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A visible-light-promoted perfluoroalkylation/cyclization cascade towards perfluoroalkyl-substituted iminoisobenzofurans using the EDA complex

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Perfluoroalkyl-functionalized heterocycles exhibit profoundly modified bioactivities, motivating the development of efficient methods for precise perfluoroalkyl group installation. A new metal-free photoinduced radical-initiated cascade reaction was developed for the rapid synthesis of perfluoroalkyl-substituted iminoisobenzofurans by reacting various perfluoroalkyl iodides (R_F-I) under mild reaction conditions. In this reaction system, the formation of electron donor–acceptor (EDA) complexes is pivotal for the visible-light promoted transformation in the absence of additional photocatalytic species.

Introduction

Fluoroalkylated scaffolds are pivotal structures in pharmaceuticals and materials science, as they can significantly alter the physical and chemical properties of molecules, such as enhancing lipophilicity, bioavailability and metabolic stability, making them indispensable in drug design and material development.[1] Consequently, the exploration of new ways to incorporate fluoroalkyl groups into organic molecules has become a major topic in chemical research.[2] While research efforts have predominantly focused on difluoromethyl and trifluoromethyl group incorporation, significant progress has also been made in longer-chain perfluoroalkylation (C_nF_{2n+1}, $n \geq 2$) through various approaches including electron donor–acceptor (EDA) complex-mediated methodologies.[3] However, challenges remain in developing general and practical methods that accommodate diverse substrates under mild reaction conditions. As pivotal feedstocks in the chemical industry, radical-initiated fluoroalkylation of unsaturated C=C bonds in alkenes has emerged as a privileged synthetic strategy for accessing fluorinated compounds.[4,5] Over the past decade, alkene-based tandem fluoroalkylation/cyclization reactions have attracted substantial research attention in synthetic chemistry owing to their exceptional efficiency in constructing fluoroalkyl-substituted carbocyclic and heterocyclic motifs.[6,7] Mechanistically, these transformations involve the generation of reactive fluoroalkyl radical intermediates from diverse fluoroalkyl precursors under transition-metal catalysis, thermal activation, photoredox catalysis, or electrochemical conditions. These fluoroalkyl radicals subsequently undergo regioselective addition to the alkene π-system, followed by an intramolecular cascade cyclization reaction with tethered nucleophiles to form cyclic molecules bearing fluoroalkyl substituents (Scheme 1a). Fluoroalkyl iodides serve as readily available and economical precursors for generating fluoroalkyl radicals that efficiently undergo addition to alkene C=C bonds, establishing a fundamental platform for developing diverse synthetic methodologies.[8]

The isobenzofuran motif serves as a key structural feature in numerous natural products and bioactive molecules.[9] Given its significance, researchers have developed various efficient strategies for constructing this scaffold. Among these derivatives, iminoisobenzofurans have gained increasing attention from synthetic chemists in recent years as particularly pivotal structures.[10] Notably, radical-mediated tandem addition/cyclization strategies have emerged as innovative approaches for constructing functionalized iminoisobenzofurans from *ortho*-vinyl amides (Scheme 1b).[11] Representative transformations in this domain include (a) $K_2S_2O_8$-promoted thiocyano cyclization, (b) electrochemical oxidative seleno/thio-cyclization, and (c) visible-light-induced sulfonamidylative cyclization. While methods for incorporating fluoroalkyl groups into iminoisobenzofuran frameworks remain limited, significant progress has been made in recent years (Scheme 1c). In 2018, the Xiao group pioneered a visible-light mediated photocatalytic radical addition/cyclization reaction between *o*-vinyl-*N*-alkoxybenzamides and Umemoto's reagent.[12] Building upon this work, we recently reported a copper-catalyzed radical addition/cyclization cascade of *o*-alkenyl-*N*-alkylbenzamides with Togni's

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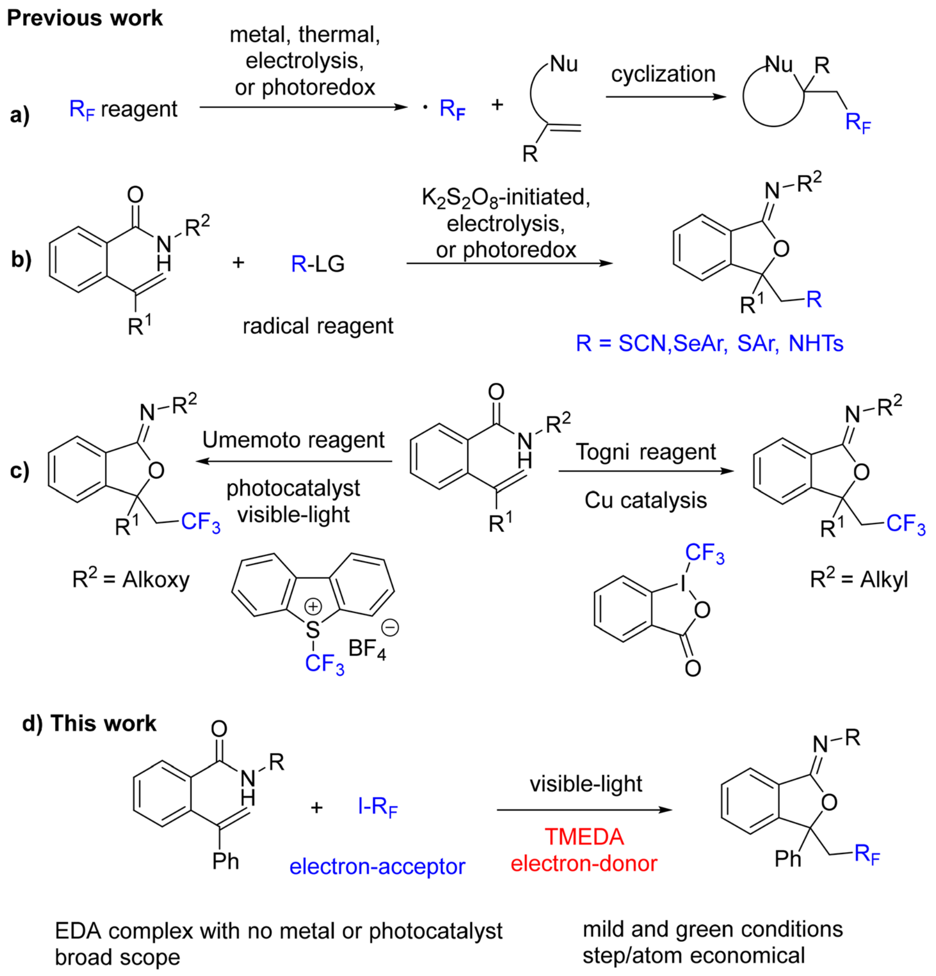


Scheme 1 Strategies for synthesizing perfluoroalkyl-substituted iminoisobenzofurans.

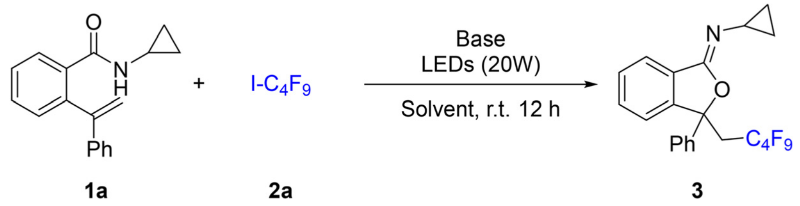
reagent, which provides complementary access to diverse CF_3-containing iminoisobenzofuran analogs.[13]

Visible-light-induced reactions *via* EDA complexes in the absence of organic and metal photocatalysts have emerged as an increasingly important research focus since 2000.[14] In these studies, perfluoroalkyl iodides assemble into EDA complexes with electron-rich species through electrostatic stabilization. Photoexcitation of these complexes induces intramolecular electron transfer from heteroatom lone pairs to the C–I σ* antibonding orbital of R_F-I, thereby generating reactive perfluoroalkyl radicals.[15] This innovative strategy eliminates the need for exogenous photocatalysts while maintaining exceptional selectivity under visible-light irradiation, representing a significant advancement in sustainable synthetic methodologies. For instance, Yu achieved efficient synthesis of perfluoroalkyl-substituted benzimidazo[2,1-*a*]isoquinolin-6(5*H*)-ones and indolo[2,1-*a*]isoquinolin-6(5*H*)-ones through a visible-light-induced tandem radical addition/cyclization strategy, using an EDA complex formed between perfluoroalkyl iodides and *N*,*N*,*N*′,*N*′-tetramethylethane-1,2-diamine (TMEDA).[16] Building upon a similar strategy, Yang *et al.* established a photocatalytic protocol involving a radical perfluoroalkylation/cyclization cascade to construct polycyclic quinazolinones containing perfluoroalkyl groups from *N*-cyanamide alkenes and perfluoroalkyl iodides.[17] Inspired by these elegant precedents, we herein report a radical cascade addition/cyclization reaction of *o*-alkenylbenzamides using an EDA complex under visible-light irradiation for constructing perfluoroalkylated iminoisobenzofuran derivatives (Scheme 1d).

Results and discussion

We initially chose *o*-alkenylbenzamide **1a** as a model substrate and C_4F_9I **2a** as the perfluoroalkyl radical precursor under visible-light irradiation to investigate the cyclization (Table 1). We found that in the presence of *N*,*N*,*N*′,*N*′-tetramethylethane-1,2-diamine (TMEDA) under 420–430 nm LED irradiation for 12 h, this reaction proceeded in MeCN to generate the C_4F_9-substituted iminoisobenzofuran **3a** in 81% yield (entry 1). Further optimization using different organic bases, including Et_3N, DBU, DABCO, and Cs_2CO_3, demonstrated that other bases were less effective (entries 2–5). We then examined the effect of irradiation wavelength on the

Table 1 Optimization of reaction conditions[a]



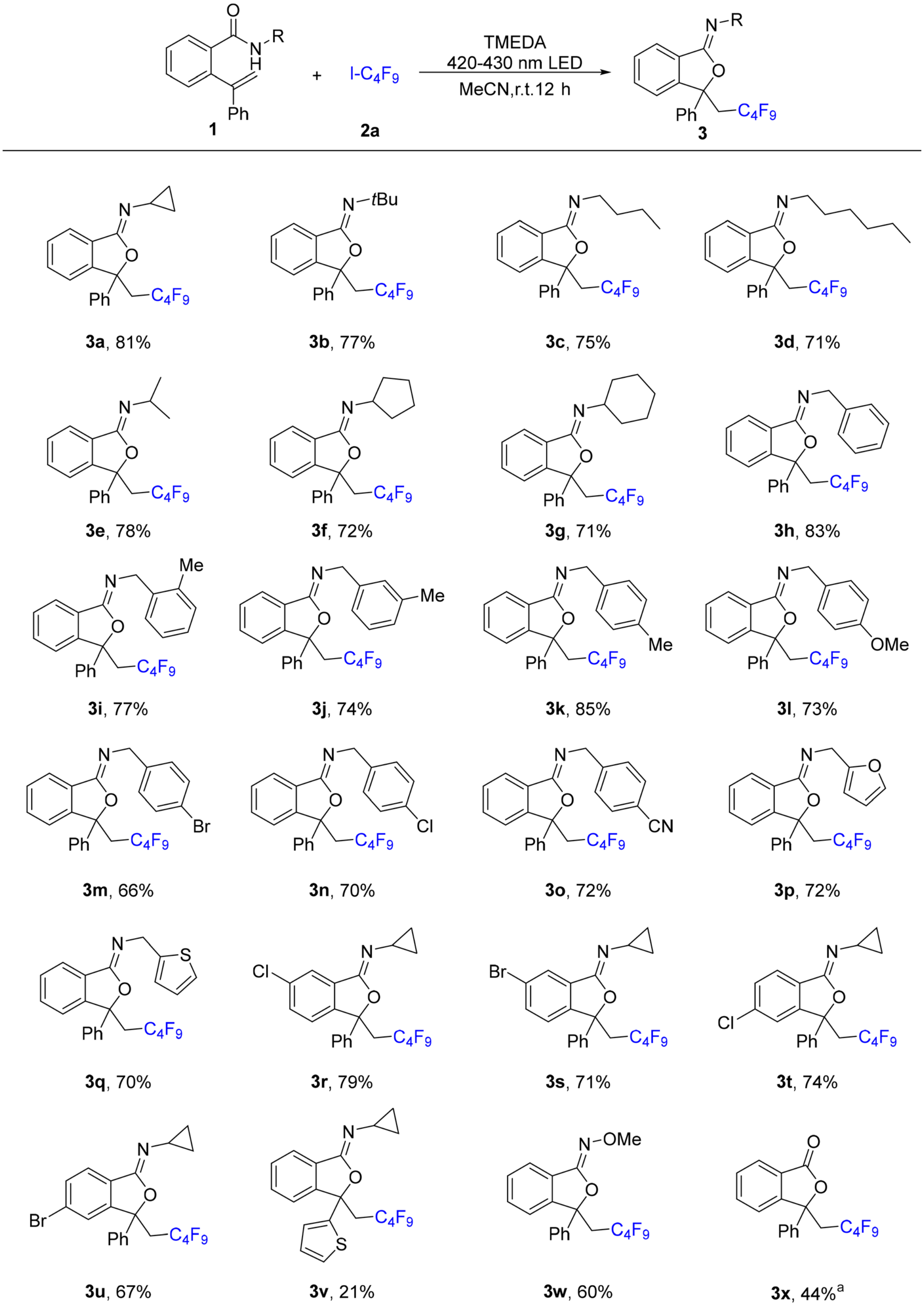
Entry	Base	LED	Solvent	Yield[b] (%)
1	TMEDA	420–430 nm	MeCN	81
2	Et_3N	420–430 nm	MeCN	42
3	DBU	420–430 nm	MeCN	65
4	DABCO	420–430 nm	MeCN	75
5	Cs_2CO_3	420–430 nm	MeCN	28
6	TMEDA	380–390 nm	MeCN	79
7	TMEDA	520–530 nm	MeCN	Trace
8	TMEDA	400–800 nm	MeCN	56
9	TMEDA	420–430 nm	THF	35
10	TMEDA	420–430 nm	DCE	45
11	TMEDA	420–430 nm	DMSO	53
12	TMEDA	420–430 nm	1,4-Dioxane	20
13	TMEDA	420–430 nm	MeOH	41
14[c]	TMEDA	420–430 nm	MeCN	53
15	—	420–430 nm	MeCN	0
16	TMEDA	Dark	MeCN	0

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), base (0.4 mmol), in dry solvent (2.0 mL) under N_2 at room temperature with LED irradiation for 12 h. [b] Isolated yield based on **1a**. [c] TMEDA (0.2 mmol) was added. DABCO, 1,4-diazocyclic [2.2.2] octane; DBU, 8-diazabicyclo [5.4.0] undec-7-ene; THF, tetrahydrofuran; DCE, dichloroethane; and DMSO, dimethyl sulfoxide.

reaction using LEDs with different wavelengths. Notably, the reaction maintained a comparable yield when a 380–390 nm LED was employed. However, a significant yield reduction to 56% was observed when a broader-spectrum 400–800 nm LED was used, and the yield showed a marked decline at longer wavelengths (entries 6–8). Substituting the solvent MeCN with THF, DCE, DMSO, 1,4-dioxane or methanol failed to significantly enhance the reaction yield (entries 9–13). When the amount of TMEDA was reduced to 0.2 mmol, the yield decreased to 53% (entry 14). Finally, the control experiments confirmed that both light irradiation and TMEDA were essential for this perfluoroalkylation/cyclization (entries 15 and 16).

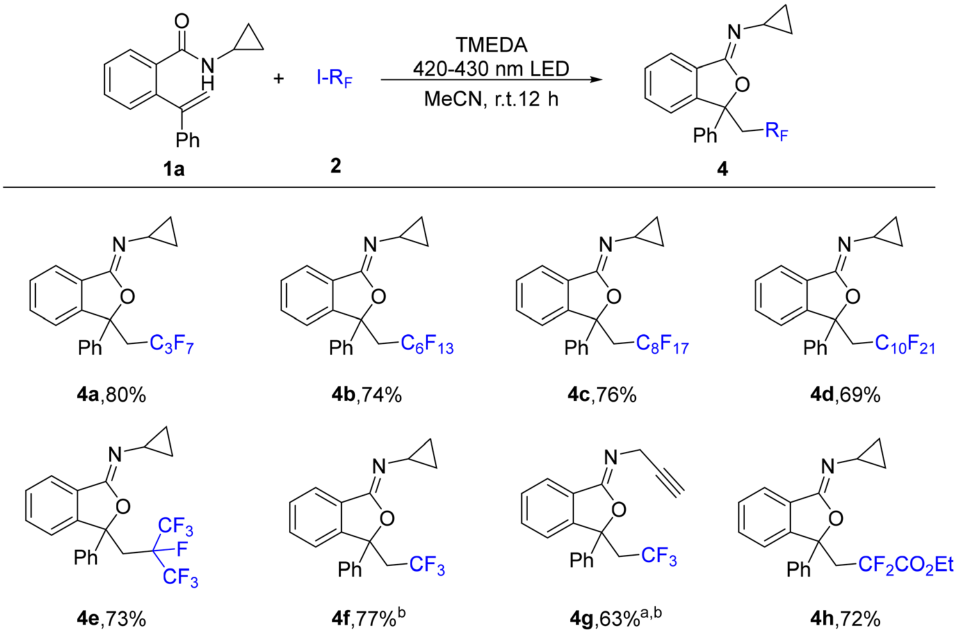
Upon establishing the optimal reaction conditions, we subsequently examined the substrate generality of the photo-induced perfluoroalkylation/cyclization tandem process with various *o*-alkenyl amide derivatives (Scheme 2). Initially, a broad range of amides bearing diverse alkyl (*tert*-butyl, *n*-butyl, *n*-hexyl, and isopropyl) and cycloalkyl (cyclopentyl and cyclohexyl) substituents all underwent the desired transformation with good to excellent yields (**3b–3g**). Notably, the *N*-benzylamide substrate showed enhanced reactivity, providing the target product **3h** in a higher yield (83%). Furthermore, the reaction exhibited excellent functional group tolerance towards various *N*-benzyl-substituted amides, with electron-donating (methyl and methoxy) and electron-withdrawing (bromo, chloro, and cyano) substituents on the phenyl ring all proving compatible, yielding products **3i–3o** in 66–85% yields. Next, when the benzene ring was replaced with furan or thiophene heterocycles, the corresponding perfluoroalkylated iminoisobenzofurans **3p** and **3q** were obtained in 72% and 75% yields, respectively. Also, substrates bearing chloro or bromo substituents at different positions on the benzene ring connected to the amide group also afforded the desired products **3r–3u** in 67–79% yields. However, the tandem reaction efficiency significantly decreased when the benzene ring was replaced with a thiophene moiety (**3v**, 21%). The conversion yield decreased significantly when the alkyl group in the substrate was replaced with an alkoxy group (**3w**, 60%). Finally, when *o*-alkenyl-substituted benzoic acid was used as the substrate, the C_4F_9-containing isobenzofuran product **3x** was obtained in 44% yield.

Following the evaluation of the substrate scope with *o*-alkenyl amides, we further investigated the reactivity of diverse perfluoroalkyl radical precursors in the cyclization reaction under optimized conditions (Scheme 3). Systematic screening of commercially available perfluoroalkyl iodides (IC_3F_7, IC_6F_{13}, IC_8F_{17}, $IC_{10}F_{21}$, and $ICF(CF_3)_2$) revealed that all derivatives could effectively participate in the transformation, consistently delivering perfluoroalkyl-substituted iminoisobenzofurans (**4a–4e**) in excellent yields (69–80%). Encouraged by these results, we subsequently examined the reactivity of trifluoroiodomethane (CF_3I) and ethyl difluoroiodoacetate (ICF_2COOEt) with *o*-alkenylbenzamide substrates. Remarkably, employing CF_3I as the trifluoromethylation reagent under the standard conditions afforded the corresponding trifluoro-

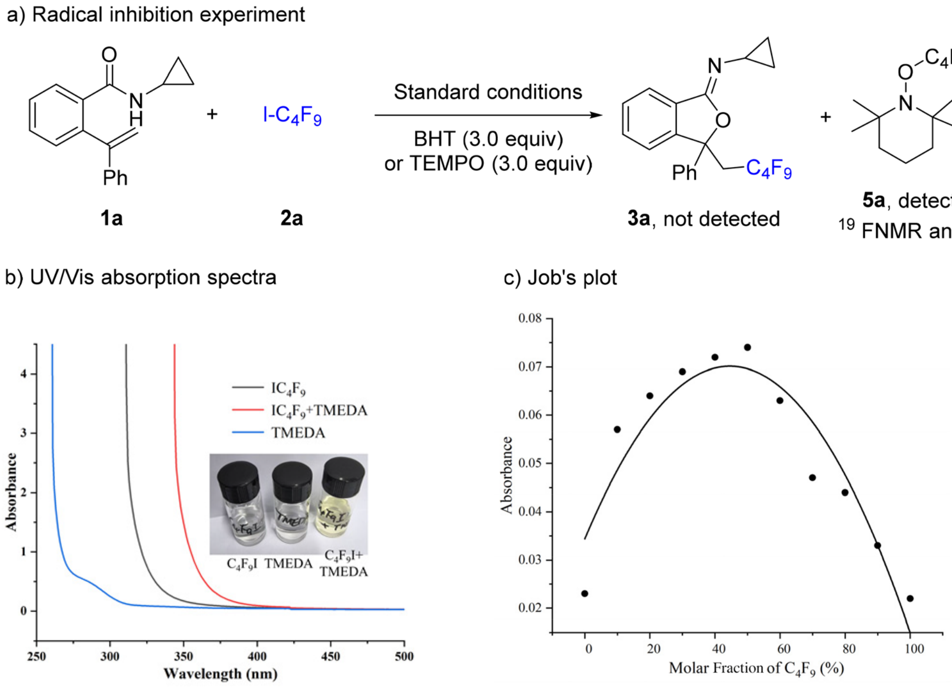


Scheme 2 Substrate scope of amides. Reaction conditions: **1** (0.2 mmol), **2a** (0.4 mmol), TMEDA (0.4 mmol), in dry MeCN (2.0 mL) under N_2 at room temperature with 420–430 nm LED irradiation for 12 h. Isolated yield. [a] *o*-Alkenylbenzoic acid was used.

methylated products **4f** and **4g** in 77% and 63% yields, respectively, demonstrating the versatility of this radical cascade process. Finally, the reaction with ICF_2COOEt as the radical precursor was performed under standard conditions, which successfully afforded the difluoroalkylated product **4h** with good compatibility.

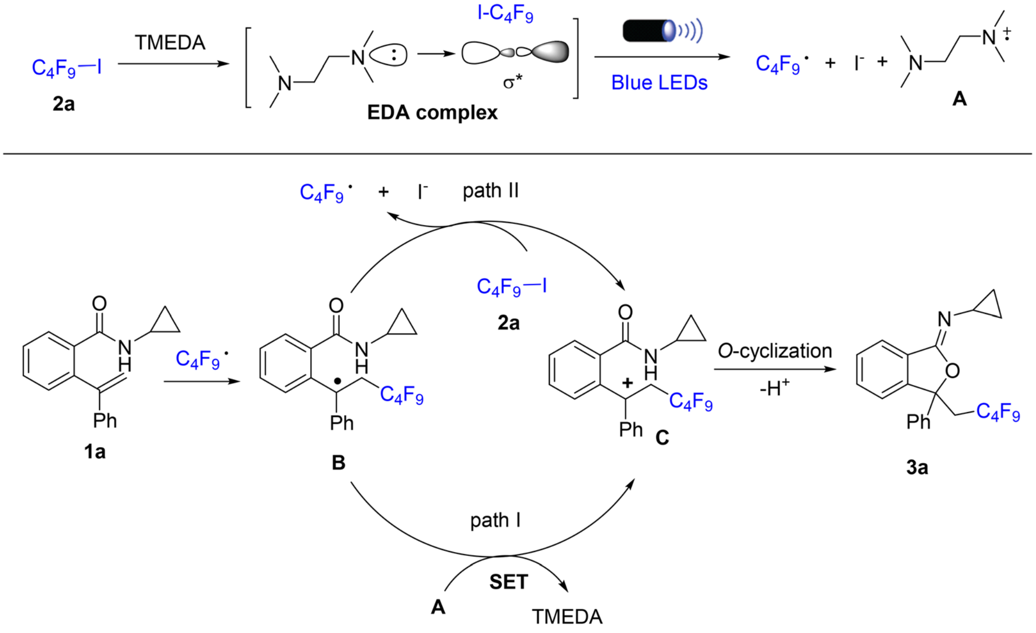


Scheme 3 Substrate scope of perfluoroalkyl iodides. Reaction conditions: **1a** (0.2 mmol), **2** (0.4 mmol), TMEDA (0.4 mmol), in dry MeCN (2.0 mL) under N_2 at room temperature with 420–430 nm LED irradiation for 12 h. Isolated yield. [a] 25 wt% CF_3I solution in THF was used. [b] *N*-Propargyl-substituted amide was used.



Scheme 4 Investigation of the reaction mechanism.

To further elucidate the reaction mechanism of the visible-light-promoted perfluoroalkylation/cyclization, we conducted several control experiments. Initially, when 3.0 equivalents of either 2,6-di-*tert*-butyl-4-methylphenol (BHT) or 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) were introduced under standard reaction conditions, the process was completely suppressed (Scheme 4a). The corresponding free radical adduct **5a** was also identified by HRMS and ^{19}F NMR, and these findings strongly suggested the crucial role of radical intermediates in this transformation. Scheme 4b displays the UV-vis absorption spectral changes of C_4F_9I (0.1 M), TMEDA (0.1 M), and their mixture in acetonitrile solution. Upon mixing the two components, the



Scheme 5 Possible reaction pathway.

solution color gradually changed from colorless to yellow, accompanied by a distinct bathochromic shift in the absorption spectrum. In addition, Job's plot analysis showed that the EDA complex was composed of C_4F_9I and TMEDA in a 1 : 1 ratio (Scheme 4c). The measured quantum yield (Φ) of 0.46 (see the SI) suggested that the reaction may not proceed through a highly efficient radical chain process, although the possibility of chain propagation cannot be completely excluded, given potential inefficiencies in the initiation step.

Based on the aforementioned mechanistic studies and previous literature,[15–17] a plausible reaction mechanism is illustrated in Scheme 5. Initially, TMEDA formed an EDA complex with C_4F_9I, which underwent dissociation under blue light irradiation to generate the C_4F_9 radical, radical cation **A**, and iodide ion. Subsequently, the C_4F_9 radical attacked the C═C bond of amide **1a**, forming the radical intermediate **B**. This key intermediate then underwent two possible pathways to form the benzylic carbocation C: (a) direct oxidation *via* SET with TMEDA radical cation **A** (path I) or (b) regeneration of the perfluoroalkyl radical and iodide anion through a SET mechanism in the presence of **2a** (path II). Ultimately, the desired product **3a** was formed through intramolecular nucleophilic attack, followed by deprotonation.

Conclusions

In conclusion, we have developed a novel and efficient visible-light-induced radical cyclization for the synthesis of perfluoroalkyl-substituted iminoisobenzofuran derivatives. This transformation employs commercially available perfluoroalkyl iodides and *o*-alkenyl benzamides as starting materials, where the iodides serve as radical precursors through the formation of EDA complexes with TMEDA under photoirradiation conditions. This one-pot protocol offers significant advantages including operational simplicity under mild conditions, photocatalyst-free conditions that eliminate the need for expensive and potentially toxic transition metal complexes, and excellent functional group tolerance with broad substrate scope. These distinctive features make this methodology particularly attractive for the construction of valuable fluorinated heterocycles with potential bioactivity.

Author contributions

Zilin Liu: investigation, data curation, and methodology. Shuo Gao: investigation and data curation. Mingxi Hu: data curation. Zhen-Hua Zhang: writing – review & editing, supervision, funding acquisition, and conceptualization. Pifeng Wei: writing – original draft. Mengmeng Zhao: supervision and writing – review & editing.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

The Supplementary Information file contains experimental details, characterization data (including NMR spectra), and additional supporting results referenced throughout the manuscript. See DOI: https://doi.org/10.1039/d5ob00888c

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