

Metal-Free Visible-Light Promoted Radical Cyclization to Access Perfluoroalkyl-Substituted Benzimidazo[2,1-*a*]isoquinolin-6(5*H*)ones and Indolo[2,1-*a*]isoquinolin-6(5*H*)-ones

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Abstract: A metal-free visible-light-induced cyclization procedure was developed for the rapid synthesis of perfluoroalkyl-substituted benzimidazo[2,1-*a*]isoquinolin-6(5H)-ones and perfluoroalkyl-substituted indolo [2,1-*a*]isoquinolin-6(5H)-ones under mild reaction conditions. In this procedure, the formation of electron-donor-acceptor (EDA) complex is critical for the visible-light promoted process to avoid the utilization of external photocatalysts.

Keywords: Visible-light; Radical cascade reaction; Metal free; Photocatalyst free; Perfluoroalkylation

Introduction

Nitrogen-containing heterocycles are important and promising compounds due to their potential biological activities.^[1] Consequently, numerous procedures have been developed for the construction of N-heterocycles in the past decades.^[2] On the other hand, in the recent years visible-light-promoted synthesis has emerged as a powerful and environmentally friendly strategy for various organic transformations.^[3] Particularly, the synthesis of N-heterocycles under visible-light-mediated conditions have been well developed recently.^[4] Notably, benzimidazo-isoquinolin-6(5H)-ones (A) as a significant structural scaffold was widely found in biologically active molecules and functional materials.^[5] Figure 1 shows some representative compounds containing benzimidazo-isoquinolin-6(5H)-one scaffold with various pharmacological activities, such as anti-inflammatory, antidiabetic.^[6]

As a consequence of its importance, substantial efforts have been contributed to the construction of benzimidazo-isoquinolin-6(5H)-ones. For instance, the



Figure 1. Selected examples of benzimidazo-isoquinolin-6(5*H*)-ones.

conventional condensation methods were found to be efficient for the construction of the target polycycles.^[7] However, those reactions suffer from tedious prefunctionalization steps, harsh reaction conditions as well as practical inconvenience. Gratifyingly, transition-metal catalysis strategies have been reported as promising alternatives for the synthesis of this important scaffold

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Previous work:



Scheme 1. Synthesis of benzimidazo[2,1-*a*]isoquinoline-6(5*H*)-ones and indolo[2,1-*a*]isoquinolin-6(5*H*)-ones.

in recent years. For example, Song and co-workers reported an elegant Cp*Rh(III)-catalyzed [4+2] annulation reaction of 2-arylimidazoles and a-diazoketoestaccess benzimidazole[2,1-*a*]isoquinolines ers to (Scheme 1a).^[8] Recently, our group developed an efficient AgNO₃ catalyzed decarboxylative cascade cyclization reaction for the construction of alkylated/ benzimidazo[2,1-a]isoquinolin-6(5H)-ones acvlated (Scheme 1b).^[9] Despite these achievements, it should be pointed out that these methods still suffer from some drawbacks, including the requirement of expensive metal catalysts, stoichiometric oxidant and high reaction temperatures. In the standpoint of green and sustainable organic synthesis,^[10] the development of straightforward, convenient and eco-friendly synthetic methods for the preparation of structurally diverse benzimidazo-isoquinolin-6(5H)-ones is of significance and highly desired.

Fluorinated functional groups are increasingly applicable in the fields of drugs, agrochemical indus-tries as well as material sciences.^[11] In particularly, the introduction of fluorinated alkyl moiety to heterocycle scaffolds has a profound impact on their physicochemical and biological properties, resulting in enhanced lipophilicity, bioavailability and metabolic stability.^[12] Among those fluorine-containing functional groups, perfluoroalkyl groups (R_f) are versatile and valuable of potential moieties for the development pharmaceuticals.^[13] Consequently, the development of efficient and convenient protocols for the installation of R_f into target molecules is highly desired. However, examples that enable the preparation of perfluoroalkylsubstituted benzimidazo [2,1-a] isoquinolin-6(5H)-ones have not been reported so far.

In most cases, an exogenous photocatalyst, e.g., transition-metal complex, or organic dye, is required to harvest visible-light energy.^[14] Alternatively, electrondonor-acceptor (EDA) complexes, composed of electron donors and acceptors through molecular noncovalent interactions, can be effectively excited by visible-light.^[15] As a result, in some visible-light promoted reactions external photocatalysts, especially those expensive and toxic metal complexes, could be avoided through the formation of EDA complexes.^[16] With our ongoing interests on green chemistry and radical reactions,^[17] we herein disclosed a metal-free procedure to synthesize perfluoroalkyl-substituted benzimidazo[2,1-a] isoquinolin-6(5H)-ones and indolo[2,1alisoquinolin-6(5H)-ones via visible-light-induced radical cyclization reaction under mild conditions (Scheme 1c-d). To the best of our knowledge, this is the first example for the preparation of perfluoroalkyl-substituted benzimidazo[2,1-a]isoquinolin-6(5H)-ones and indolo[2,1-a] isoquinolin-6(5H)-ones. All these synthesized products are new compounds.

Results and Discussion

We initiated our study by establishing optimal experimental conditions using the model reaction of *N*methacryloyl-2-phenylbenzoimidazole (**1a**) with C_4F_9I (**2b**) under visible-light irradiation. After extensive experimentation, the optimal reaction conditions were thus established as follows: **1a** (0.2 mmol), **2b** (0.4 mmol), tetramethylethylenediamine (TMEDA, 2 equiv.) in the CH₃CN (2 mL) under irradiation of 25 W blue LEDs under N₂ atmosphere at 35 °C for 12 h (see the Supporting Information for more details).

With the optimized conditions in hand, the substrate scope for this visible-light promoted reaction to access perfluoroalkyl-substituted benzimidazo[2,1-a]isoquinolin-6(5H)-ones was investigated, as illustrated in Table 1. Initially, the suitability of various perfluoroalkyl was studied under the standard conditions. It was pleased to see that seven perfluoroalkyl iodides $(IC_nF_{2n+1}, n=3-8, 10)$ react smoothly with **1 a** to give the corresponding products 3a-g in moderate to good yields (52-80%). Inspired by these exciting results, other fluoroalkyl iodides, including ICF2COOEt and ICF₂CF₂Cl were examined. To our delight, the desired products **3 h**-i were obtained in 45% and 65% yields, respectively. Afterward, the reactivity of functionalized 2-arylbenzoimidazoles was evaluated under the optimized conditions. Notably, substrates 1 bearing electron-donating groups (-Me, -Et, -Pr, -OMe) and electron-withdrawing groups (-F, -Cl, -Br, -CF₃, -CN) were all compatible with the optimal conditions, delivering the corresponding products 3 j-u in moderate to good yields (42-72%). Generally, substrates 1 bearing electron-donating groups showed better reactivity than those attached with electron-donating

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Table 1. Substrate scope for the synthesis of perfluoroalkyl-substituted benzimidazo[2,1-a]isoquinolin-6(5H)-ones.^[a]

^[a] Reaction conditions: **1** (0.2 mmol), **2** (0.4 mmol) and TMEDA (2 equiv.) in CH₃CN (2 mL) with the irradiation of 25 W blue LEDs under N_2 atmosphere at 35 °C for 12 h. Isolated yields were given.

groups. Additionally, a scale-up reaction for the synthesis of **3b** was performed *via* our designed flow reactor.^[18] Delightfully, a good yield (56%) of the desired product **3b** was obtained, demonstrating this metal-free visible-light promoted cyclization reaction is a robust protocol with great potential in practical applications (details see the Supporting Information). Notably, some entries of Table 1 gave very low yields (**3h**, **3s**, **3u**). In such cases, the decomposition of the starting material **1** into the corresponding precursors 2phenylbenzimidazoles was observed. Among all the newly synthetic products, the structure of **3b** was further confirmed by X-ray crystallography (hydrogen atoms were not shown for clarity).^[19]

Indolo[2,1-*a*]isoquinolin-6(5*H*)-ones moiety is a core structure of nature products with various biological activities.^[20] In the following research, we also made an attempt to extend this visible-light promoted cyclization reaction for the construction of biologically

Table 2. Substrate scope for perfluoroalkyl-substituted indolo [2,1-a]isoquinolin-6(5*H*)-ones.^[a]



^[a] Reaction conditions: **4** (0.2 mmol), **2** (0.8 mmol) and TMEDA (4 equiv.) in CH₃CN (3 mL) with the irradiation of 25 W blue LEDs under N₂ atmosphere at 35 °C for 12 h. Isolated yields were given.

valuable perfluoroalkyl-substituted indolo[2,1-*a*]isoquinolin-6(5*H*)-ones from the functionalized 2-arylindoles and perfluoroalkyl iodides. As it can be seen in Table 2, *N*-methacryloyl-2,3-diphenyl indole (**4a**) reacted smoothly with C_4F_9I (**2b**) to yield the product **5a** in moderate yield (65%). Encouraged by this result, other perfluoroalkyl iodides and ICF₂COOEt were employed to react with **4a**. It was observed that the desired products **5b**–**e** were generated in moderate to excellent yields (60–71%). Moreover, the reactivity of various substrates **4** with ICF₂COOEt were examined. The results showed that the optimal conditions were applicable to those substrates to access the products **5f**–**j** in moderate to excellent yields (55–80%).

Several control experiments were conducted to gain more insights into the reaction mechanism of this visible-light promoted cyclization reaction. When the model reaction was treated with radical scavengers, i.e., 2,2,6,6-tetramethylpiperidine 1-oxyl (TEMPO) or 2,6-di-*tert*-butyl-4-methylphenol (BHT), the reactions were totally suppressed, indicating that a radical pathway might be involved (Scheme 2).

Additionally, the electron paramagnetic resonance (EPR) experiment was carried out. When the radical spin trapping reagent *tert*-butyl- α -phenylnitrone (PBN) was added to the mixture of TMEDA and *n*-perfluor-obutyl iodide in the presence of blue light irradiation, a clear signal was observed. According to the previous report,^[21] the signal should be attributed to the

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Scheme 2. Control experiments.



Figure 2. EPR spectra in the presence of PBN.



Scheme 3. Plausible mechanism.

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corresponding adduct PBN–C₄F₉, suggesting perfluoroalkyl radical (•R_f) was generated under the reaction conditions. Moreover, no signal was observed without TMEDA under the identical conditions, suggesting the importance of TMEDA (Figure 2). Furthermore, the formation of EDA complex from C₄F₉I and TMEDA was confirmed by using ¹⁹F NMR titration experiment, and the Job's plot analysis revealed that donor/acceptor molar ratio for the formation of EDA complex is 1:1, with a binding constant of 0.908 M⁻¹ in CDCl₃ (see the Supporting Information for details).^[22]

Based on experimental results and previous reports, a plausible mechanism was proposed for this visiblelight promoted cyclization reaction (Scheme 3). Initially, EDA complex was formed from TMEDA and perfluoroalkyl iodide. Then perfluoroalkyl radical (${}^{\circ}R_{f}$) and radical cation TMEDA^{+•} (7) were generated under the irradiation of visible-light. Then, the addition of ${}^{\circ}R_{f}$ to the C=C bonds of **1a** leaded to the formation of radical intermediate **8**, which subsequently underwent an intramolecular cyclization to afford the radical intermediate **9**. Following that, **9** was further oxidized by radical cation 7 *via* an intermolecular single electron transfer (SET) process to form carbocation 10, which was finally converted to the final product 3 by deprotonation.

Conclusion

In conclusion, we have developed an efficient and convenient visible-light-induced radical cyclization reaction for the synthesis of perfluoroalkyl-substituted benzimidazo[2,1-a]isoquinolin-6(5H)-ones and perfluoroalkyl-substituted indolo[2,1-a]isoquinolin-6(5H)ones from perfluoroalkyl iodides and easily available starting materials. To the best of our knowledge, this is the first example for the preparation of perfluoroalkylsubstituted benzimidazo [2,1-a] isoquinolin-6(5H)-ones and indolo[2,1-a]isoquinolin-6(5H)-ones. Various fluoroalkyl iodides were successfully employed as the radical precursors to initiate this radical addition/ cyclization reaction. The remarkable merits of this photochemical strategy include one-pot procedure, no external added photocatalysts, wide reaction substrates, mild reaction conditions, easy scaleup as well as experimental simplicity. Additional biological studies and the extension of this visible-light promoted radical cyclization reaction is currently underway in our laboratory.

Experimental Section

General Procedure for the Synthesis of 3 and 5: In a 25 mL Schlenk tube, 1 or 4 (0.2 mmol) was dissolved in MeCN (2 mL, 0.1 M), then 2 (2.0 equiv. or 4.0 equiv.) and TMEDA (2.0 equiv. or 4.0 equiv.) were added. The mixture was allowed to stir with irradiation of 25 W blue LEDs under N_2 atmosphere at 35 °C for 12 h. The reaction was monitored by TLC. After substrate 1 (or 4) was completely consumed, the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel to afford the desired product 3 (or 5).

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FULL PAPER

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