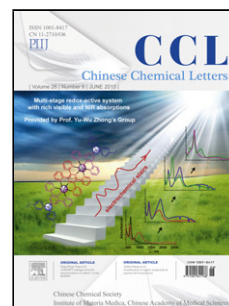


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Communication

A practical synthesis of α -bromo/iodo/chloroketones from olefins under visible-light irradiation conditions

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ABSTRACT

A practical synthesis of α -bromo/iodo/chloroketones from olefins under visible-light irradiation conditions has been developed. In the presence of $\text{PhI}(\text{OAc})_2$ as promoter and under ambient conditions, the reactions of styrenes and triiodomethane undergo the transformation smoothly to deliver the corresponding α -iodoketones without additional photocatalyst in good yields under sunlight irradiation. Meanwhile, the reactions of styrenes with tribromomethane and trichloromethane generate the desired α -bromoketones and α -chloroketones in high yields by using $\text{Ru}(\text{bpy})_3\text{Cl}_2$ as a photocatalyst under blue LED (450–455 nm) irradiation.

Keywords:

α -Bromoketones α -Iodoketones α -Chloroketones, Olefins

α -Haloketones are not only useful building blocks and important intermediates in organic synthesis but also in medicinal chemistry [1–3]. A range of pharmaceutically important heterocycles including thiazoles, imidazoles and indoles were prepared from α -haloketones as starting materials [2–3]. In addition, α -haloketones can be transformed into a number of useful organic compounds, such as aldols stereospecifically in the presence of $\text{Cr}(\text{II})$ -salt [4], α -allyl carbonyl compounds with allyl-Ga and allyl-In reagents [5], arylacetic acids by Ag-promoted rearrangement of α -bromo-alkylarylketones [6], and β -diketones through organic Zn-reagents [7]. In general, α -haloketones are prepared from the corresponding ketones. For the synthesis of α -bromoketones from ketones, a number of procedures have been recommended, including the reactions of ketones with Br_2/acid [8], silica/dioxane/ Br_2 [9], [hydroxy(tosyloxy)iodo]benzene/ MgBr_2 [10], *N*-methylpyrrolidin-2-one hydrotribromide [11], CuBr_2 [12], and NBS [13]. On the other hand, a variety of synthetic methods have been developed for the direct conversion of ketones to their corresponding α -iodoketones from reactions of ketones with I_2 [14a], I_2/CAN [14b], I_2/HgCl_2 [15], I_2/SeO_2 [16], $\text{I}_2/\text{H}_2\text{O}_2$ [17], I_2 -trimethyloxymethane [18], and NIS [19]. However, the direct synthesis of α -haloketones from olefins, which are less expensive than ketones is more preferable comparing their synthesis from ketones. For the preparation of α -bromoketones, the developed transformations are finished through the reactions of olefins with $\text{KBr}/\text{K}_2\text{S}_2\text{O}_8$ [20], IBX/TBAB [21], NBS/IBX [22], bromide/bromate [23], $\text{HBr}-\text{H}_2\text{O}_2$ - KBr/V [24], $\text{HBr}-\text{H}_2\text{O}/\text{oxidant}$ [25], NaBrO_2 [26] and TsNBr_2 [27]. Meanwhile, α -iodoketones are synthesized by the reactions of olefins with $\text{I}_2/\text{Ag}_2\text{CrO}_4$ [28], I_2/PDC [29], $\text{I}(\text{collidine})_2\text{BF}_4/\text{DMSO}$ [30], and $\text{I}_2/\text{O}_2/\text{UV}$ [31].

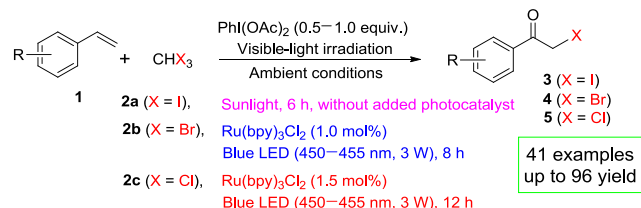
In general, the direct conversion of ketones into α -chloroketones can be accompanied by using chlorination agents including CuCl_2 [32], *p*-toluenesulfonylchloride [33], sulfurylchloride [34], tetraethylammoniumtrichloride [35], and NCS [36], as well as the combinations of $\text{TMSCl}/\text{KNO}_3$ [37], TMSCl/DMSO [38], $\text{TMSCl}/\text{SeO}_2$ [39], NCS/DMSO [40], NCS/Amberlyst-15 [41], NCS/Lewis acid [42], NCS/thiourea [43], $[\text{Hmim}][\text{NO}_3]/\text{HCl}$ [44], $\text{AlCl}_3/\text{urea}/\text{H}_2\text{O}_2$ [45], and $\text{NaClO}_2/\text{Mn}(\text{acac})_3/\text{Al}_2\text{O}_3$ [46]. However, most of these systems have drawbacks, such as long reaction time, use of extra oxidizing agent, tedious procedures, relatively harsh reaction conditions, and the consumption of large amounts of organometallic reagents. It should be noted that a general procedure for the preparation of α -bromoketones, α -iodoketones and α -chloroketones is still scarce. Therefore, the development of a facile synthesis of α -bromo/iodo/chloroketones from olefins under mild reaction conditions is highly desirable.

Herein, we wish to report a practical and efficient strategy toward synthesis of α -bromo/iodo/chloroketones from olefins under visible-light irradiation conditions. In the presence of $\text{PhI}(\text{OAc})_2$ as promoter and under ambient conditions, the reactions of styrenes and triiodomethane undergo smoothly to deliver the corresponding α -iodoketones without additional photocatalyst in good yields under sunlight irradiation. Meanwhile, the reactions of styrenes with tribromomethane and trichloromethane generate the desired α -bromoketones and α -chloroketones in high yields by using $\text{Ru}(\text{bpy})_3\text{Cl}_2$ as photocatalyst under blue LED irradiation (Scheme 1).

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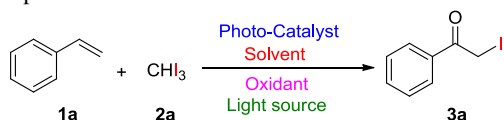
Scheme 1. Synthesis of α -bromo/iodo/chloroketones from olefins under visible-light irradiation conditions.

To optimize the reaction conditions for the preparation of α -iodoketones, styrene (**1a**) and triiodomethane (**2a**) were selected as the model substrates. When the model reaction was performed in the presence of $\text{Ru(bpy)}_3\text{Cl}_2$ (1.0 mol%) as a photocatalyst and PhI(OAc)_2 (50.0 mol%) as a promoter under ambient conditions in acetone with blue LED (450–455 nm) irradiation for 6 h, the desired product 2-iodo-1-phenylethanone (**3a**) was isolated in 57% yield (Table 1, entry 1). The solvent plays an important role in the reaction. After examination of the selected solvents, it was found that dioxane gave the highest reactivity (entry 2). Other solvents, such as DMC, CH_3CN , DME and PhMe exhibited less reactivity, generating the product **3a** in 55%–23% yields (entries 3–6). However, DCE, CH_3OH , DMF, DMSO and THF showed no reactivity (entries 7–11). To our delight, when the model reaction was carried out in the absence of additional photocatalyst under blue LED (410–415 nm) or sunlight irradiation, the excellent yields of product **3a** were obtained (entries 12 and 13). Addition of TBHP as promoter showed poor reactivity in the reaction (entry 14). As the model reaction was performed without light irradiation (in the darkness), no desired product **3a** was observed (entry 15). In the absence of PhI(OAc)_2 , the model reaction afforded 17% yield of **3a** (entry 16). The model reaction exhibited no reactivity under green LED (530–535 nm) irradiation (entry 17). Further investigation indicated that optimal amount of PhI(OAc)_2 is in the range of 50%–100 mol% (Table 1, entries 18–20).

Based on the optimized conditions in our hand, the generality of α -iodoketones preparation from olefins was investigated, as shown in Scheme 2. In general, a number of styrenes (**1**) bearing different substituents on the benzene rings underwent the reaction with triiodomethane smoothly to yield the corresponding products in good yields. It should be noted that styrenes with an electron-donating group (Me, *t*-Bu) at the *para*-position of the phenyl rings, are excellent substrates, which could react with **2a** to complete the reactions, affording the desired products (**3b–3c**) in 88%–89% yields. It was found that styrenes with an electron-withdrawing group (F, Cl, Br, CO_2Me , CF_3 , NO_2 or CN) at the *para*-position of the benzene rings, generating the corresponding products (**3d–3j**) in 87%–93% yields. When styrenes containing a Me, F or Br at the *ortho*-position of the aromatic rings were employed, the desired products (**3k–3m**) were achieved in 73%–83% yields, indicating steric effect. Meanwhile, styrenes with a F, Cl or Br at the *meta*-position of the phenyl rings reacted with triiodomethane to afford the products (**3n–3p**) in 85%–88% yields. The employment of 2-vinylnaphthalene and 2-vinylthiophene afforded the desired products (**3q** and **3r**) in 95% and 96% yield, respectively. When aliphatic alkenes, such as cyclohexene and 1-octene were used, no desired products (**3s** and **3t**) were obtained.

Table 1

Optimization of the reaction conditions.^a



Entry	Solvent	Promoter	Photocatalyst	Yield (%) ^b
1	Acetone	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	57
2	Dioxane	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	81
3	DMC	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	55
4	CH_3CN	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	53
5	DME	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	33
6	PhMe	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	23
7	DCE	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	N. R.
8	CH_3OH	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	N. R.
9	DMF	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	N. R.
10	DMSO	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	N. R.
11	THF	PhI(OAc)_2	$\text{Ru(bpy)}_3\text{Cl}_2$	N. R.
12	Dioxane	PhI(OAc)_2	–	90 ^c
13	Dioxane	PhI(OAc)_2	–	90 ^d
14	Dioxane	TBHP	–	25
15	Dioxane	PhI(OAc)_2	–	N. R. ^e
16	Dioxane	–	–	17
17	Dioxane	PhI(OAc)_2	–	N. R. ^f
18	Dioxane	PhI(OAc)_2	–	75 ^g
19	Dioxane	PhI(OAc)_2	–	90 ^h
20	Dioxane	PhI(OAc)_2	–	90 ⁱ

^a Reaction conditions: Styrene (**1a**, 0.20 mmol), triiodomethane (**2a**, 0.20 mmol), solvent (2.0 mL), promoter (50.0 mol%), $\text{Ru(bpy)}_3\text{Cl}_2$ (1.0 mol%) at room temperature under blue LED (450–455 nm, 1.5 W) irradiation in air for 6 h.

^b Isolated yield.

^c Blue LED (410–415 nm, 1.5 W) was used.

^d Sunlight was used.

^e In the darkness.

^f Green LED (530–535 nm, 1.5 W) was used.

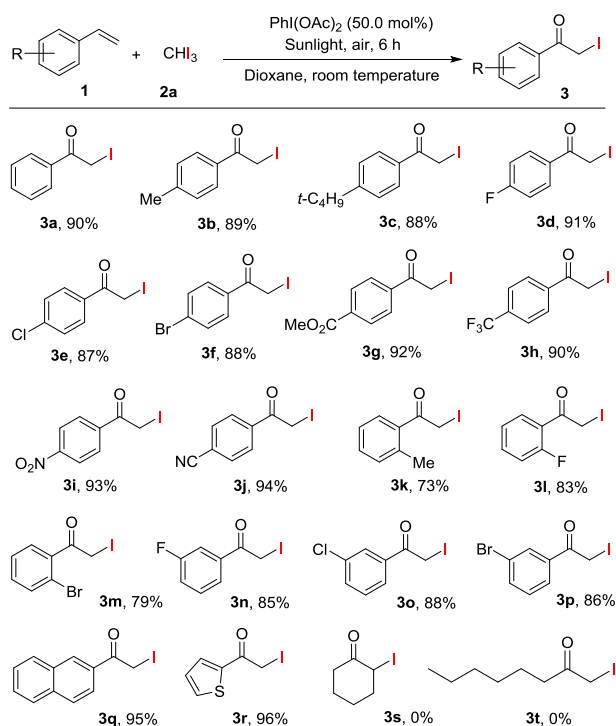
^g PhI(OAc)₂ (30.0 mol%) was added.

^h PhI(OAc)₂ (70.0 mol%) was added.

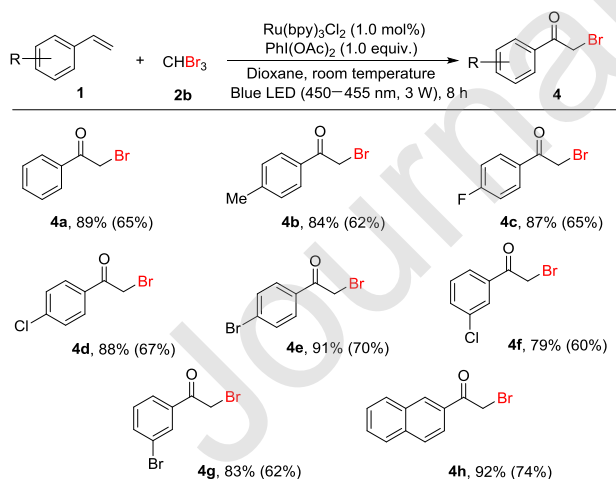
ⁱ PhI(OAc)₂ (100.0 mol%) was added.

N. R. = No Reaction.

Then, the scope of α -bromoketones synthesis from olefins was evaluated using this visible-light induced protocol. As can be seen from the Scheme 3, a variety of styrenes (**1**) including styrene, 4-methylstyrene, 4-fluorostyrene, 4-chlorostyrene, 4-bromostyrene, 3-chlorostyrene and 3-bromostyrene underwent the reaction with tribromomethane to generate the corresponding products (**4a–4h**) in good yields. It is important to note that the reactions performed in the presence of Ru(bpy)₃Cl₂ under blue LED (450–455 nm) irradiation are more efficient than that carried out in the absence of Ru(bpy)₃Cl₂ under sunlight irradiation.



Scheme 2. Preparation of α -iodoketones from olefins. Reaction conditions: styrene (**1**, 0.20 mmol), triiodomethane (**2a**, 0.20 mmol), dioxane (2.0 mL), PhI(OAc)₂ (50.0 mol%) at room temperature under sunlight irradiation in air for 6 h; isolated yields of the products.

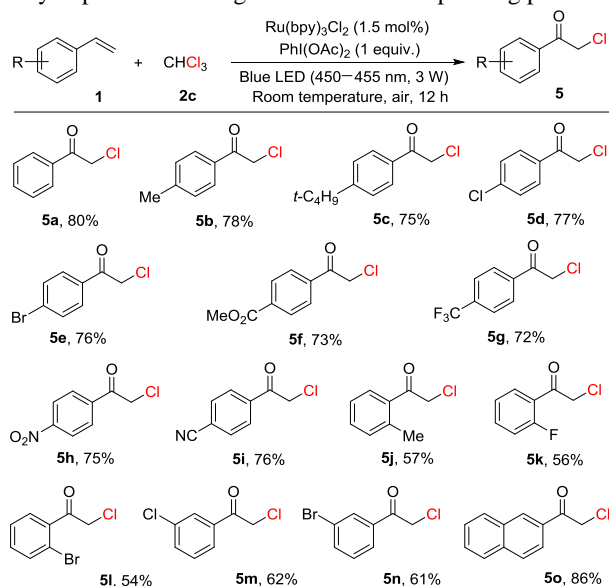


Scheme 3. Preparation of α -bromoketones from olefins. Reaction conditions: styrene (**1**, 0.20 mmol), tribromomethane (**2b**, 0.20 mmol), Ru(bpy)₃Cl₂ (1.0 mol%), PhI(OAc)₂ (1.0 equiv.), dioxane (2.0 mL) at room temperature under blue LED (450–455 nm, 1.5 W) irradiation in air for 8 h; isolated yields of the products. Yields in the brackets were from the reaction in the absence of Ru(bpy)₃Cl₂ under sunlight irradiation in air for 12 h.

In order to extend the scope of α -chloroketones preparation from olefins, the model reaction of styrene (**1a**) with trichloromethane (**2c**) under the reaction conditions in the absence of any additional photocatalyst, no desired product was detected. Interestingly, when Ru(bpy)₃Cl₂ (1.0 mol%) as a photocatalyst was added to the reaction, and the desired product (**5a**) was isolated in 80% yield, as shown in Scheme 4. When the synthesis of α -chloroketones was performed in the presence of Ru(bpy)₃Cl₂ (1.5 mol%) as a photocatalyst and

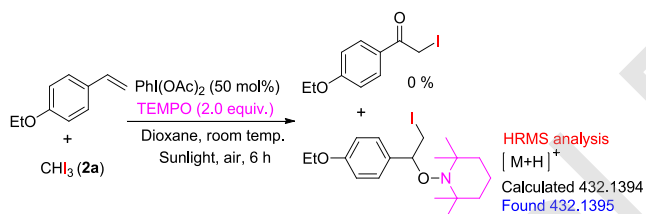
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PhI(OAc)₂ (1.0 equiv.) under ambient conditions with blue LED (450–455nm, 3W) irradiation for 12 h, the corresponding products 2-chloro-1-arylethanones (**5b–5o**) were obtained in 54%–86% yields (Scheme 4). It should be noted that styrenes bearing electric-rich groups such as Me and *t*-Bu or electric-poor groups including Cl, Br, CO₂Me, CF₃, NO₂, CN and F on the *para*-, *meta*- or *ortho*-position of the benzene rings reacted with trichloromethane to afford the according products (**5b–5n**) in satisfactory yields. The reaction of 2-vinylnaphthalene also generated the corresponding product (**5o**) in 86% yield.



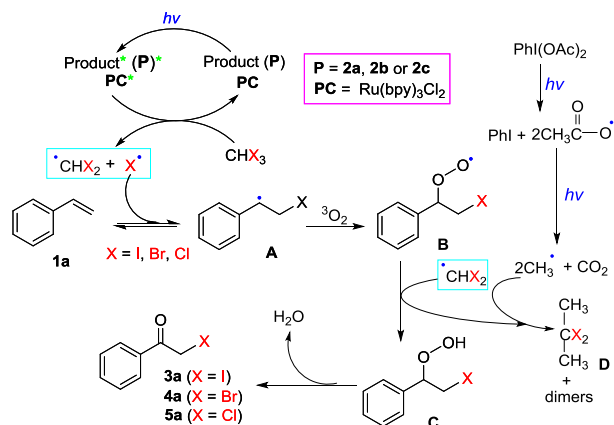
Scheme 4. Preparation of α -chloro ketones from olefins. Reaction conditions: styrene (**1**, 0.20 mmol), trichloromethane (**2c**, 2.0 mL), $\text{Ru}(\text{bpy})_3\text{Cl}_2$ (1.5 mol%), $\text{PhI}(\text{OAc})_2$ (1.0 equiv.) at room temperature under blue LED (450–455 nm, 1.5 W) irradiation in air for 12 h; isolated yields of the products.

To gain insight into the reaction mechanism, a control experiment was conducted, as shown in Scheme 5. The reaction of 1-ethoxy-4-vinylbenzene with **2a** was completely inhibited when a radical scavenger, 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) was added to the reaction, along with the formation of a TEMPO adduct (Scheme 5), which was detected by HRMS analysis (Fig. S2 in Supporting Information). The result indicated a radical process was involved in the reaction.



Scheme 5. Control experiment.

On the basis of above investigation on the control experiment and related papers [47], a possible reaction mechanism is proposed, as in Scheme 6. Photocatalyst (PC) or product α -iodo ketone (P), which has more efficient absorbance in the range visible light wavelength than that of α -bromoiodo ketone and α -chloroiodo ketone (Fig. S1 in Supporting Information), is excited by visible-light irradiation to generate the excited-state of (PC)* or P*. The formed (PC)* or P* then undergoes an energy transfer process with reagents **2a/2b/2c**, generating the corresponding (CHX₂)' and X' radicals via a homolytic cleavage of C–X bond (reaction order: C–I > C–Br > C–Cl). The obtained X' undergoes an addition to styrene **1a** to afford a radical intermediate **A**, which subsequently reacts with ³O₂ to result in the formation of an intermediates **B**. The formed **B** abstracts a hydrogen radical from (CHX₂)' to generate an intermediate **C**, which loses H₂O to provide the desired product **3a/3a/5a**. On the other hand, PhI(OAc)₂ as a promoter undergoes a homolysis to generate PhI and acetoxy radical (CH₃CO₂'), which proceeds a decarboxylation to methyl radical and carbon dioxide under light irradiation [48]. The obtained methyl radical reacts with (CHX₂)', and undergoes further reaction with **B** and itself to give the more stable compound **D** along with the formation of dimers.



Scheme 6. The proposed mechanism.

In summary, we have developed a practical synthesis of α -bromo/iodo/chloroketones from the corresponding olefins under visible-light irradiation conditions. In the presence of $\text{PhI}(\text{OAc})_2$, the reactions of styrenes and triiodomethane undergo to deliver the desired α -iodoketones without additional photocatalyst in high yields under sunlight irradiation and ambient conditions. In addition, the reactions of styrenes with tribromomethane and trichloromethane generate the corresponding α -bromoketones and α -chloroketones in good yields using $\text{Ru}(\text{bpy})_3\text{Cl}_2$ as a photocatalyst under blue LED irradiation. Further studies on mechanism are currently underway.

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