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Light-Mediated Cascade Transformation of Activated Alkenes: BiOBr Nanosheets as Efficient Photocatalysts for the Synthesis of α -aryl- β -trifluoromethyl Amides

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A facile light-induced, BiOBr nanosheets promoted one-pot tandem transformation of activated alkenes is presented. A wide variety of acyclic α -aryl- β -trifluoromethyl amides are synthesized via the consecutive trifluoromethylation/aryl migration/desulfonylation and N–H bond formation process.

Tandem or cascade transformation has been proved to be an efficient and atom economic strategy in organic synthesis as it enables a rapid increase in molecular complexity from readily available starting materials. Additionally, the advantage of such transformation is the simultaneous formation of two or more bonds in a single manipulation process. Alkenes are privileged motifs for tandem reactions and have been intensively studied to date. However, transformations involving construction of C-CF₃ bonds limited to halotrifluoromethylation.² hydrotrifluoromethylation,³ aminotrifluoromethylation,⁴ oxytrifluoromethylation.⁵ Recently, aryltrifluoromethylation of alkenes has spurred intense interest from synthetic chemists.⁶ Following the pioneering work of Liu and co-workers, alternative methods to enable the introduction of aryl and trifluoromethyl groups across the double bond of alkenes by transition-metalmediated or metal-free protocols have been independently developed. Although these strategies are effective, in most cases, these transformations proceed in an intramolecular fashion and some β -trifluoromethylated oxindoles derivatives with various biologically activity are synthesized from N-aryl acrylamide substrates (Scheme 1a). In sharp contrast, the reports on the formation of the products in an acyclic manner through the cascade aryltrifluoromethylation of alkenes are relatively rare. Very recently, the Nevado group described respectively the copper- and tetrabutylammonium iodide-catalyzed aryltrifluoromethylation of conjugated tosyl amides via a one-pot trifluoromethylation/aryl migration/desulfonylation sequence by employing Togni's reagent

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as the CF_3 source, and a series of linear α -aryl- β -trifluoromethyl amides bearing a quaternary stereocenter were well established (Scheme 1b). Despite effectiveness of Nevado's methods, increasing the diversity of available methodologies that realize the successful formation of acyclic α -aryl- β -trifluoromethyl amides from activated alkenes is still of considerable interest.

Light-driven chemical transformations including photoredox catalysis are becoming one of the efficient and sustainable tools in synthetic chemistry. Undoubtedly, searching for particularly useful catalysts that can promote the organic transformations effectively is a key in this field. Over the past decade various photocatalysts or sensitizers have been synthesized. In general, they can be mainly classified into three types: organic dyes, ruthenium(II) or iridium(III) or copper(I) metal complexes, and inorganic semiconductors. Among them, inorganic semiconductors have been recognized as the highly promising type due to their unique characteristics, such as easy-preparation, cheap, nontoxic, efficient, recyclable and so on. Usually, TiO2, ZnO, ZnS and CdS are typical semiconductors for organic chemical transformations because of their unique wide band gaps. 10 In recent years, inorganic bismuth-containing nanomaterials have been found to be the potential photocatalysts, which show strong absorption in UV or visible-light region. 11 Apart from their photocatalytic applications for the degradation of organic dyes and splitting of water into hydrogen and oxygen, the use of them for photocatalytic synthesis of organic molecules has also been reported. For example, Pericas and König groups reported a light-driven asymmetric α-alkylation of aldehydes by combining Bi₂O₃ or PbBiO₂Br as the low-band-gap photocatalyst with the MacMillan imidazolidinone as the chiral catalyst. 12 Later, the Pericas group demonstrated that Bi₂O₃ could be used as efficient photocatalysts for the atom transfer radical addition (ATRA) reaction of organobromides to diversely functionalized terminal olefins. 13 The Fu et al. made an advance on finding a surfacechlorinated BiOBr/TiO₂ hybrid composites to realize the selective C_{sp3}-H bonds functionalization of alkanes. ¹⁴ The asymmetric reactions, difunctionalization of alkenes, and C-H bonds functionalizations are the current hot research topics in chemistry and materials science, revealing that the inorganic bismuth semiconductors are a kind of potential and alternative

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photocatalysts for chemical transformations. Therefore, the development of bismuth-based photocatalysts for new organic reactions, particularly the tandem transformations, is highly desirable.

Herein, we present a light-mediated, BiOBr nanosheets promoted one-pot cascade trifluoromethylation/1,4-aryl migration/desulfonylation and N–H bond formation reaction of conjugated tosyl amides, and a series of α -aryl- β -trifluoromethyl amides with diverse functional groups are successfully prepared (Scheme 1c). These trifluoromethyl functionalized organic compounds maybe find potential applications in pharmaceuticals, agrochemicals, and functional materials. 15

Scheme 1 Cascade aryltrifluoromethylation of alkenes.

The as-prepared BiOBr is a layer-structured semiconductor. 16 Inorganic nanosheets are considered as one kind of efficient photocatalyts due to the dramatically improving intrinsic catalytic properties of nanosheets over other correspond inorganic couterparts.¹⁷ The reductive potential of electrons in the conduction band of BiOBr semiconductor is -0.27 V (vs. SCE), 11a which is higher than CF₃SO₂Cl (-0.18 V vs. SCE). 18 It means that the photogenerated electron of BiOBr can effectively reduce CF₃SO₂Cl and thus produce the CF3 radical, which be used for subsequent transformation of alkenes. Our group has recently reported the trifluoromethylation/arylation of N-aryl acrylamide using BiOBr nanosheets as photocatalysts. 15 We speculate that this photocatalytic method may also facilitate the trifluoromethylation/aryl migration/desulfonylation conjugated tosyl amide substrates. To validate the hypothesis, Nphenyl-N-tosylmethacrylamide 1a and CF₃SO₂Cl 2 are selected as model substrates in conjunction with BiOBr nanosheets, K2HPO4 and HSiEt₃ in DMF under light excitation (280~780 nm) for 6 h. It is found that the one-pot sequential transformation of 1a definitely occurs, and 74% yield of α -aryl- β -trifluoromethyl amide is obtained (Table S1, entry 1). When DMF is replaced with DMAC, the yield of the product is slightly increased (Table S1, entry 2). However, when the reaction is carried out in the absence of HSiEt₃, a comparable yield of 3a is obtained (Table S1, entry 3). Although the reaction efficiency is a little improved due to the addition of HSiEt₃, in order to reduce the complexity of the operation, we determine that the HSiEt₃ is not a necessary additive. Encouraged by this result, othe solvent are screened next. We observe that only DMAC gives the best result and the others are inferior or even noneffective. These results suggest a significant solvent dependence for this photocatalytic reaction (Table S1, entries 4-10). Notably, no product ${\bf 3a}$ is detected either in the dark or in the absence of BiOBr nanosheets, strongly illustrating that the light and the photoredox catalysts are important to induce this reaction (Table S1, entries 11, 12). We have attempted to use CdS nanosheets and Ru(bpy)₃Cl₂.6H₂O as alternative photocatalysts, which are demonstrated exceptional photocatalytic properties for organic transformations, however, dissatisfactory results are obtained (Table S1, entries 13-14).

Table 1 Substrate scope of the photocatalytic cascade reaction of alkenes^a

 a Reaction conditions: ${\bf 1a}$ (0.2 mmol), ${\bf 2}$ CF $_3$ SO $_2$ CI (1.0 mmol), K $_2$ HPO $_4$ (0.6 mmol), BiOBr nanosheets (20 mg), DMAC (1.0 mL), 300 W Xe arclamp, RT.

Under the optimized reaction conditions, the scope of this transformation is explored and displayed in Table 1. We firstly examine the substitution pattern on the aromatic ring directly bound to the N atom. When the substituents at the *ortho* position, these one-pot sequential reactions can produce the corresponding aryltrifluoromethylated amides in moderate to good yields (Table 1, **3b-e**). The 1,4-dimethyl substituted tosyl amide does not hinder the reaction, and the product **3g** can be isolated in 52% yield. Substrate **1h** with a methyl on the *meta* position of phenyl ring is also amenable to the standard conditions, and 37% yield of the product

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is obtained. The introduction of either electron-donating or electron-withdrawing groups at the *para* position is well tolerated, and 57% to 65% yields of the β -trifluoromethylated amides are constructed (Table 1, **3i-k**, **3m-o**). In contrast, the presence of idoine seems to decrease the reaction efficiency no matter where the iodine atom lies on the aryl ring (Table 1, **3f** and **3l**). Next, the influence of the substituents on the aromatic ring of the sulfonamide group is also investigated. Replacement of the methyl group by either a hydrogen, methoxyl, or chlorine giving rise to the corresponding products in slightly lower yields (Table 1, **3p-r**). All the obtained results indicate that our photocatalytic methodology is particularly universal, and may provide a new facile access to the synthesis of CF₃-containing pharmaceutical blocks.

In order to verify whether these reactions proceed through a radical pathway, the control experiments are performed (Scheme 2). Treatment of **1c** with **2** under standard conditions with the addition of radical trapping agent 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) delivers no desirable product, further supports that a radical process is involved in our photocatalytic arylation/trifluoromethylation of alkenes.

Based on the above results and related photocatalytic literatures, 8,10b a plausible reaction mechanism is proposed in Scheme 3. Light absorption of BiOBr nanosheets promotes the electron to transfer from the valence band (VB) to the conduction band (CB), which is a key photochemical step. CF_3SO_2Cl is then reduced by the photogenerated electron to generate the corresponding free radical anion of triflyl chloride, which experiences fast collapses to form the relative stable CF_3 radical with sulfur dioxide and chloride

anion. ¹⁸ Subsequently, the CF₃ radical interacts with alkene **1a** affording the activated radical intermediate **IN1**. A dearomatization/5-ipso cyclization then takes place on the aromatic ring generating aryl radical **IN2**. Rearomatization of **IN2** with concomitant desulfonylation leads to amidyl radical **IN3**. The photogenerated hole obtains an electron from the solvent DMAC via a single-electron transfer process to close the catalytic cycle and meanwhile give the radical cation **IN4**. The amide functions as an effficient electron doner could be found in many reactions. ¹⁹ Finally, the formed intermediate **IN3** abstracts the hydrogen radical released from **IN4** to deliver the desired product **3a**. The preliminary mechanism may need further theoretical understanding.

Conclusions

In conclusion, we have developed a facile light-induced, BiOBr nanosheets promoted one-pot tandem trifluoromethylation/1,4-aryl migration/desulfonylation and N–H bond formation reaction of conjugated N-tosyl amides. This strategy enables a practical access to a series of α -aryl- β -trifluoromethyl amides bearing a quaternary stereocenter in moderate to good yields. In this heterogeneous reaction media, electron-exchange occurs between the organic intermediates in solution and the semiconductor surface. Control experiments illustrate that the synergistic effect of photogenerated electrons and holes plays a key role for the reaction efficiency. This low-cost, green and efficient photocatalytic strategy can be considered as a viable alternative to the previously mentioned methods. Further explorations on light-mediated cascade reactions and mechanistic understanding are currently being investigating in our laboratory.

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Graphic Abstract

Light-Mediated Cascade Transformation of Activated Alkenes: BiOBr Nanosheets as Efficient Photocatalysts for the Synthesis of α-aryl-β-trifluoromethyl Amides

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A facile light induced, BiOBr nanosheets promoted one-pot consecutive trifluoromethylation/aryl migration/desulfonylation and N–H bond formation of activated alkenes is proposed.

$$R^{1}$$
 + $CF_{3}SO_{2}CI$ R^{2} + $CF_{3}SO_{2}CI$ R^{2} + $CF_{3}SO_{2}CI$ R^{2} + R^{2} R