Bu-BDN exhibited much better catalytic activity (81%, entry 10). Other commonly used reducing photocatalysts, 10-phenylphenothiazine (PTH) and *fac*-[Ir(ppy)$_3$], resulted in lower yields (entries 11 and 12: For reactions with other photocatalysts, see the ESI). Visible light irradiation and photocatalyst are essential for the present reaction (entries 13 and 14). Reduction of the catalyst loading significantly deteriorated reaction efficiency (entry 15).

Table 1 Optimization of reaction conditions.



Entry	Photocat.	Solvent	Yield of **3ba**/%
1	BDN	acetone-d_6	65
2	BDN	CD_2Cl_2	18
3	BDN	CD_3CN	37
4	BDN	C_6D_6	35
5	BDN	acetone-d_6/D_2O (19/1)	73
6	BDN	DMSO-d_6/D_2O (19/1)	31
7[a]	BDN	DMF/H_2O (19/1)	19
8	BDN	CD_3OD/D_2O (19/1)	0
9	4^{t}Bu-BDN	acetone-d_6/D_2O (19/1)	69
10	2^{t}Bu-BDN	acetone-d_6/D_2O (19/1)	81
11[b]	PTH	acetone-d_6/D_2O (19/1)	48
12	*fac*-[Ir(ppy)$_3$]	acetone-d_6/D_2O (19/1)	21
13[c]	2^{t}Bu-BDN	acetone-d_6/D_2O (19/1)	0
14	–	acetone-d_6/D_2O (19/1)	0
15	2^{t}Bu-BDN (1 mol%)	acetone-d_6/D_2O (19/1)	16

Yields were determined by ^{1}H NMR spectroscopy using tetraethylsilane as an internal standard. General reaction conditions: **2a** (25.0 μmol), **1b** (37.5 μmol, 1.5 equiv.), photoredox catalyst (1.25 μmol, 5 mol%), solvent (0.50 mL). [a] **2a** (50 μmol), **1b** (75 μmol, 1.5 equiv.), BDN (2.5 μmol, 5 mol%), DMF (0.95 mL) and H_2O (0.05 mL) were used. The NMR yield was determined using 1,3,5-trimethoxybenzene as a standard. [b]380 nm LEDs were used. [c]In the dark.

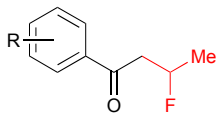


Scheme 2 Preparation of sulfoximine-based monofluoroalkylating reagents **1**. [a]ORTEP diagrams with ellipsoids shown at the 50% contour percent probability level.

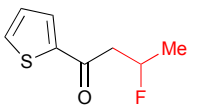
With the optimized reaction conditions in hand, the scope of the present reaction with respect to α-monofluoroethylation was examined (Table 2a). In addition to synthesis of **3ba** (76% isolated yield), 3-fluoro-1-(*m*-tolyl)butan-1-one (**3bb**) and 3-fluoro-1-(*o*-tolyl)butan-1-one (**3bc**) could be also obtained from the corresponding alkenyl acetates (**2b** and **2c**) in moderate yields (**3bb**: 58%, **3bc**: 40%). The present α-monofluoroethylation could be applied to electronically unbiased alkenyl acetates, α-acetoxystyrene (**2d**) and α-acetoxy-4-phenylstyrene (**2e**) (**3bd**: 76%, **3be**: 73%). In addition, functional groups such as OMe (**2f**), halogen (**2g**,**h**,**i**), NO_2(**2j**), CN(**2k**), CO_2Me(**2l**),‡ and thienyl (**2m**) groups were compatible with the reaction conditions (**3bf**–**3bm**: 23–73%). Furthermore,

Table 2 Scope of photocatalytic α-monofluoroalkylation[a].

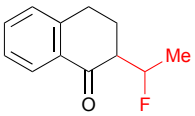
(a) α-monofluoroethylation of **2**



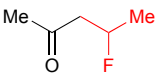
R = *p*-Me (**3ba**): 76% | *p*-F (**3bg**): 73%
m-Me (**3bb**): 58% | *p*-Cl (**3bh**): 71%
o-Me (**3bc**): 40%[b,c] | *p*-Br (**3bi**): 66%[d]
H (**3bd**): 76% | *p*-NO_2 (**3bj**): 23%[d]
p-Ph (**3be**): 73% | *p*-CN (**3bk**): 49%[d]
p-OMe (**3bf**): 76% | *p*-CO_2Me (**3bl**): 63%



3bm: 60%

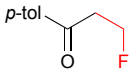


3bn[c,d]: 37% (dr = 53:47)

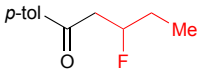


3bo: 0[e]

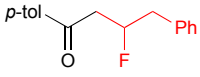
(b) α-monofluoroalkylation of **2a**



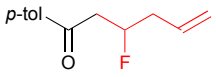
3aa: 81%



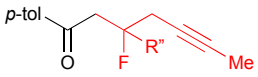
3ca: 64%



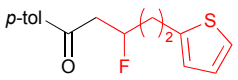
3da: 77%



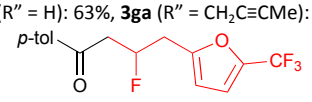
3ea: 61%



3fa (R″ = H): 63%, **3ga** (R″ = $CH_2C{\equiv}CMe$): 56%,



3ha: 67%



3ia: 72%

[a]Reaction conditions: **2** (0.250 mmol), **1** (0.375 mmol, 1.5 equiv.), 2[t]Bu-BDN (12.5 µmol, 5 mol%), acetone (4.75 mL), H_2O (0.25 mL), isolated yields. [b]Reaction time = 72 h. [c]10 mol% of 2[t]Bu-BDN was used. [d]Reaction time = 48 h. [e]Isopropenyl acetate **2o** (25.0 µmol), **1b** (37.5 µmol, 1.5 equiv.), 2[t]Bu-BDN (1.25 µmol, 5 mol%), and acetone-d_6/D_2O = 19/1 (0.5 mL) were used. Determined by ^{1}H NMR analysis using tetraethylsilane as an internal standard.

an internal alkenyl acetate (**2n**) afforded the corresponding β-monofluoroketone derivative (**3bn**: 37%, dr = 53:47), selective access to which is difficult as reported for the synthesis *via* ring-opening fluorination of cyclopropanols.[9c,d] Additionally, an aliphatic alkenyl acetate (**2o**) did not afford the corresponding product.

Next, the monofluoroalkylating reagents **1** were used for modular synthesis of β-monofluoroketones **3** (Table 2b). The photocatalytic reaction of **2a** with **1** in the presence of 2[t]Bu-BDN afforded the corresponding β-monofluoroketones **3** in good yields (**3aa**: 81%, **3ca**: 64%, **3da**: 77%, **3ea**: 61%, **3fa**: 63%, **3ha**: 67%, **3ia**: 72%). Remarkably, the reagent **1g** afforded the ketone bearing a fluorine-containing quaternary carbon center at the β-position (**3ga**) in a 56% yield. It should be noted that vinyl, alkynyl, and heteroaromatic units, which are potentially susceptible to radical addition, are not deteriorated under the present reaction conditions.

A plausible reaction mechanism is shown in Scheme 3. The reducing power of 2[t]Bu-BDN in the excited state was determined to be E_{ox}* = –2.39 V vs. Cp_2Fe in acetone,[11] which is enough to reduce the sulfoximine-based monofluoroalkylating

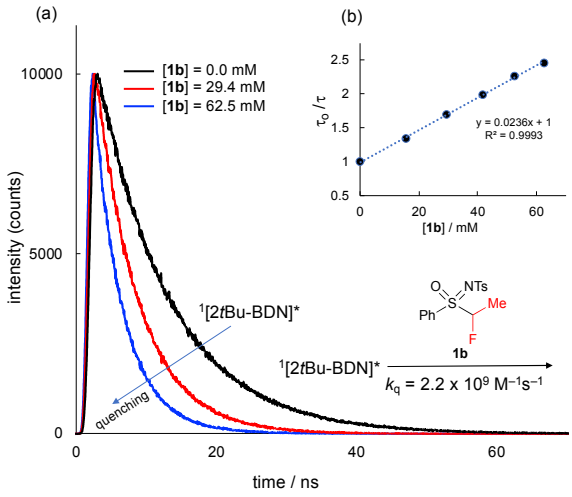
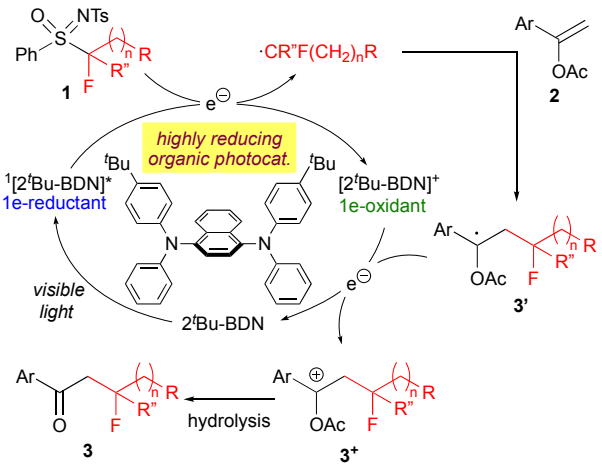


Figure 1 Measurements of fluorescence lifetime (τ, excited at 390 nm and monitored at 458 nm in acetone at ≈20 °C under N_2) of 2[t]Bu-BDN in the presence of **1b** (a); Stern-Volmer plot (b).



Scheme 3 A plausible reaction mechanism.

reagents **1** (E_{red} = –2.55 - –2.44 V). Indeed, the fluorescence lifetime (τ = 10 ns) of 2[t]Bu-BDN was significantly shortened by **1b** in acetone under N_2 atmosphere (Figure 1a). The Stern-Volmer analysis provided the quenching rate constant of k_q = 2.2 x 10^9 $M^{-1}s^{-1}$, which is close to the diffusion-controlled rate constant in acetone at 20°C (2.0 x 10^{10} $M^{-1}s^{-1}$) (Figure 1b). In contrast, alkenyl acetate **2a** did not quench the fluorescence efficiently (See Figure S5 in the ESI). Furthermore, the radical trapping experiments as well as EPR experiments supported generation of the α-monofluoroethyl radical from **1b** under the photocatalytic conditions (See Figure S6, S10, and S11 in the ESI). These results suggest that the short-lived photoexcited ^{1}[2[t]Bu-BDN]* is enough to undergo SET to the monofluoroalkylating reagent **1**, leading to generation of the α-monofluoroalkyl radical. The generated radical reacts with alkenyl acetate **2** to yield the radical intermediate **3′**. The second SET by the oxidized catalyst, [2[t]Bu-BDN]$^+$, is followed by hydrolysis,[12] leading to the β-monofluoroketone product **3**. Addition of a small amount of water might promote hydrolysis of **3**$^+$. The quantum yield in the

reaction of **2a** with **1b** ($\Phi_{reaction}$ = 8%) and light ON/Off experiment indicate small contribution of the radical chain process to the mechanism (See the ESI).

In summary, we have developed the metal-free strategy for generation of various α-monofluoroalkyl radicals from bench-stable sulfoximine-based fluoroalkylating reagents. The newly developed organic photoredox catalyst, 1,4-bis{*N*,*N*-(*p*-^{t}Bu-phenyl)(phenyl)amino}naphthalene, serves as a good photocatalyst, leading to operationally easy synthesis of β-monofluoroketone derivatives. LFP studies strongly support efficient electron transfer from the short-lived excited photocatalyst to the sulfoximine-based fluoroalkylating reagent. Further development of reactions triggered by generation of α-monofluoroalkyl radicals is ongoing in our laboratory.

Dedicated to all doctors, nurses, and researchers who are fighting against COVID-19 pandemic. The financial support from the JSPS (KAKENHI Grants 19H02711 and 16H06038), JST CREST (Grant Number JPMJCR18R4), and Naito Foundation are gratefully acknowledged. This work was performed under the Cooperative Research Program of the "Network Joint Research Center for Materials and Devices".

Conflicts of interest

There are no conflicts to declare.

Notes and references

‡ Molecular structures of **1b**, **1d**, 2^{t}Bu-BDN, and **3bl** are shown in the ESI.† CCDC 2047104–2047107.
§ Photo- and electro-chemical data of 2^{t}Bu-BDN are shown in the ESI.†

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