Organic Letters Scite This: Org. Lett. XXXX, XXX, XXX–XXX

Letter pubs.acs.org/OrgLett

Light-Induced Intramolecular Iodine-Atom Transfer Radical Addition of Alkyne: An Approach from Aryl lodide to Alkenyl lodide

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Supporting Information

ABSTRACT: A light-induced intramolecular iodine-atom transfer radical addition was reported toward iodinesubstituted fluorene derivatives. A thioxanthone derivative was employed as a visible light sensitizer for this 5-exo-dig exclusive radical addition, and the newly formed vinyl iodide compounds were further proved to be effective partners for several cross-coupling reactions.



 I bonds are commonly considered as the most reactive C-halogen bond in organic transformations, including nucleophilic substitutions, cross-coupling reactions, and transformations that involve radicals.¹ However, the reactive nature of the C-I bond makes the synthesis of the C-I bond very challenging because it requires accurate control to avoid further cleavage of the newly generated C-I bond.

Vinyl iodide is one of the most useful scaffolds for direct alkene functionalization. The vinyl C-I bond could easily undergo oxidative addition to form a carbon-metal intermediate for varies transformations.² Thus, the construction of vinyl iodide has attracted a growing amount of attention in the past few decades. One of the typical methods is to use a substituted alkene with leaving groups [Br,³ B(OH)₂,⁴ TMS,⁵ $COOH_{0}^{\circ}$ and H^{\prime} to react with the corresponding iodine source, such as I⁻, I₂, or NIS (Scheme 1A). Although many studies were carried out for this type of transformation, the substrate scope is greatly limited due to the leaving groups and the atom economy of the reaction is low. Another way to synthesize vinyl iodide is difunctionalization of alkynes.⁸ Normally, an "initiating reagent (R³ source)" is employed and an iodinating reagent serves as a quenching reagent for the formation of vinyl iodide (Scheme 1B).

Iodine-atom transfer radical addition (I-ATRA) shows a high atom economy in C-I bond formation. Pioneering research on I-ATRA reactions by Curran,⁹ Taguchi,¹⁰ and others¹¹ provided useful organic synthesis methods. However, the classic I-ATRA reaction requires toxic and hazardous initiators, such as AIBN, organotin, Et₃B, or peroxides. Transition metal catalysts could also be used for I-ATRA reactions.¹² Methods involving other intermediates, such as anion,¹³ cation,¹⁴ benzyne formation,¹⁵ and ligand-controlled reductive elimination,¹⁶ have been reported but are relatively rare. It has been reported that a radical intermediate could be formed with the assistance of visible light photoredox catalysis (VLPC).¹⁷ Utilizing the photogenerated radical intermediate, several groups developed intermolecular halogen ATRA via Mn-based,¹⁸ Cu-based,¹⁹ Ru-based,²⁰ or Ir-based²¹ VLPC (Scheme 1C). These reactions used a wide range of halogen sources and alkenes and showed great potential in applications. Consistent with the electron transfer process (VLPC), another aspect of photochemistry is the energy transfer process.²² The excited photocatalyst donates the excitation energy directly to the substrate to excite the reactant. With the direct energy transfer process, the Melchiorre group realized photo-organocatalyzed halogen ATRA to alkene employing 4-methoxybenzaldehyde as the photosensitizer via a direct energy transfer process.²³

Nevertheless, previous works in photochemical activation mainly focused on C(sp³)-I bond cleavage-based I-ATRA; photoinduced C(sp²)-I bond cleavage was reported only recently.²⁴ The C(sp³)–I bond has a bond energy (\sim 55 kcal/ mol) that is lower than that of the $C(sp^2)$ -I bond (~65 kcal/ mol).²⁵ Because of the high frequency of the $C(sp^2)$ -I bond in organic compounds, methods aiming at $C(sp^2)$ -I bond cleavage coupled with ATRA are highly desirable. A few issues should be addressed with respect to this transformation. First, the high bond energy of the $C(sp^2)$ -I bond implies that a higher excited energy is required, but a higher energy may result in the unexpected decomposition of the substrate. Second, after the I-ATRA process, the newly formed C-I bond in the product remains reactive for further cleavage. Thus, accurate control of C-I bond cleavage is necessary for effective I-ATRA reaction. With these considerations in mind, we envisioned that intramolecular I-ATRA from aryl iodide to alkenyl iodide is reasonable to achieve with a proper photosensitizer, because the Calkenvl-I bond is relatively more stable than the C_{aryl}-I bond according to bond energy,²⁵ so that the product will be stable under the reaction conditions (Scheme 1D). To overcome the problems mentioned above, a suitable photosensitizer must be carefully designed, which is active enough to excite the substrate without unpredictable decomposition but not too active to react with the newly

Received: October 4, 2019

Scheme 1. Synthesis of Vinyl Iodide



generated product. Herein, we report our recent results on intramolecular I-ATRA of alkyne under visible light irradiation.

Initially, the visible light-induced I-ATRA reaction was explored using 2-iodo-2'-[(4-methoxyphenyl)ethynyl]-1,1'-biphenyl (1a) as a model substrate with a variety of visible light photosensitizers. The key photophysical characteristics of the selected visible light photosensitizers, including absorption peaks (λ_{max}), triplet energies (E_T), and triplet lifetimes (τ_T), are listed in Table 1.²⁶ Commercially available organic dye



^{*a*}All reactions were carried out using **1a** (0.2 mmol) and a photosensitizer (5 mol %) in anhydrous CH₂Cl₂ (10 mL) irradiated by a 23 W household lamp under an argon atmosphere at rt for 48 h. Abbreviations: lamp, 23 W household lamp; λ_{max} , maximum absorption wavelength; $E_{\rm T}$, triplet energy; $\tau_{\rm T}$, triplet lifetime ^{*b*}See ref 26. ^cYield determined by ¹H NMR analysis of the crude reaction mixture using CH₂Br₂ (0.2 mmol) as the internal standard. ^{*d*}Recovered yield of **1a**.

methylene blue was first tried in this reaction. Unfortunately, no reaction took place (Table 1, entry 1). Other photosensitizers with either a higher triplet energy or a longer triplet lifetime were also tested, but no desired results were obtained (Table 1, entries 2-4). When thioxanthone was employed as a photosensitizer, the desired I-ATRA product 2a was formed, although the reaction rate was very low (Table 1, entry 5). Considering that thioxanthone has the highest triplet energy among these tested photosensitizers to enable the I-ATRA reaction and the absorbance maxima of thioxanthone lies in the visible region, thioxanthone was chosen as the photosensitizer for the following optimizations.

With the original screening of the proper photosensitizer, we continued to optimize the reaction conditions (Table 2). A purple light-emitting diode (LED) was used instead of a 23 W household lamp with consideration of the absorption spectra of

Table 2. Optimization of the Reaction Conditions^a



entry	photosensitizer	light	concn (mM)	time (h)	yield (%) ^b
1	I	lamp	20	48	5 (93) ^c
2	Ι	PLED	20	48	36 (61) ^c
3	Ι	PLED	10	48	59 (40) ^c
4	Ι	PLED	2	48	93 (7) ^c
5	Ι	PLED	2	24	90 (10) ^c
6	II	PLED	2	24	38 (61) ^c
7	III	PLED	2	24	54 (44) ^c
8	IV	PLED	2	24	94 (90) ^d
9	V	PLED	2	24	92 (8) ^c
10	VI	PLED	2	24	19 (77) [°]
11	_	PLED	2	24	NR
12	IV	dark	2	24	NR

^{*a*}All reactions were carried out using **1a** (0.1 mmol for 2 mM reactions, 0.2 mmol for 10 and 20 mM reactions) and a thioxanthone derivative (5 mol %) in anhydrous CH_2Cl_2 irradiated by the corresponding light source under an argon atmosphere at rt. Abbreviations: lamp, 23 W household lamp; PLED, purple LED (for the detailed setup and emission spectra, see the Supporting Information). ^{*b*}Yield determined by ¹H NMR analysis of the crude reaction mixture using CH_2Br_2 as the internal standard. ^{*c*}Recovered yield of **1a**. ^{*d*}Isolated yield.

thioxanthone (for the emission spectra of a purple LED, see Figure S1). A dramatically increased yield was observed (Table 2, entry 2). Concentration is also an important parameter in photochemistry, which can greatly influence the efficiency of absorbing light according to Lambert-Beer's law. Thus, a reaction with a 10-fold lower concentration was carried out, affording 93% 2a with complete conversion (Table 2, entry 4). Then, photosensitizers of different substituted thioxanthone derivatives were tested, among which 4-methoxy thioxanthone (IV) showed the best yield (Table 2, entry 8). Finally, control experiments without a catalyst (Table 2, entry 11) or light (Table 2, entry 12) were carried out. No reaction occurred under either condition. These results indicated that both a catalyst and light were necessary in this reaction. Thus, condition A [1, IV (5 mol %), CH₂Cl₂, purple LED, and rt] was applied as the optimized condition for further studies.

With the optimized conditions in hand, the substrate scope of this visible light-induced I-ATRA reaction was explored. As

shown in Scheme 2, substrates with a strong electron-donating group (1a), a weak electron-donating group (1b), or no



^{*a*}All reactions were carried out using 1 (0.1 mmol) and IV (5 mol %) in anhydrous CH_2Cl_2 (50 mL) irradiated by a purple LED under an argon atmosphere at rt for 24 h. Isolated yield reported. ^{*b*}Gram-scale reaction. ^{*c*}Reaction carried out at 60 °C.

substitution (1c) in Ar³ showed decent reactivity. Halogen atoms, like Br (1d), Cl (1e), and F (1f), were introduced onto Ar³, affording similar excellent yields of the desired products. Notably, the C-Br bond normally shows a reactivity similar to that of the C-I bond in the aspect of C-X bond cleavage of both organometallic catalysis or photoinduced cleavage, but in this case, the C-Br bond was tolerated while the C-I bond proceeded the I-ATRA reaction (2d). This observation indicated a selectivity between the C-Br bond and the C-I bond in this reaction. A methoxycarbonyl group (1g), as a strong electron-withdrawing group, was introduced onto Ar³ and gave a decreased yield. A cyano group (1h) dramatically inhibited this reaction. A higher temperature was required for this transformation. Next, some trisubstituted substrates were applied under the optimized condition. With an electrondonating group in Ar¹, Ar², and Ar³ (1i and 1j), the I-ATRA

reaction gave the desired product in good to excellent yield. A slightly decreased yield was obtained for 1k-n. Similarly, no other C-X bond cleavage occurred during the I-ATRA process (1l-n). For the substrate bearing one strong electron-withdrawing group and two strong electron-donating groups (1o), the I-ATRA reaction yielded the corresponding product with a good yield. The substrate with only Ar^2 and $Ar^3 (1p-r)$ did not show reactivity under optimized conditions (for a detailed discussion, see Scheme S1). A gram-scale reaction using 1a was carried out and gave a good yield.

With those readily available compounds in hand, we also tested some coupling reactions with **2a**. As shown in Scheme 3,





using **2a** as a coupling partner, Suzuki coupling was achieved in 95% yield and the product was **3a**. Sonogashira coupling of **2a** with TMSA also gave **3b** in an excellent yield, and Kumada coupling was also feasible for **2a**. Thus, the formed vinyl iodide products are viable substrates for the coupling reactions, indicating the potential application of the I-ATRA product.

To gain insight of the mechanism, several control experiments were designed (Table 3). First, to define the exact role

Table 3. Mechanistic Studies^a



^{*a*}All reactions were carried out using **1a** (0.1 mmol) and **IV** (5 mol %) in anhydrous CH_2Cl_2 (50 mL) irradiated by a purple LED under an argon atmosphere at rt for 24 h unless otherwise noted. Yield determined by ¹H NMR analysis of the crude reaction mixture using CH_2Br_2 as the internal standard. ^{*b*}Recovered yield of **1a**.

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of catalyst in this I-ATRA reaction, three triplet quenchers that are especially sensitive to thioxanthone were added to the reaction system.²⁶ Oxygen could quench the reaction effectively. On the other hand, 75% of 1a decomposed into some undefined compounds under an oxygen atmosphere (Table 3, entry 1). Oxygen might be the triplet quencher but could also inhibit this reaction by oxidative decomposition of 1a due to its oxidative property. When styrene was applied, 49% of 2a was formed and 41% of 1a was recovered (Table 3, entry 2). The reaction in the presence of another triplet quencher, ethyl 4-(dimethylamino)benzoate, showed a similar result (Table 3, entry 3). These results demonstrated that the addition of triplet quenchers for thioxanthone restrained this I-ATRA reaction effectively, which strongly indicated that the triplet excited state of IV was likely to be involved in this transformation.

After obtaining a clear understanding of the role of the catalyst, we focused on how this reaction proceeded. Upon excitation of 1a, the C-I bond was likely to undergo homolysis, giving the radical intermediates. Thus, 5 equiv of TEMPO was added under condition A; 17% of 2a was formed, and 30% of 1a was recovered (Table 3, entry 4). Although the reaction was strongly inhibited, it was still confusing that even 5 equiv of TEMPO could not completely inhibit the reaction, so we considered that the radical might be the predominant but not the only intermediate. Heterolysis might also occur during the initial transformation.^{24g,h} Hence, a competitive reaction between the bromo anion and iodine anion was designed, using 5 equiv of tetrabutylammonium bromide (TBAB) as the bromo anion source. Bromo-substituted product 4a was formed, and the 2a:4a ratio was 52:40 (Table 3, entry 5).^{24h} This result proved that there was indeed heterolysis that involved a cation during the reaction.^{24g,h} Upon careful comparison of the ratio of the "radical intermediate" and "cation intermediate", they seemed not to be consistent in the TEMPO experiment and TBAB experiment (Table 3, entries 4 and 5, respectively). These two control experiments seemed to have a contrast between the ratio of the radical intermediate and cation intermediate. An equilibrium between homolysis and heterolysis could possibly explain this observation. The equilibrium would tune to heterolysis to form the ion intermediate and gave more 4a with 5 equiv of TBAB. Thus, a mechanism including both homolysis and heterolysis was reasonable for this I-ATRA reaction.

With the evidence presented above and literature precedent, a complete mechanism was proposed as shown in Scheme 4. Upon visible light irradiation, IV underwent an $n-\pi^*$ transition to its singlet excited state and relaxed to its first singlet excited state, $IV(S_1)$. Due to its high instability, $IV(S_1)$ proceeded via intersystem crossing (ISC) to its first triplet excited state, IV (T_1) . This is a relatively long lifetime intermediate, which was also the key excited state in this reaction.²⁶ Then, an energy transfer occurred between IV (T_1) and substrate 1 to form triplet 1^* (T₁), and IV returned to its ground state for another catalytic cycle. 1^* (T₁) could proceed via reversible homolysis to generate radical 5 and an iodine radical. Intramolecular radical addition of 5 produced cyclized alkenyl radical 6, which would couple with the iodine radical to furnish product 2. Meanwhile, a reversible heterolysis occurred, producing cation 7 and an iodine anion.^{24g,h} The intramolecular $C \equiv C$ bond might attack the cation to form cyclized alkenyl cation 8. The iodine anion could couple with 8

Scheme 4. Proposed Mechanism



to yield **2**. The bromo anion could also couple with **8** to generate the corresponding bromide **4**.

In conclusion, we developed a visible light-induced I-ATRA reaction of $C(sp^2)$ -I bonds using thioxanthone derivatives as the photosensitizer. This photochemical method enabled the cleavage of one $C(sp^2)$ -I bond and formation of another $C(sp^2)$ -I bond under visible light irradiation without any transition metal catalyst. Further study indicated that the catalyst played the role of a visible light triplet photosensitizer. Both homolysis and heterolysis of the $C(sp^2)$ -I bond were likely involved in this transformation. Further study of the applications of this I-ATRA reaction to organic synthesis is in progress in our laboratory.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.or-glett.9b03519.

Experimental details and characterization data for the products (PDF)

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Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

The authors greatly acknowledge the financial support from Shanghai Science and Technology Committee (18DZ1201607) and the International Science & Technology Cooperation Program of China (2014DFE40130).

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