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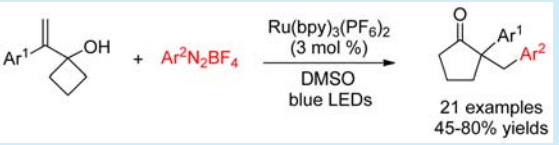
Visible Light Photoredox-Catalyzed Arylative Ring Expansion of 1-(1-Arylvinyl)cyclobutanol Derivatives

Su Jin Kwon and Dae Young Kim*

Department of Chemistry, Soonchunhyang University, Asan, Chungnam 31538, Korea

Supporting Information

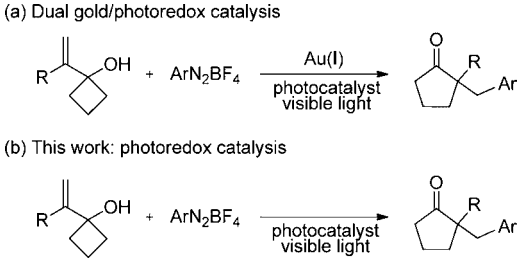
ABSTRACT: A visible light mediated photocatalytic arylation/ring expansion of alkenylcyclobutanols has been developed. This approach provides a mild and operationally simple access to the synthesis of functionalized cyclic ketones from the coupling reaction of alkenylcyclobutanols with aryldiazonium salts.



The difunctionalization of alkenes, which involves the introduction of two functional groups across a double bond, has developed into a powerful strategy for synthesizing useful building blocks for natural products and biologically active compounds.[1] Numerous studies have been conducted on the difunctionalization of alkenes with the aim of developing novel and efficient methods.[2] Recently, a novel application of visible light mediated photoredox catalysis was described for chemical transformations in synthetic organic chemistry and proven to be a powerful tool with a host of attractive features such as mild and environmentally benign reaction conditions, excellent functional group tolerance, and high reactivity.[3] With the use of appropriate radical sources, the photocatalytic difunctionalization of alkenes mediated by visible light offers a viable option for obtaining functionalized molecules.[4] Several groups reported the radical addition and 1,2-carbon migration sequences of α,α-disubstituted allylic alcohol derivatives with various radicals including phosphoryl, sulfonyl, trifluoromethyl, and alkyl radicals using metal-free oxidation and metal- or photomediated oxidation.[5]

Toste and co-workers recently reported a ring expansion and oxidative arylation reaction of alkenylcycloalkanols with aryldiazonium salts promoted by dual gold and photoredox catalysis (Scheme 1a).[6] This reaction involves generation of an electrophilic gold(III)−aryl intermediate through the combination of aryl radicals and a gold(I) catalyst.[7,8] We envisioned the transformation of the alkenylcyclobutanols to the functionalized cyclic ketones by visible light mediated photocatalytic arylation/ring expansion without gold catalyst (Scheme 1b).[9]

Scheme 1. Arylative Ring Expansion of Alkenyl Alcohols



As part of a research program related to the redox reaction and cyclization sequences, we recently reported internal redox reactions via C−H bond functionalization[10] and photoredox-catalytic fluoroalkylation/ring expansion.[11] In this paper, we describe a visible light mediated photocatalytic arylation/ring expansion via 1,2-carbon migration of 1-(1-arylvinyl)-cyclobutanol derivatives.

To determine suitable reaction conditions for the visible light mediated photocatalytic arylation/ring expansion of 1-(1-arylvinyl)cyclobutanols, we examined the visible light mediated photocatalytic reaction of 1-(1-phenylvinyl)cyclobutanol (**1a**) with phenyldiazonium tetrafluoroborate (**2a**) in the presence of 3 mol % of photocatalyst under visible light irradiation with blue LEDs (5 W, λ_{max} = 455 nm) in CH_3CN at room temperature (Table 1). By screening photocatalysts (Table 1, entries 1−6), we found that $Ru(bpy)_3(PF_6)_2$ was the best photocatalyst for this arylation/ring expansion, affording the corresponding product **3a** in 52% yield (Table 1, entry 3). Among the solvents evaluated (Table 1, entries 3 and 7−13), the best result was achieved when the reaction was conducted in DMSO (Table 1, entry 13). Reducing the photocatalyst loading to 1 mol % slightly reduced the product yield (Table 1, entry 14). The control experiment showed that the reaction could not proceed in the absence of a photocatalyst and visible light (Table 1, entries 15 and 16).

With the optimal reaction conditions in hand, we investigated the scope of this visible light mediated photocatalytic arylation/ring expansion via the 1,2-carbon migration sequence of 1-(1-arylvinyl)cyclobutanols **1** with aryldiazonium tetrafluoroborates **2** in the presence of 3 mol % of $Ru(bpy)_3(PF_6)_2$ under light irradiation with blue LEDs in DMSO at room temperature. As shown in Scheme 2, various 1-(1-arylvinyl)cyclobutanols **1** with electron-withdrawing or electron-donating aryl groups furnished the corresponding migration products with moderate to good yields (Scheme 2,

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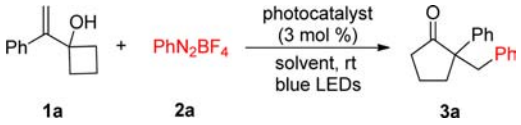
Table 1. Optimization of Reaction Conditions[a]



1a 2a 3a

entry	photocatalyst	solvent	time (h)	yield (%)[b]
1[c]	$Ru(phen)_3Cl_2·H_2O$	CH_3CN	2	43
2[c]	$Ru(bpy)_3Cl_2·6H_2O$	CH_3CN	2	50
3[c]	$Ru(bpy)_3(PF_6)_2$	CH_3CN	6	52
4[c]	*fac*-Ir(ppy)$_3$	CH_3CN	6	trace
5[c]	fluorecein	CH_3CN	6	trace
6[c]	eosin Y	CH_3CN	6	trace
7	$Ru(bpy)_3(PF_6)_2$	DMF	6	trace
8	$Ru(bpy)_3(PF_6)_2$	CH_3CN	5	37
9	$Ru(bpy)_3(PF_6)_2$	CH_2Cl_2	6	0
10	$Ru(bpy)_3(PF_6)_2$	DMF,CH_2Cl_2 (1:1)	6	0
11[c]	$Ru(bpy)_3(PF_6)_2$	acetone	6	0
12[c]	$Ru(bpy)_3(PF_6)_2$	DMF	6	0
13	$Ru(bpy)_3(PF_6)_2$	DMSO	5	75
14[d]	$Ru(bpy)_3(PF_6)_2$	DMSO	6	52
15[e]	$Ru(bpy)_3(PF_6)_2$	DMSO	5	0
16		DMSO	5	0

[a]Reaction conditions: 1-(1-phenylvinyl)cyclobutanol (**1a**, 0.3 mmol), PhN_2BF_4 (**2a**, 0.6 mmol), photocatalyst (0.009 mmol), solvent (2.0 mL) at room temperature under visible light irradiation. [b]Isolated yield. [c]H_2O (10 equiv) was added. [d]1 mol % of photocatalyst loading. [e]The reaction was performed in the dark.

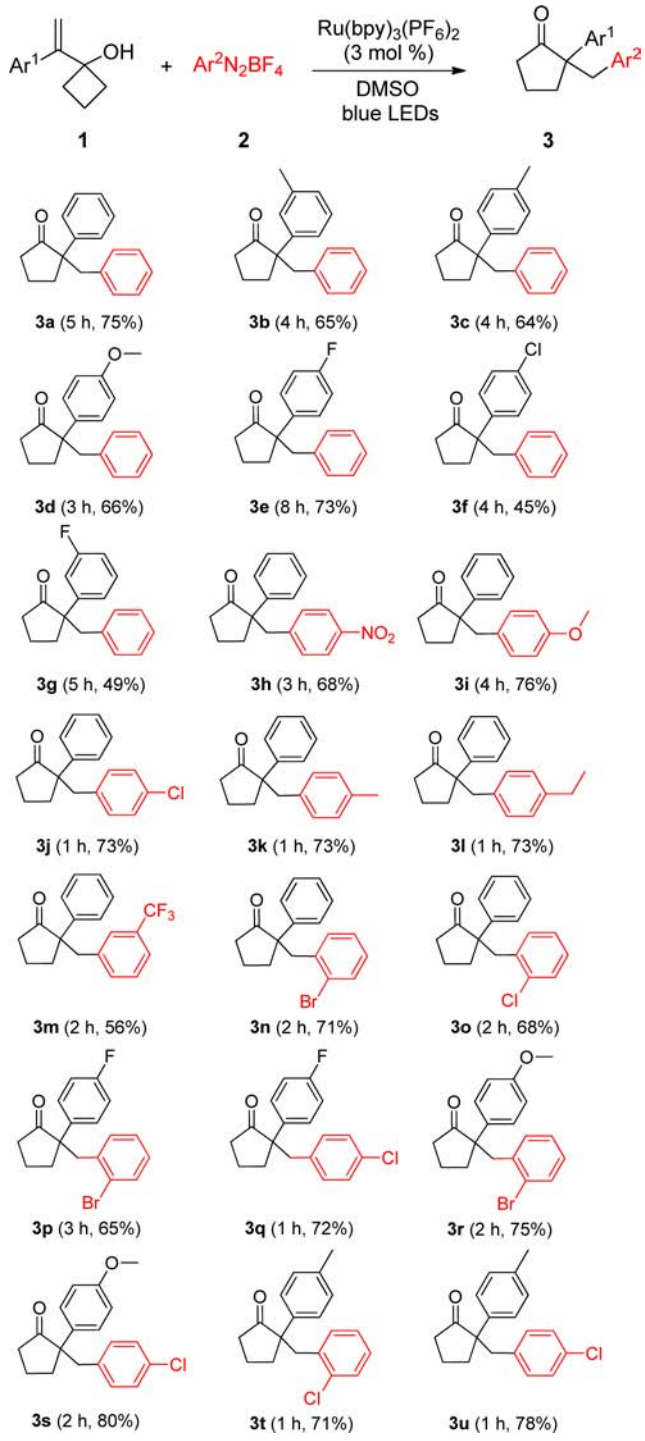
3a–**g**). Additionally, various aryldiazonium tetrafluoroborates **2** with electron-withdrawing or electron-donating aryl groups furnished the corresponding migration products with moderate to good yields (Scheme 2, **3h**–**u**).

1-(3-Phenylprop-1-en-2-yl)cyclobutanol (**1h**) as an aliphatic alkene substrate provided the desired product **3v** with 38% yield under slightly harsh reaction conditions (Scheme 3). In addition, 3-(1-phenylvinyl)oxetan-3-ol (**4**) could undergo photoredox-catalytic arylation/ring expansion under the optimum reaction conditions. Furthermore, 1-(1*H*-inden-3-yl)cyclobutanol derivatives (**6** and **8**) were also used as substrates in this visible light mediated photoredox reaction. It was found that the corresponding products **7** and **9** were obtained in 45 and 39% yield with 3.7:1 dr (Scheme 3).

To illustrate the synthetic utility, we further conducted some functional group transformations. Condensation of cyclopentanone **3u** with H_2NOH in methanol delivered oxime derivative **10** in 72% yield. The cyclopentanone **3u** was reduced to the corresponding cyclopentanol **11** with 95% yield and 4:1 diastereoselectivity (Scheme 4).

To gain mechanistic insights into this transformation, some preliminary experiments were performed. The absence of either the photocatalyst $Ru(bpy)_3(PF_6)_2$ or visible light shut down the reactivity completely, thus suggesting a crucial role for both of these elements for this transformation (Table 1, entries 15 and 16). A trace of the product was detected in the presence of a radical scavenger, 2,2,6,6-tetramethylpiperidin-1-yloxyl (TEMPO). We propose the reaction mechanism shown in Figure 1 based on the results. Irradiation with visible light induces a metal-to-ligand charge transfer in the photocatalyst $Ru^{2+}(bpy)_3(PF_6)_2$, resulting in the excited-state $Ru^{2+}(bpy)_3(PF_6)_2$*. Afterward, a single-electron transfer to aryldiazonium tetrafluoroborate **2** generates $Ru^{3+}(bpy)_3(PF_6)_2$ and aryl radical **I**. Radical **I** then reacts with 1-(1-arylvinyl)-

Scheme 2. Variation of Substrates 1[a,b]



[a]Reaction conditions: 1-(1-arylvinyl)cyclobutanol **1** (0.3 mmol), ArN_2BF_4 **2** (0.6 mmol), $Ru(bpy)_3(PF_6)_2$ (0.009 mmol), DMSO (2.0 mL) at room temperature under visible light irradiation. [b]Isolated yield.

cyclobutanol **1**, yielding intermediate **II**, which is oxidized by the $Ru^{3+}(bpy)_3(PF_6)_2$ to afford the cation **III**, and by the aryl diazonium salt **2** in a radical-chain transfer mechanism. 1,2-Carbon migration of cation **III** leads to a ring expansion that yields the product **3**.

In conclusion, we have developed a novel and environmentally benign process for the visible light mediated

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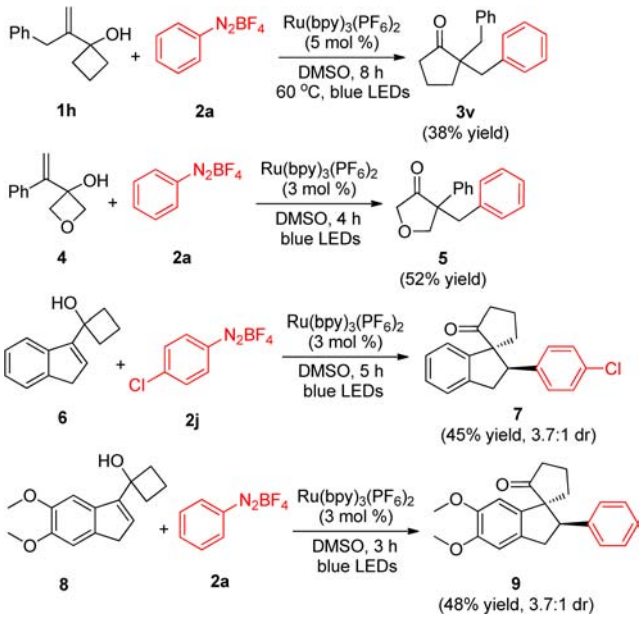
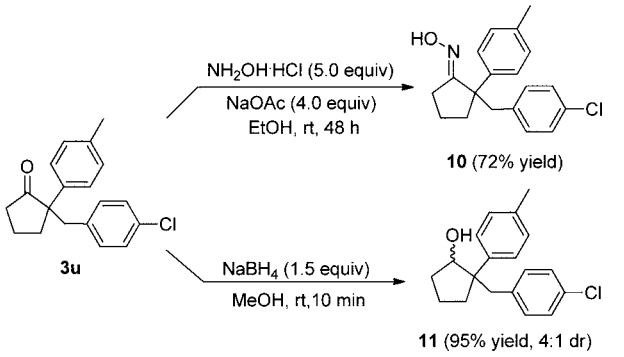
Scheme 3. Visible Light-Mediated Photocatalytic Arylation/Ring Expansion of 1-(3-Phenylprop-1-en-2-yl)cyclobutanol (1h), 3-(1-Phenylvinyl)oxetan-3-ol (4), and 1-(1*H*-Inden-3-yl)cyclobutanol Derivatives 6 and 8



Scheme 4. Transformation of Cyclopentanone Derivative 3



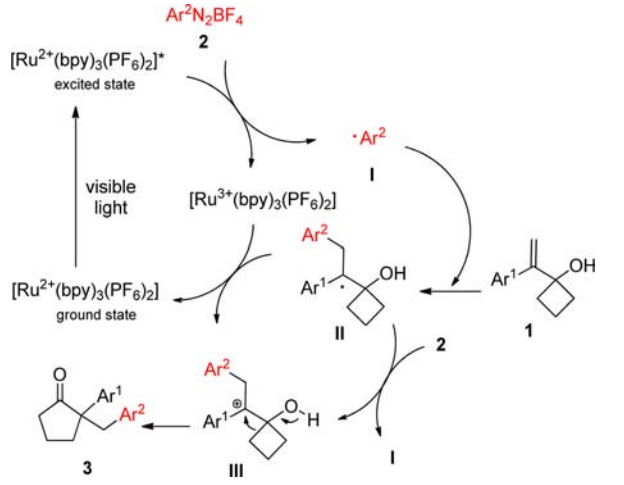


Figure 1. Proposed reaction mechanism.

photoredox-catalyzed arylation/ring expansion via 1,2-carbon migration sequences of 1-(1-arylvinyl)cyclobutanols **1** with aryldiazonium tetrafluoroborates **2** in the presence of 3 mol % of $Ru(bpy)_3(PF_6)_2$. The reaction was completed after a short period without any other metal catalysts. The proposed technique is an efficient option for synthesizing functionalized cyclic ketone derivatives.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b02201.

Detailed experimental procedure and spectral data for all new compounds (PDF)

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: dyoung@sch.ac.kr.

Notes

The authors declare no competing financial interest.

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