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Photocatalyzed generation of acyl radicals from aryl/alkyl acyl halides *via* nucleophilic substitution

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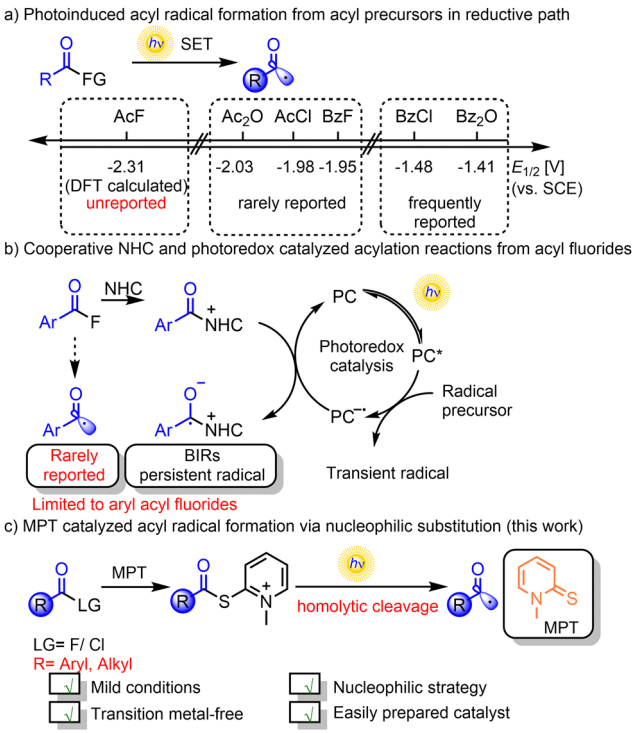
Herein, we introduce a nucleophilic catalysis strategy based on a 1-methylpyridine-2(1*H*)-thione (MPT) catalyst, which mediates the *in situ* generation of photoactive intermediates from various acyl halides. This strategy enables the efficient formation of both aryl and alkyl acyl radicals upon visible-light irradiation, including those derived from otherwise unreactive alkyl acyl fluorides/chlorides.

Acyl halides are inexpensive, abundant, highly reactive, and commercially available chemicals that have been extensively employed in organic synthesis,[1,2] traditionally engaging in transformations *via* acyl cation intermediates. Concurrently, visible-light photoredox catalysis has emerged as a powerful, sustainable, and environmentally benign synthetic strategy.[3–5] This strategy has unlocked diverse acyl radical precursors, including ketoacids, aldehydes, and notably, acyl chlorides, thereby significantly expanding the synthetic toolbox for radical acylation reactions with high synthetic potential.

Through photooxidative pathways, ketoacids have been established as versatile aryl and alkyl acyl sources, enabling efficient construction of C–C[6–9] and C–X[10] bonds. Aldehydes are also used as acyl radical sources *via* a photoinduced hydrogen atom transfer process, participating in radical addition reactions,[11,12] coupling reactions[13] and functional group conversion reactions.[14,15] Through the photoreductive pathway, acid anhydride[16] and acid chloride[17–20] have been utilized for acyl radical generation, though the latter have been limited to aryl acid chlorides. Additional methods, such as the phosphorus-mediated deoxygenation of carboxylic acids,[21–23] along with thioester[24–26] and acyl silanes,[27] further complement the toolbox for acyl radical generation.

However, the photocatalytic application of carboxylic acid derivatives, particularly alkyl acyl halides and acid anhydrides, remains constrained by redox potential limitations[28–30] (Scheme 1a). For instance, acetyl chloride ($E_{1/2}^{red}$ = −1.98 V *vs.* SCE[29]) and acetyl anhydride ($E_{1/2}^{red}$ = −2.03 V *vs.* SCE[29])



Scheme 1 (a) Photoinduced acyl radical formation. (b) Cooperative NHC and photoredox catalyzed acylation reactions. (c) MPT-catalyzed acyl radical formation *via* nucleophilic substitution.

exceed the typical reducing capacity of *fac*-Ir(ppy)$_3$ $\left(E_{1/2}^{IV/*III} = -1.73\ \text{V}\ vs.\ \text{SCE}^3\right)$. As for acyl fluorides, the high C–F bond dissociation energy makes direct generation of acyl radicals through homolytic C–F bond cleavage highly challenging. Additionally, the low leaving-group tendency of F^- renders acyl fluorides chemically inert among acyl halides. Although there have been a few recent reports describing acyl radical formation from aryl fluorides through charge-transfer complex (CTC) activation[31] or transition-metal catalyzation,[32] such transformations remain underexplored, especially for

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alkyl acyl fluorides, which currently constitute uncharted territory in photocatalytic radical generation.

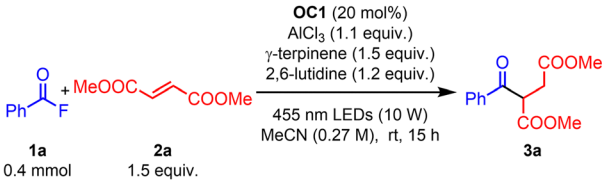
Recently, Scheidt's group and Studer's group independently demonstrated the single-electron reduction of NHC–acyl adducts to generate Breslow intermediate-driven radicals (BIRs), using *N*-benzoylimidazole[33] and acyl fluorides,[34] respectively. Later, the cooperative NHC and photoredox catalyzed acylation reactions proceeding from acyl fluorides *via* BIRs were further developed by Studer,[35–39] Zhang[40–42] and Feng.[43,44] In addition, other researchers have extended these co-catalytic systems to accommodate complex coupling reactions.[45–51] In these reactions, NHC catalysis is vital for forming Breslow intermediates, which are more easily reduced to ketyl radicals than acyl fluorides are. However, these acylation reactions are limited to aryl acyl fluorides (Scheme 1b). Thus, it is important to develop a new, simple, and universal methodology using only one photocatalyst for the generation of acyl radicals from aryl/alkyl acyl fluorides.

Recently, Melchiorre's group[52–55] and our group (MPT catalyst)[56] reported the generation of carbon radicals through an S_N2 pathway. This MPT catalyst exhibits good nucleophilicity and visible-light absorptivity.[56,57] Herein, we report a photoinduced MPT-catalyzed nucleophilic substitution of acyl halides to generate acyl radicals. Both acyl fluorides and acyl chlorides can be applied in this reaction to form the nucleophilic substitution intermediate, which can be photoexcited and undergo homolytic cleavage of the C(sp^2)–S bond to generate acyl radicals. In particular, the aryl and alkyl acyl fluorides/chlorides are all tolerated in this reaction (Scheme 1c).

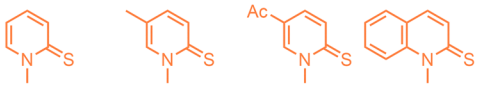
To test our hypothesis, we initially used benzoyl fluoride (**1a**) and dimethyl fumarate (**2a**) as model substrates. To increase the reactivity of the benzoyl fluoride, we added $AlCl_3$ as the additive. To our delight, the desired product **3a** could be obtained in 33% yield (see the SI, Table S1). After screening the reaction conditions (see the SI, Tables S1–S7), the highest yield could be obtained as 71% (Table 1, entry 1). Other MPT catalysts gave lower yields (Table 1, entries 2–4). The yields dropped a lot without $AlCl_3$ or light (Table 1, entries 5 and 6). The reaction was shut down without **OC1**, 2,6-lutidine or γ-terpinene (Table 1, entries 7–9).

With the optimized conditions in hand, we then investigated the scope of the reaction (Scheme 2). We first tested the *para*-substituted benzoyl fluorides (**3b–3m**). *para*-Alkyl/phenyl benzoyl fluorides (**3b–3e**) gave moderate yields (56–65%). Halogens (**3f–3h**) were tolerated in the reactions and showed similar reactivity (52–63%). Benzoyl fluorides with electron-donating groups on the *para* position (**3j–3m**) achieved higher yields (62–68%). Meanwhile, the yields dropped when the electron-donating groups were on the *meta* and *ortho* positions (**3n–3o**). Electron-withdrawing groups could also be applied in this reaction in which 1.4 equiv. of $AlCl_3$ was added to yield **3p** in 51% yield. It should be pointed out that carbonyl-containing groups (**1p**) may deactivate $AlCl_3$ and decrease the formation of the intermediate, while N-containing groups (**1i**) may cause $AlCl_3$ to precipitate. To our delight, di- and tri-substituted benzoyl fluorides showed good reactivities in this reaction

Table 1 Optimization of the reaction conditions



Entry	Deviation from standard conditions[a]	Yield of **3a** (%)[b]
1	none	71 (71)[c]
2	**OC2** instead of **OC1**	19
3	**OC3** instead of **OC1**	21
4	**OC4** instead of **OC1**	N. D.
5	no $AlCl_3$	8
6	No light	8
7	No **OC1**	trace
8	No 2,6-lutidine	trace
9	No γ-terpinene	trace



[a] Reaction conditions: **1a** (0.4 mmol), **2a** (1.5 equiv.), **OC1** (20 mol%), $AlCl_3$ (1.1 equiv.), γ-terpinene (1.5 equiv.), 2,6-lutidine (1.2 equiv.), MeCN (1.5 mL) with irradiation by a 10 W 455 nm LED at room temperature for 15 h under a N_2 atmosphere. [b] Yield determined by ^{1}H NMR analysis of the crude mixture using 0.4 mmol of mesitylene as an internal standard. [c] Isolated yield.

(**3q–3u**). 2-Naphthoyl fluoride also reacted smoothly and afforded the product in 41% yield (**3v**). Notably, the alkyl acyl fluoride **1w** and **1x** could be applied in this reaction and afford **3w** and **3x** in 40% and 41% yields, respectively. These alkyl acyl fluorides were not suitable for the cooperative NHC and photoredox catalyzed system.[34–51]

Aryl acyl chlorides[17–20] could also be applied in this reaction. Acyl chlorides were more reactive than acyl fluorides. Both electron-rich and electron-poor acyl chlorides were good substrates for this reaction and gave the product in moderate to good yields (**3y–3ad**). These results suggested that electronic effects play a dominant role in this reaction (**3ab–3ad**, **3af–3ag**). Interestingly, heteroaromatic acyl chlorides (**1ae–1ag**) and alkyl acyl chlorides (**1w–1x**, **1ah–1ai**) were suitable for this reaction. Meanwhile, *N*-substituted aminoacyl chlorides and carbonochloridates were not good substrates in these reactions.

We also investigated several Michael acceptors in the Giese reactions with 4-MeS benzoyl fluoride as the model substrate (Scheme 3). Fumarate diesters were suitable for this reaction, yielding **4b–4e** in 53–66% yields. Acrylic ester derivatives (**4f–4h**) were reactive with lower yields (30%–36%). Electron-poor 1,1-disubstituted alkenes could also be applied in this reaction and afforded the corresponding products in moderate yields (**4i–4l**). Alkenes bearing various electron-withdrawing groups such as nitrile, sulfone, and ketone could afford the products (**4m–4o**) in moderate yields.

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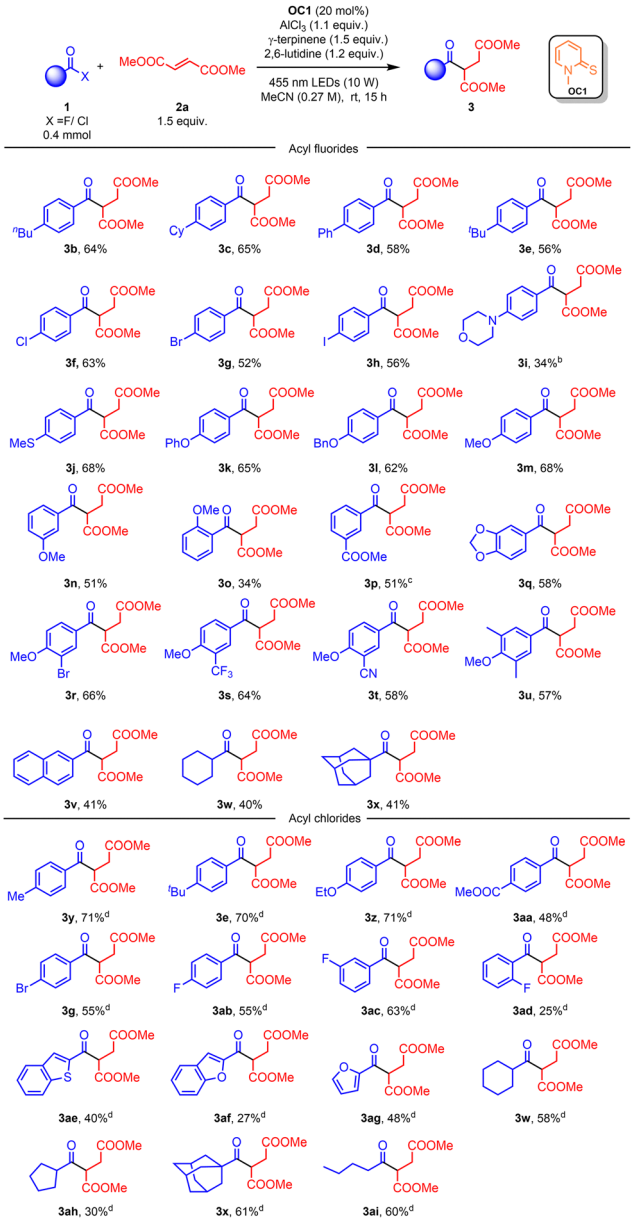
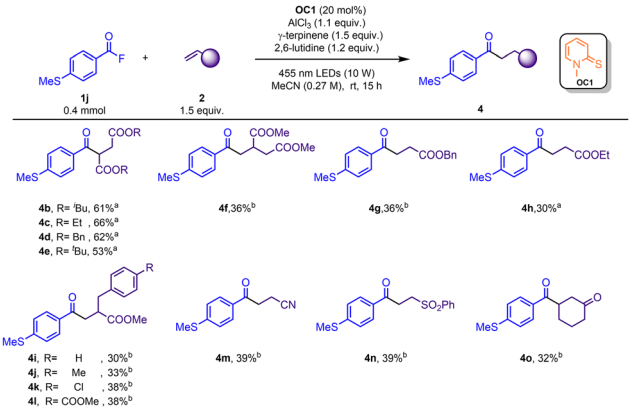


Scheme 2 Substrate scope of acyl fluorides and acyl chlorides. [a] Reaction conditions: **1** (0.4 mmol), **2a** (1.5 equiv.), **OC1** (20 mol%), $AlCl_3$ (1.1 equiv.), γ-terpinene (1.5 equiv.), 2,6-lutidine (1.2 equiv.) in MeCN (1.5 mL) with irradiation by a 10 W 455 nm LED at room temperature for 15 h under a N_2 atmosphere. [b] 20 μL of DMAc was added. [c] $AlCl_3$(1.4 equiv.) was added. [d] **1**(0.4 mmol), **2a** (2.0 equiv.), **OC2** (0.2 equiv.), γ-terpinene (1.8 equiv.), 2,6-lutidine (1.2 equiv.) in MeCN (2.0 mL) with irradiation by a 10 W 455 nm LED at 40 °C for 18 h under a N_2 atmosphere.

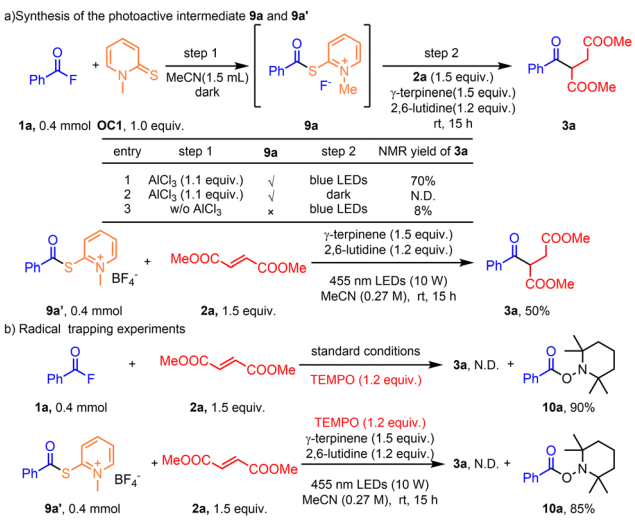
To demonstrate the synthetic utility, we smoothly performed a gram-scale preparation of **3a** and obtained **3a** in 53% yield (see the SI, Scheme S3a). Subsequent derivatization of **3a** was efficiently achieved (see the SI, Scheme S3b). Lactam[58] and lactone[59,60] could be synthesized from **3a** in 60–72% yields (**5**–**7**). Interestingly, α-bromide derivative **8**[61,62] could be formed in 74% yield, serving as an intermediate for the synthesis of hydantoins.[61]

Control experiments were conducted to gain more insights into the reaction mechanism. In the presence of $AlCl_3$, **1a** and



Scheme 3 Substrate scope of Michael acceptors. [a] Reaction conditions: **1j** (0.4 mmol), **2** (1.5 equiv.), **OC1** (20 mol%), $AlCl_3$ (1.1 equiv.), γ-terpinene (1.5 equiv.), 2,6-lutidine (1.2 equiv.) in MeCN (1.5 mL) with irradiation by a 10 W 455 nm LED at room temperature for 15 h under a N_2 atmosphere. [b] 20 μL of DMAc was added.

OC1 could form the photoactive intermediate **9a** in the dark. **9a** could be photoexcited to generate an acyl radical and afford the corresponding product **3a**. The reaction could not work without $AlCl_3$ or without light (Scheme 4a). We tried to isolate intermediate **9a**, but failed. Therefore, we synthesized **9a′**[63] instead and subjected **9a′** to the standard reaction conditions affording product **3a** in 50% yield. Radical trapping experiments with TEMPO showed that no desired product was found and adduct **10a** was isolated in 90% and 85% yields, respectively (Scheme 4b). Additionally, no redshift phenomenon was observed with UV-Vis spectroscopic analysis (see the SI, Scheme S8). The possibility of an electron donor–acceptor (EDA) complex process could be excluded. The light on/off experiment showed that this reaction was not a chain reaction (see the SI, Scheme S10). The addition of triplet quenchers[64–66] significantly suppressed the reaction (see the SI, Table S8), which indicated the involvement of a triplet state species. These experimental results confirm the



Scheme 4 (a) Synthesis of the photoactive intermediate. (b) Radical trapping experiments.

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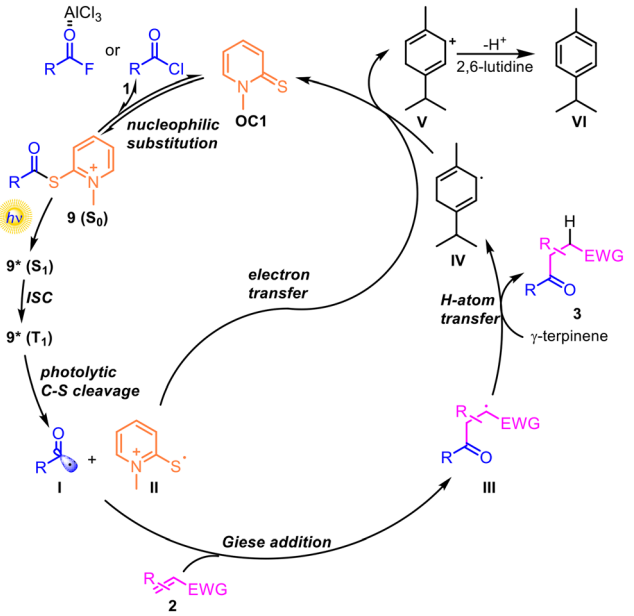

Scheme 5 Proposed mechanism.

generation of nucleophilic intermediates **9**, which, upon absorption of blue light, undergo homolytic cleavage to produce acyl radicals.

Based on the above results and related reports,[52–56] a plausible catalytic mechanism is proposed (Scheme 5). In the presence of $AlCl_3$, the nucleophilic catalyst **OC1** reacted with acyl halides to generate the key intermediate **9** through a nucleophilic substitution pathway. Upon visible-light irradiation, **9** was photoexcited to the S_1 state and underwent intersystem crossing (ISC) to the T_1 state, from which homolytic C–S bond cleavage generated acyl (**I**) and thiyl (**II**) radicals. Then, the acyl radical was trapped by Michael acceptors, followed by HAT to form the corresponding product **3**. Meanwhile, the thiyl radical **II** could be reduced by carbon radical **IV** through a SET process to reproduce the nucleophilic catalyst **OC1**, thereby closing the catalytic cycle. Carbocation **V** generated after single-electron transfer underwent aromatization with the base 2,6-lutidine to give **VI**.

In summary, we developed a photoinduced MPT-catalyzed nucleophilic substitution of acyl halides to generate acyl radicals. Aryl/alkyl acyl fluorides and chlorides were suitable in this reaction. Notably, alkyl acyl fluorides proved ineffective as substrates in previously reported photoinduced acylation reactions. The MPT catalyst and acyl halides could form the photoactive nucleophilic substitution intermediates, which then could be photoexcited to generate acyl radicals *via* C(sp^2)–S bond homolytic cleavage. This nucleophilic substitution strategy does not rely on the redox properties of acyl halides, including those with low redox potentials such as alkyl acyl fluorides. It offers a powerful, modular, and practical strategy for constructing valuable aroyl/alkanoyl motifs. The easily prepared catalyst, mild reaction conditions, good yields, and broad scope show the potential synthetic utility of this method. Further studies including other acylation reactions are being conducted in our laboratory.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: experimental details and NMR spectra. See DOI: https://doi.org/10.1039/d6cc00687f.

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