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Multiphoton photoredox catalysis enables selective hydrodefluorinations

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ABSTRACT

Late-stage modification of complex molecules *via* site-selective hydrodefluorination is a challenging endeavor. The selective activation of carbon-fluorine (C–F) bonds in the presence of multiple C–F bonds is of importance in organic synthesis and drug discovery. Herein, we describe the activation of C-F bonds *via* multiphoton photoredox catalysis to selectively produces a series of hydrodefluorinated compounds by simply tuning the reaction conditions. Moreover, this protocol was successfully applied to the late-stage functionalization of different drug-derivatives and the corresponding mono-, di-, and tri-defluorinated products were obtained in good to excellent yields. A detailed mechanistic investigation provides insight into the unprecedented hydrodefluorination pathway.

Developing novel catalytic methods for the site-selective hydrodefluorination (HDF) is of long-standing interest in organic chemistry due to the widespread presence of C-F bonds in agrochemicals, pharmaceuticals, and materials [1–9]. Mono- and difluorinated compounds have significant potential in drug discovery research due to their hydrogen bonding abilities, exceptional pharmacokinetic (PK) properties, and enzymatic stability [10–13]. However, late-stage modification of the R-CF_3 group into an R-CHF_2 or R-CH_2F is exceedingly difficult due to the strong C–F bond dissociation energy [2]. Indeed, the reduction and replacement of the first F atom from the CF_3-group requires a strong reducing reagent or catalyst due to the high reduction potential. Traditionally, DAST or Selectfluor was used as a fluorinating agent to incorporate *gem*–difluoromethylene groups with the generation of a stoichiometric amount of waste [14–16]. Given that the mono-defluorinated compound R-CHF_2 has less reduction potential than the parent molecule R-CF_3, controlling selectivity in HDF to mono-, di-, or tri-defluorination is exceptionally challenging [17].

Selective HDF has numerous advantages, including its complementarity to classical C-F bond formation, the avoidance of multistep synthesis, a more cost-effective process, and the possible use of feedstock chemicals such as trifluoroacetamide or trifluoroacetate [17,18]. There are several methods known for C–F bond formations [2,10,14–16]. However, methods for selective HDF are limited [18–24]. The functionalization of CF_3 groups generally proceeds through heterolytic cleavage of a C–F bond, affording difluoro-substituted carbocation or carbanion intermediates. In contrast, trapping of homolytically cleaved fluoroalkyl radical intermediates to new bond-forming reactions is less explored because of the high bond dissociation energy (BDE) of the C–F bond [17,18]. Recently, several research groups [17,18,21,25–31] disclosed strategies to eliminate an F-atom from CF_3- substituted arenes to generate aryldifluoromethyl radicals *via* single-electron reduction.

An elegant new methodology has been developed by Wang and Houk group [17] for fluorine elimination of polyfluorinated aliphatic esters and amides *via* a boryl radical enabled spin-center shift (SCS) [32] strategy (Scheme 1a). Furthermore, the Molander [30] and Glorius [31] groups have independently reported selective defluorination of trifluoroacetate/amide *via* a HAT process. However, the reported methods are limited to the mono-defluorination and liberate a stoichiometric amount of CO_2, which may hamper the reaction when olefin-trifluoroacetamide was used and lead to hydrocarboxylation as a side reaction.

In recent years, consecutive photoinduced electron transfer (conPET) and multiphoton excitation catalysis have emerged as strategies to overcome the thermodynamic limitations of visible-light photoredox catalysis [33–45]. In this context, conPET photocatalysts, mainly organic dyes, have been explored for dehalogenation reactions (mainly iodo, bromo or chloro arenes) [33,35,37,46]. Similarly, multiphoton excitation Ir-catalysis was applied for reduction and hydrofunctionalization of olefins *via* a two-photon tandem photoredox process [39]. However, conPET or multiphoton catalysis has not been used for selective defunctionalization, especially for HDF of non-activated CF_3-groups, possibly due to the high redox potential of the photo-excited anionic catalyst, which often leads

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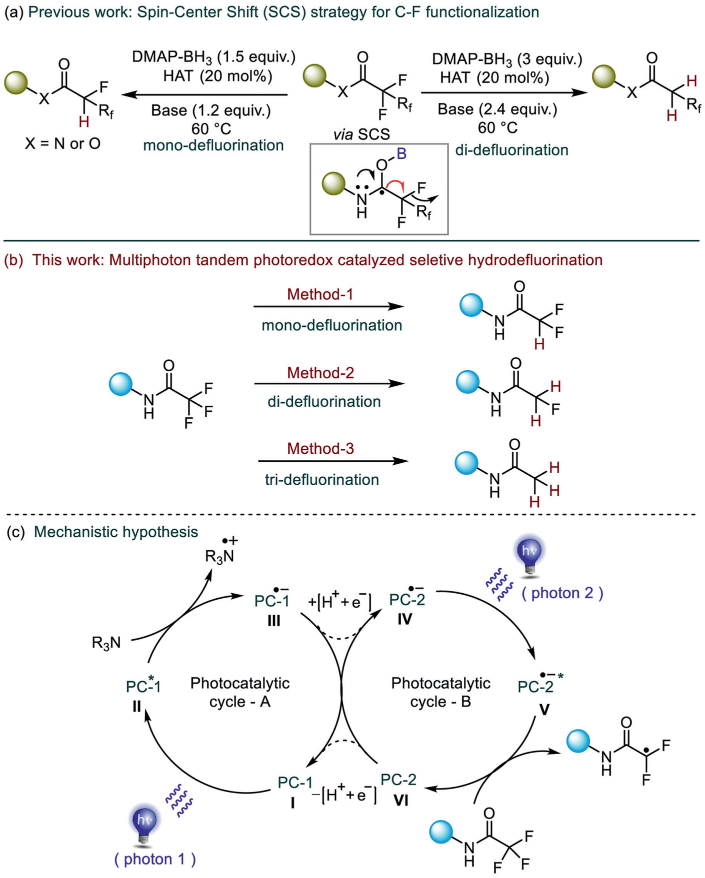
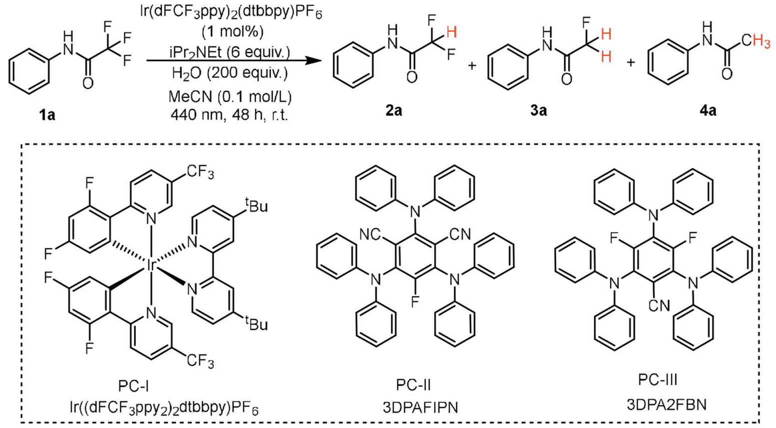


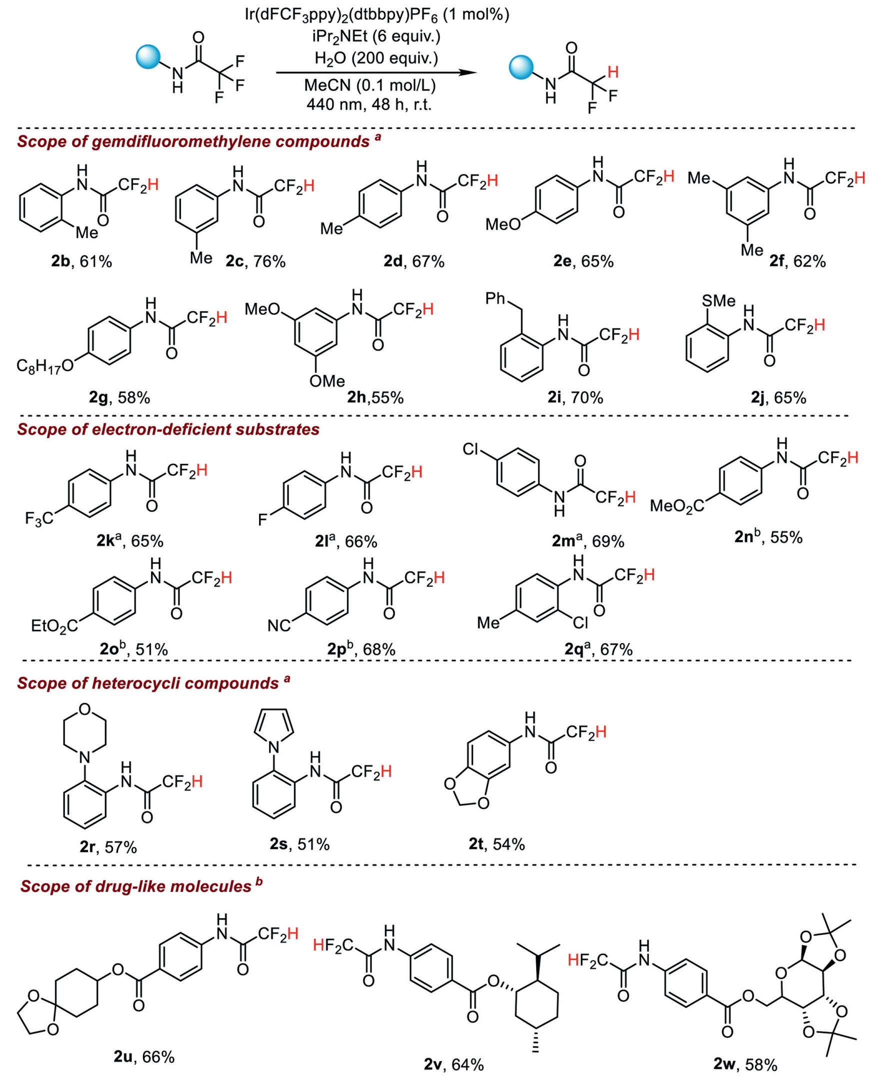
Scheme 1. Hydrodefluorination of trifluoroacetamides. (a) Spin-center shits for HDF. (b) Our work on HDF. (c) Proposed mechanistic hypothesis.

Table 1
Control experiments.[a]



Entry	Deviation from the standard condition	Yield of **2a:3a:4a** (%)
1	None	70:14:0
2	Absence of PC	NR
3	Absence of iPr_2NEt	NR
4	Absence of light	NR
5	W/O water	35:28:13
6	HEH (4 equiv.) instead of iPr_2NEt	NR
7	HCO_2Na (4 equiv.) instead of iPr_2NEt	NR
8	HCO_2Na (4 equiv.) and cyclohexanethiol (20 mmol%) instead of iPr_2NEt.	NR
9	$Ir(ppy)_3$ instead of PC-I	Trace
10	$Ir(ppy)_2(bpy)PF_6$ instead of PC-I	60:18:0
11	$Ir(ppy)_2(dtbbpy)PF_6$ instead of PC-I	65:15:0
12	PC-II instead of PC-I	47:32:0
13	PC-III instead of PC-I	38:40:0

[a] Reaction conditions: 0.1 mmol substrate, 1 mol% $Ir(dFCF_3ppy)_2(dtbbpy)PF_6$, 6 equiv. iPr_2NEt, 200 equiv. H_2O, 1 mL dry MeCN (0.1 mol/L), 440 nm, room temperature, 48 h, ^1H NMR yield using 1,3,5-trimethoxybenzene as internal standard.



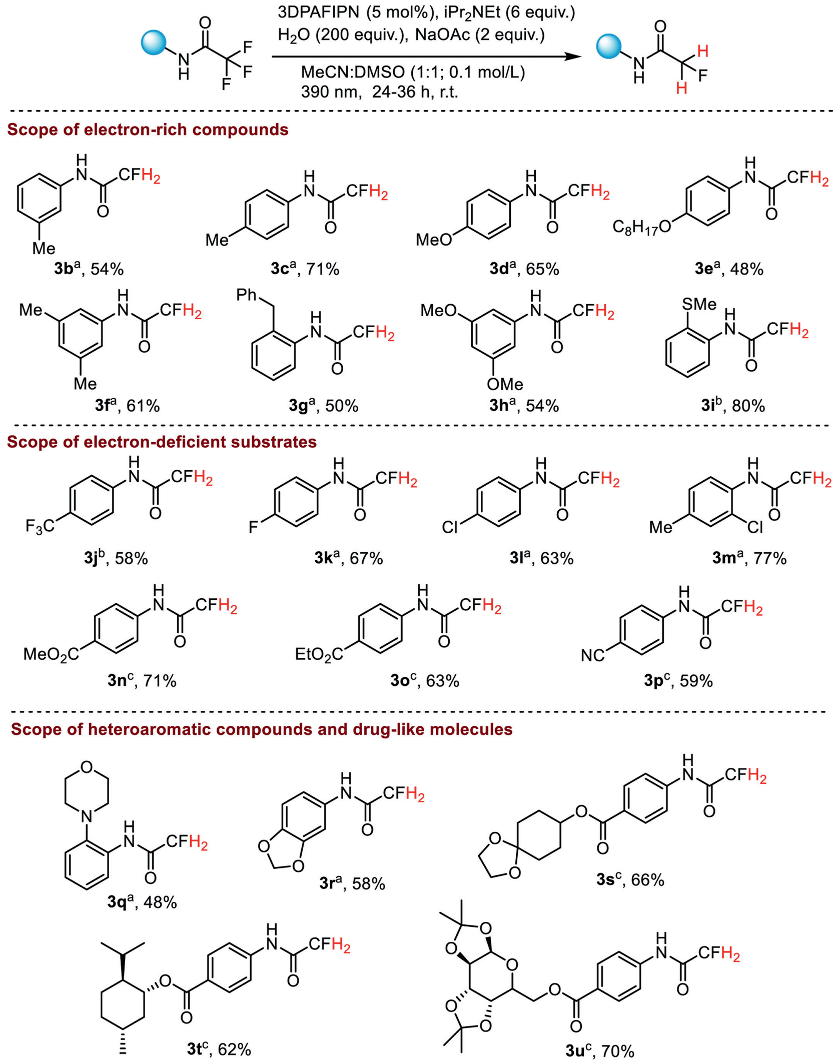
Scheme 2. Modular synthesis of *gem*–difluoromethylene compounds. Reaction conditions: [a] Ir(dFCF$_3$ppy)$_2$(dtbbpy)PF$_6$ (1 mol%), trifluoroacetamide (0.2 mmol, 1 equiv.), iPr$_2$NEt (1.2 mmol, 6 equiv.), and H$_2$O (40 mmol, 200 equiv.) in MeCN (0.1 mol/L) under argon, 440 nm, 48 h, room temperature. Yield of isolated product. [b] Ir(dFCF$_3$ppy)$_2$(dtbbpy)PF$_6$ (1 mol%), trifluoroacetamide (0.2 mmol, 1 equiv.), iPr$_2$NEt (0.4 mmol, 2 equiv.), NaOAc·3H$_2$O (0.2 mmol, 1 equiv.), HOAc (0.4 mmol, 2 equiv.) and H$_2$O (40 mmol, 200 equiv.) in dioxane (0.05 mol/L) under argon, 440 nm, 48 h, room temperature. Yield of isolated product. Detailed condition please see Supporting information.

to poor selectivity. We hypothesized that by employing this concept we may achieve a high selectivity for mono-, di, tri-HDF of trifluoroacetamide motifs. Thus, the successful accomplishment of the proposed concept would not only provide a novel protocol for the sequential HDF of trifluoroacetamide but would represent the first example of a selective defluorination using multiphoton photoredox catalysis and a conPET process (Schemes 1b and c).

At the start of our investigation, trifluoro-*N*-phenylacetamide was selected as a model substrate (**1a**) to investigate the selective hydrodefluorination and to form specifically monofluorinated, difluorinated, or fully hydrodefluorinated phenylacetamide. A detailed evaluation of the HDF reaction (complete optimization process given in Supporting information) demonstrated that Ir((dFCF$_3$ppy$_2$)$_2$dtbbpy)PF$_6$ was the optimal PC in combination with *N,N*-diisopropylethylamine (DIPEA) as a reductant and hydrogen atom donor to give the desired difluoro- (**2a**) and monofluoro- (**3a**) *N*-phenylacetamide in 70% and 14%, respectively (Table 1, entry 1). It is worth mentioning that the fully defluorinated product **4a** was not observed.

Control experiments revealed that light, PC, and the reductant are essential for this transformation (Table 1, entries 2–4). In the absence of water, the selectivity was significantly reduced, and **2a** was obtained in 35% NMR yield (Table 1, entry 5). Changing the reductant from DIPEA to Hantzsch ester failed to give the desired product (Table 1, entry 6). Previously reported photocatalytic procedures required sodium formate (HCOONa) and thiol as HAT substrates for the HDF [30,31]. Hence, we examined HCOONa and HCOONa/cyclohexane thiol as reductants instead of DIPEA. However, no product was observed (Table 1, entries 7 and 8). Interesting, our newly developed system operates with only DIPEA as a reductant for HDF. It is known that tertiary amines will be oxidized by the PC to form an amino radical cation that enables back electron transfer to deactivate the PC radical anion, which is one of the significant side reactions in this catalytic system [47]. Noteworthy, the tandem photoredox catalyst Ir((ppy)$_2$dtbbpy)PF$_6$ that was used for olefin reduction reaction in the previous report [39], and other Ir-PCs and organic dyes gave comparatively lower yield and selectivity than Ir((dFCF$_3$ppy$_2$)$_2$dtbbpy)PF$_6$ (PC-I) (Table 1, entries 9–13, Table S5 in Supporting information).

After successfully optimizing the reaction condition, the applicability of this protocol was tested with different trifluoroacetamide substrates for mono-defluorination, giving pharmaceuti-



Scheme 3. Scope of dihydrodefluorination. Reaction conditions: [a] 3DPAFIPN (5 mol%), trifluoroacetamide (0.2 mmol, 1 equiv.), iPr_2NEt (1.2 mmol, 6 equiv.), $NaOAc\cdot H_2O$ (2 equiv.) and H_2O (40 mmol, 200 equiv.) in MeCN and DMSO (0.1 mol/L, 1:1) under argon, 390 nm, 24–36 h, room temperature. Yield of isolated product. [b] $Ir(dFCF_3ppy)_2(dtbbpy)PF_6$ (1 mol%), trifluoroacetamide (0.2 mmol, 1 equiv.), iPr_2NEt (1.2 mmol, 6 equiv.), and H_2O (40 mmol, 200 equiv.) in MeCN (0.1 mol/L) under argon, 440 nm, 48 h, room temperature. Yield of isolated product. [c] $Ir(dFCF_3ppy)_2(dtbbpy)PF_6$ (1 mol%), trifluoroacetamide (0.2 mmol, 1 equiv.), iPr_2NEt (0.4 mmol, 2 equiv.), $NaOAc\cdot 3H_2O$ (0.2 mmol, 1 equiv.), KH_2PO_4 (0.2 mmol, 1 equiv.) and H_2O (40 mmol, 200 equiv.) in MeCN (0.05 mol/L) under argon, 440 nm, 36–48 h, room temperature. Yield of isolated product. For detailed description of the conditions please see Supporting information.

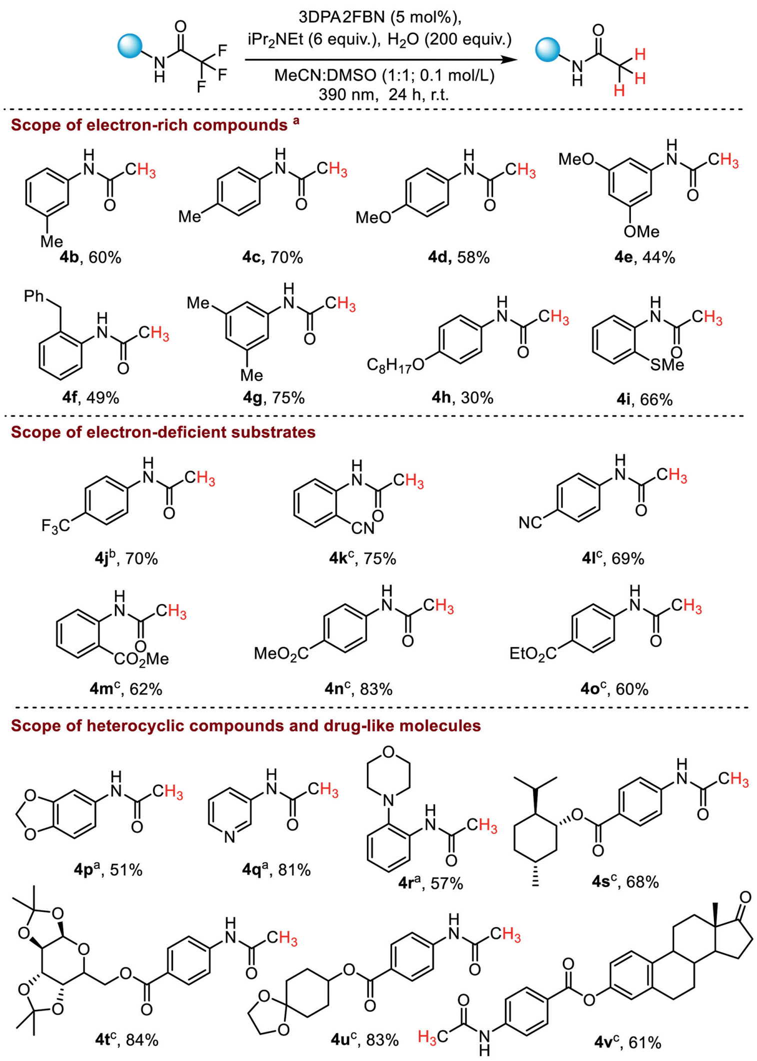
cally important *gem*–difluoromethylene analogues in good to excellent yield (Scheme 2). More challenging electron-neutral/donating trifluoroacetamide derivatives underwent this protocol to give *gem*–difluoromethylene acetamides in up to 76% yield (**2a-2g**). Interestingly, the developed protocol is also suitable for sterically crowded substrates (**2h-2j**). In addition, electron-deficient and heteroaromatic compounds containing CF_3- group were selectively mono-defluorinated to give desired compounds in good to excellent yields. Functional groups such as chloro (Cl), cyano (CN), S-Me, benzyl, and ester were tolerated (**2i**, **2j**, **2m-2p**). Synthetically important drug-like molecules could be applied and gave the desired product in 58%–64% yields with good selectivity (**2u-2w**). In the case of electron-withdrawing and complex molecules, 2 equiv. of base are sufficient for the conversion, while sodium acetate and acetic acid were added for the improvement of selectivity (Table S2 in Supporting information).

The scope of fluoroacetamide was also investigated with slightly different conditions. Interestingly, organic dye 3DPA2FIPN (PC-II) was a suitable catalyst for dihydrodefluorination. The optimal combination with DMSO:MeCN as a solvent at 390 nm gave more than 70% selectivity (detailed optimization see Tables S3 and S4 in Supporting information). Likewise, both electron-donating and electron-withdrawing groups containing trifluoroacetamides underwent dihydrodefluorination smoothly and furnished the desired products in good to high yields (Scheme 3).

This protocol also has good functional group compatibility (**3b-3r**). Indeed, late-stage functionalization of complex molecules, especially in a selective fashion, is a challenging and key component in drug-discovery research. In this context, compounds **3s-3u** were synthesized in 62%–70% yield.

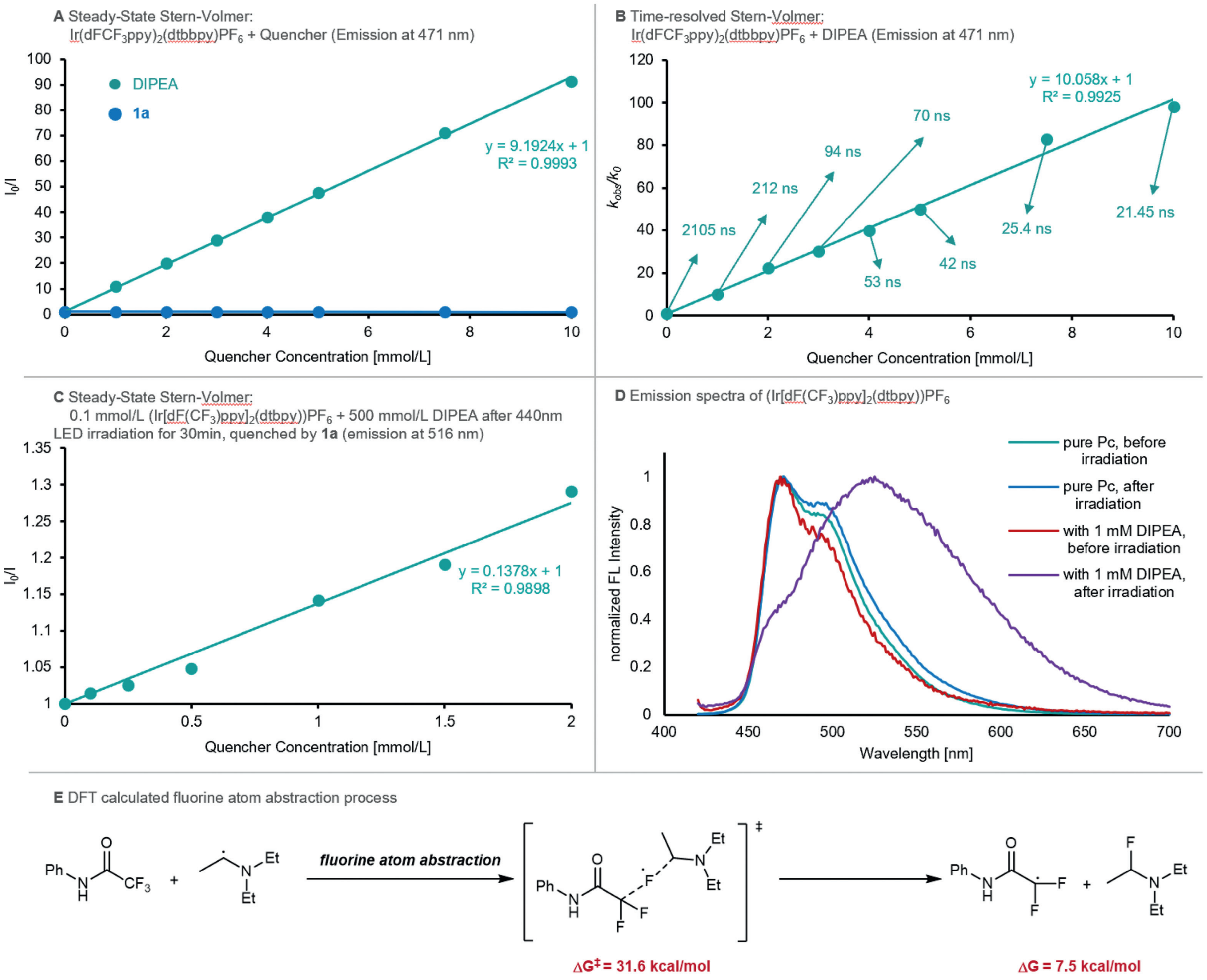
Next, we explored the complete hydrodefluorination of the trifluoroacetamide derivatives. In this section, 3DPA2FBN (PC-III) or Ir-PC-I was used depending on the electronic nature of the substrates. Similarly, wavelength, solvent, and reaction time play a crucial role in complete defluorination (Tables S3 and S4). Some sensitive functional groups were unaffected by the reaction (Scheme 4, **4f–4r**). Bioactive compounds such as steroid derivatives gave desired product in 61% yield (**4s-4v**).

Essential components within photoredox catalysis, encompassing processes like multiphoton excitation and electrophotocataly-



Scheme 4. Divergent synthesis of acetamides from trifluoroacetamides. Reaction conditions: [a] 3DPA2FBN (5 mmol%), trifluoroacetamide (0.2 mmol, 1 equiv.), iPr_2NEt (1.2 mmol, 6 equiv.) and H_2O (40 mmol, 200 equiv.) in degassed MeCN and DMSO (0.1 mol/L, 1:1) under argon, 390 nm, 24 h, room temperature. Yield of isolated product. [b] 3DPAFIPN (5 mol%), trifluoroacetamide (0.2 mmol, 1 equiv.), iPr_2NEt (1.2 mmol, 6 equiv.), $NaOAc\cdot H_2O$ (2 equiv.) and H_2O (40 mmol, 200 equiv.) in MeCN and DMSO (0.1 mol/L, 1:1) under argon, 390 nm, 24–36 h, room temperature. [c] $Ir(dFCF_3ppy)_2(dtbbpy)PF_6$ (1 mol%), trifluoroacetamide (0.2 mmol, 1 equiv.), iPr_2NEt (1.2 mmol, 6 equiv.), and H_2O (40 mmol, 200 equiv.) in MeCN (0.1 mol/L) under argon, 440 nm, 48 h, room temperature. Yield of isolated product. Yield of isolated product. For detailed description of the conditions please see Supporting information.

sis, have been proposed to involve photosensitizers reduced by a single electron. Nonetheless, the study of these diminished chromophores has encountered certain limitations, which has hindered a thorough investigation into the complicated mechanisms of the electron transfer processes they encompass. In this particular scenario, Knowles group [42] has recently disseminated research outcomes pertaining to iridium photocatalysts that have been singularly reduced and can be isolated. The disclosed spectroscopic characteristics of these catalysts, emphasizing reduction centered on ligands, provide compelling support for our hypothesis. Based on the previous reports [34,39,40,42] and the proposed hypothesis, we conducted quenching studies with Ir-PC and organic dyes. The Ir-photocatalyst (PC-I) is exhibiting an excited state redox potential of $E_{1/2}(\mathrm{Ir^{III}}{*}/\mathrm{Ir^{II}}) = +1.21$ V *vs.* SCE, which can potentially oxidize the DIPEA ($E_{1/2} = +0.78$) *vs.* SCE (MeCN) [48] to its radical amino cation and to form the reduced photocatalyst ($E_{1/2}(\mathrm{Ir^{II}}/\mathrm{Ir^{III}}) = -1.37$) (Scheme 1c, **II**), as supported by fluorescence quenching experiments (Scheme 5A). Thermodynamically, the resulting photo-excited catalyst (Scheme 1c, **III**) cannot reduce the trifluoroacetamide (**1a**) *via* SET because of the reduction potential of the substrate ($E_{1/2} = -2.26$ V *vs.* SCE), which is almost a 1 V difference with the photocatalyst, a result further supported by emission quenching studies (Scheme 5B). The substrate (**1a**) does not interact with the excited $\mathrm{Ir^{III}}{*}$ (PC-1*), while DIPEA quenched the excited state PC-1* (Schemes 5A and B). The involvement of the second excited state of $[\text{PC-2}^{\cdot-}{*}]$ was demonstrated by emission quenching studies, where PC-I with 500 mmol/L of DIPEA was irradiated at 440 nm light. The resulting slow quenching rate of substrate **1a** possibly arises from the poor solubility of trifluoroacetamide (Scheme 5C). However, the new emission spectra shift was observed from 470 nm to 522 nm, indicating the formation of $[\text{PC-2}^{\cdot-}{*}]$ (Scheme 5D). Further, to support the high reduction potential of the *in situ* formed $[\text{PC-2}^{\cdot-}{*}]$, 1,1-diphenylethylene ($E_{1/2} = -2.25$ V *vs.* SCE) [39] was tested as a quencher. The resulting Stern-Volmer plot was linear, confirming the interaction between the $[\text{PC-2}^{\cdot-}{*}]$ and 1,1-diphenylethylene (Fig. S10 in Supporting information). Like Ir-PC (PC-1), the organic dyes 3DPA2FBN and 3DPAFIPN were quenched with DIPEA, confirming the excited state PC-1* interacted with DIPEA and also yielding linear Stern-Volmer plots. In addition, the emission spectra of both organic dyes also



Scheme 5. Mechanistic studies. (A) Emission quenching of PC-I with a DIPEA and PC-I with substrate **1a**. (B) Time resolved emission quenching of PC-I with a DIPEA. (C) Stern-Volmer plot of PC-I+DIPEA after irradtion and quenching with substrate **1a**. (D) Emission spectra of PC-I with or without DIPEA before and after irradiation. (E) DFT calculated fluorine atom abstraction process. Free energies in solution (kcal/mol) calculated at SMD (MeCN)-M06–2X/Def2-TZVPP//ωB97XD/Def2-SVP.

shifted towards the lower wavelength region (Figs. S13 and S16 in Supporting information); this might be the reason that di- and tri-defluorination occurred at 390 nm.

The bond dissociation energy (BDE) of an α-C-H bond of DIPEA [48] radical cation is 90.7 kcal/mol which is relatively low compared to that of the C-F bond (BDE = 118.1 kcal/mol) in trifluoroacetamide [49]. Hence, halogen atom abstraction is not likely to operate in the mechanistic cycle, which is further supported by density functional theory (DFT) calculation (Scheme 5E). The calculation showed that the fluorine atom abstraction by α-amino radical is kinetically (31.6 kcal/mol energy barrier) and thermodynamically (7.5 kcal/mol less stable) unfavored. Next, we examined the source of hydrogen atoms (DIPEA, H_2O, and MeCN) for HDF. The reaction was conducted using model substrate **1a** under condition **A** with the addition of D_2O instead of H_2O. The product **2a** was obtained in 59% isolated yield along with 15% mono deuterium incorporated **3a** (Fig. S2). A similar tendency was noted in the presence of D_2O and CD_3CN. Therefore, we conclude that the major hydrogen atom comes from DIPEA which also acts as an effective hydrogen atom donor for the formation of PC-2 *via* reduction and semisaturation of the ancillary dtb-bpy ligand.

In conclusion, we have developed a novel and straightforward methodology for the site-selective hydrodefluorination of trifluoroacetamides. Applying this process, a wide range of molecules have been selectively defluorinated to give mono-, di-fluoro substituted acetamides and simple acetamide derivatives. This protocol also functions for late-stage functionalization of drug derivatives. Mechanistic and spectroscopic studies demonstrate that the multiphoton tandem photoredox catalyst reaches a high reduction potential under irradiation, allowing it to carry out selective transformations in a controlled manner.

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.

CRediT authorship contribution statement

Jiaqi Jia: Methodology, Writing – original draft, Writing – review & editing, Conceptualization, Data curation, Formal analysis, Investigation. **Kathiravan Murugesan:** Writing – original draft, Writing – review & editing. **Chen Zhu:** Investigation, Writing – original draft, Writing – review & editing. **Huifeng Yue:** Investigation. **Shao-Chi Lee:** Investigation. **Magnus Rueping:** Writing – review & editing, Funding acquisition, Project administration, Resources, Supervision.

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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.2024.109866.

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