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Visible Light-Induced Aerobic Oxyamidation of Indoles: A Photocatalytic Strategy for the Preparation of Tetrahydro-5*H*-indolo[2,3-*b*]quinolinols

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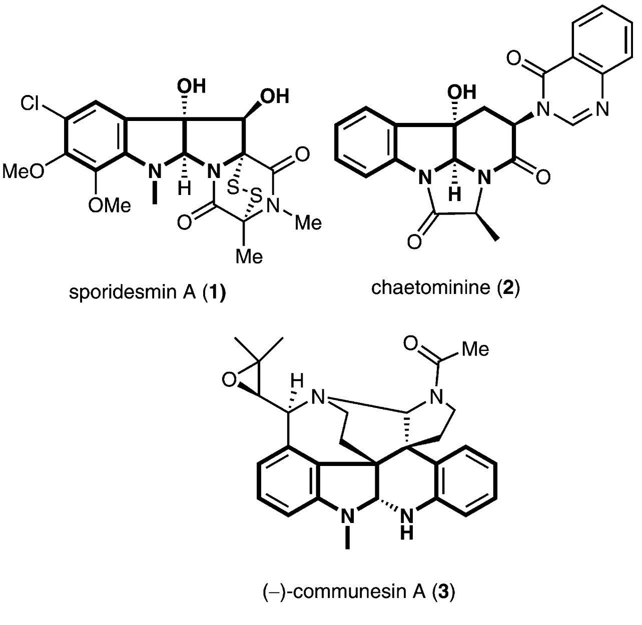
Abstract: A visible light-induced, aerobic oxyamidation reaction of indoles, using air as the sole oxidant, has been developed. This process serves as a photocatalytic strategy to generate efficiently tetrahydro-5*H*-indolo[2,3-*b*]quinolinols, which may have interesting biological and pharmacological activities owing to their privileged indoline structure.

Keywords: aerobic oxidation; indoles; oxyamidation; visible light photocatalysis

The development of strategies for the rapid assembly of novel "drug-like" heterocycles which combine privileged scaffolds of bioactive natural products in one framework has been a focus of intense research work in both organic and medicinal chemistry.[1] In this regard, the 3-hydroxyindolines have attracted much attention due to the numerous bioactive natural alkaloids and pharmaceuticals such as sporidesmin A (**1**)[2] and chaetominine (**2**)[3] containing this architecture. In addition, tetrahydro-5*H*-indolo[2,3-*b*]quinolines form the core of the communesin family of alkaloids[4] which exhibit a wide range of biological activities (Figure 1). Therefore, we became interested in developing an efficient and mild approach to tetrahydro-5*H*-indolo[2,3-*b*]quinolinols which may have interesting biological and pharmacological activities by combining the two privileged framework.

The oxyamidation of indoles[5] has provided a powerful method for the synthesis of hydroxyindolines. In this context, the use of chemical oxidants such as *N*-bromosuccinimide, DMDO and *m*-CPBA constitute the conventional procedures for indole oxyamidation.[6] Recently, the development of reactions that use molecular oxygen in the air as an oxidant[7] and that are promoted by visible light irradiation[8–13] has become an appealing goal because of the natural abundance and cleanliness of the reagents, and the ease and potential applications of the processes. In 2003, Zen and co-workers described a visible light-promoted, photocatalyzed oxidation reaction of sulfides that forms sulfoxides.[12f] Later, Wang and Blechert developed a selective oxidation reaction of alcohols and amines that employs O_2 and the photocatalyst mpg-C_3N_4.[12g,h] Recently, the group headed by Jiao uncovered an aerobic oxidative transformation of benzylic halides to α-aryl carbonyl compounds.[12i]



sporidesmin A (**1**)

chaetominine (**2**)

(–)-communesin A (**3**)

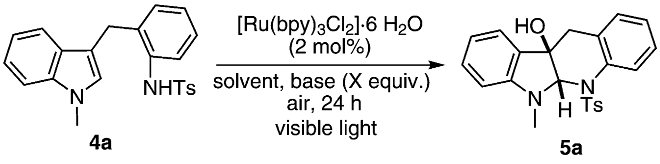
Figure 1. Representative biologically active indoline derivatives.

Quite recently, Yoon and co-workers described a photocatalytic aerobic [2+2+2] cycloaddition reaction used for the synthesis of endoperoxides.[10e]

Our studies in this area also have led to the development of a visible light-initiated, oxidative hydroxylation reaction of arylboronic acids that utilizes air as a terminal oxidant.[13a] As part of an ongoing program targeted at the development of methodologies for carbocyclic and heterocyclic ring synthesis and one whose particular focus is on photocatalytic oxidation processes, we herein describe a visible light-induced, aerobic oxyamidation reaction of indole derivatives that produces tetrahydro-5*H*-indolo[2,3-*b*]quinolinols.

The initial phase of this investigation focused on an examination of the visible light promoted reaction of the linked indole-*N*-tosylamide **4a** catalyzed by $[Ru(bpy)_3Cl_2]\cdot 6H_2O$ (2 mol%) in the presence of Et_3N (2.0 equiv.) and air (air without bubbling). Under these conditions and using a 24 h irradiation period, an oxyamidation reaction occurs to give the tetrahydroindolo[2,3-*b*]quinolinol product **5a** in 51% yield (Table 1, entry 2). Note that either low or no conversion of **4a** to **5a** was observed to take place when any one of the components of the reaction mixture was absent. A brief survey of solvent for the reaction revealed that CH_3CN is optimal (Table 1, entries 2–4). The results of screening several organic and inorganic bases showed that reactions promoted by tetramethylguanidine (TMG) produce the highest yield of **5a** (Table 1, entries 4–9). We observed that the efficiency of the process is improved to 71% yield when 1.0 equivalent of TMG is present in the mixture (Table 1, entries 8, 10 and 11). Finally, a catalyst loading as low as 0.5 mol% can be employed without incurring a significant loss in reaction efficiency (Table 1, entry 12).

Table 1. Optimization of the reaction conditions.[a]



Entry	Solvent	Base	X	Yield [%][b]
1	DMF	–	–	trace
2	DMF	Et_3N	2.0	51
3	DMSO	Et_3N	2.0	32
4	CH_3CN	Et_3N	2.0	52
5	CH_3CN	TMEDA	2.0	42
6	CH_3CN	DABCO	2.0	64
7	CH_3CN	pyridine	2.0	11
8	CH_3CN	TMG	2.0	65
9	CH_3CN	KOH	2.0	49
10	CH_3CN	TMG	1.0	71
11	CH_3CN	TMG	0.3	62
12[c]	CH_3CN	TMG	1.0	71

[a] *Reaction conditions:* **4a** (0.30 mmol), $[Ru(bpy)_3Cl_2]\cdot 6H_2O$ (2 mol%), base (X equiv), solvent (3 mL), 36 W fluorescent lamp irradiation, air.

[b] Yield of isolated product.

[c] 0.5 mol% $[Ru(bpy)_3Cl_2]\cdot 6H_2O$ were used and the quantum yield was 0.16 (see the Supporting Information for details).

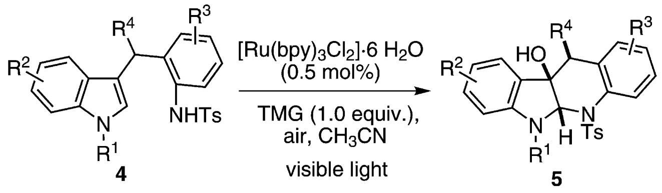
With optimal conditions in hand, the scope of substrates that undergo the oxyamidation reaction was explored. As the results summarized in Table 2 show, a broad range of electronically and sterically diverse, and both free N−H and N-protected indole-derived *N*-tosylanilides participate in this photoinduced reaction (Table 2, entries 1–4). Moreover, incorporation of methyl, methoxy and benzyloxy groups at various positions of the indole ring or anilide benzene ring does not compromise the efficiency of the process (Table 2, entries 5–10, 14 and 15). Variations of the electronic nature of the indole ring or benzene ring are also well tolerated, as demonstrated by the efficient reactions of electron-deficient halogen-substituted substrates (Table 2, entries 11–13 and 16–18). Moreover, the substrate containing a methyl group at the benzylic position undergoes the reaction to give the corresponding product **5s** in good yield and with an excellent level of diastereoselectivity (Table 2, entry 19).

In order to gain insight into the mechanism of the oxyamidation reaction, two ^{18}O-labeling experiments summarized in Eqs. (1) and (2) were carried out (Scheme 1). The results show that the hydroxy oxygen atom in the product originates exclusively from molecular oxygen rather than water.[15]

Based on the observations described above, the pathway for the formation of the tetrahydro-5*H*-indolo[2,3-*b*]quinolinol products **5**, shown in Scheme 2, is possible. The indolyl radical cation **I** is formed through photoinduced one-electron oxidation of the indole-derived *N*-tosylanilide **4a** (+0.4 V *vs.* SCE)[15–17] promoted by $Ru^*(bpy)_3^{2+}$ (+0.8 V). This electrophilic intermediate reacts with superoxide radical anion, produced through reduction of molecular oxygen by the Ru(I) complex,[12b,15] to form the intermediate **II** or **III**, which then undergoes intramolecular cyclization to afford the corresponding anion intermediate **IV**. The formation of **5a** occurs in the final steps by protonation and subsequent cleavage of the O−O bond.[18,19]

Saito and Nakagawa[20] demonstrated earlier that tryptophan derivatives undergo oxidative conversion to generate 3-hydroperoxyindolines in the presence of singlet oxygen,[21] which was generated by using a dye photosensitizer, bubbling O_2 and halogen lamp irradiation. These workers observed that the photooxidation reaction is inhibited by DABCO or Et_3N,[22] which are known to be singlet oxygen quenchers even at low concentration. However, the results of the current effort (Table 1, entries 2–6) show that the trans-

Table 2. Visible-light-induced aerobic oxyamidation reactions of indoles.[a]



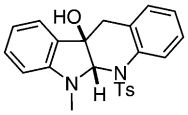
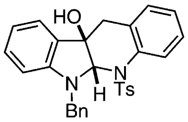
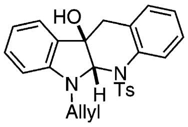
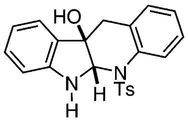
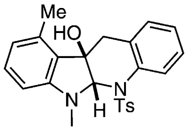
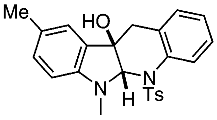
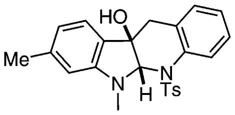
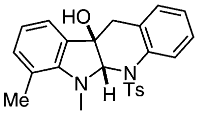
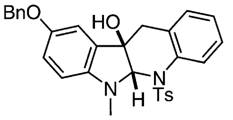
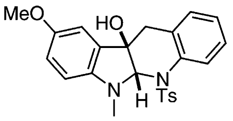
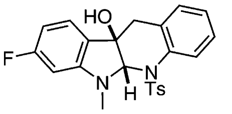
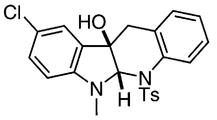
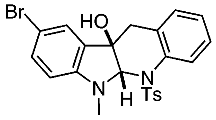
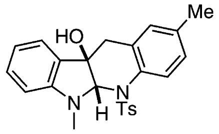
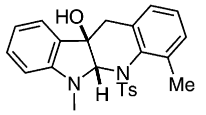
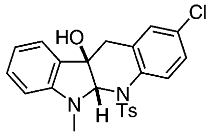
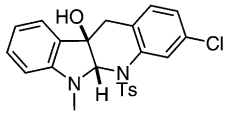
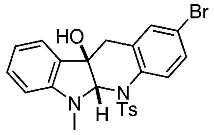
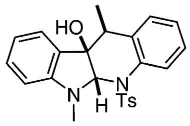
Entry	Product		Time [h]	Yield [%][b]	*dr*[c]
1		**5a**	16	71	>95:5
2		**5b**	21	70	>95:5
3		**5c**	24	66	>95:5
4		**5d**	24	49	>95:5
5		**5e**	16	67	>95:5
6		**5f**	16	60	>95:5
7		**5g**	20	69	>95:5
8		**5h**	20	64	>95:5
9[d]		**5i**	54	60	>95:5
10[d]		**5j**	54	62	>95:5
11		**5k**	16	64	> 95:5
12		**5l**	16	66	>95:5

Table 2. (Continued)

Entry	Product		Time [h]	Yield [%][b]	*dr*[c]
13		**5m**	20	72	>95:5
14		**5n**	24	65	>95:5
15		**5o**	30	61	>95:5
16		**5p**	30	69	>95:5
17		**5q**	30	63	>95:5
18		**5r**	24	79	>95:5
19[e]		**5s**	24	77	5:1

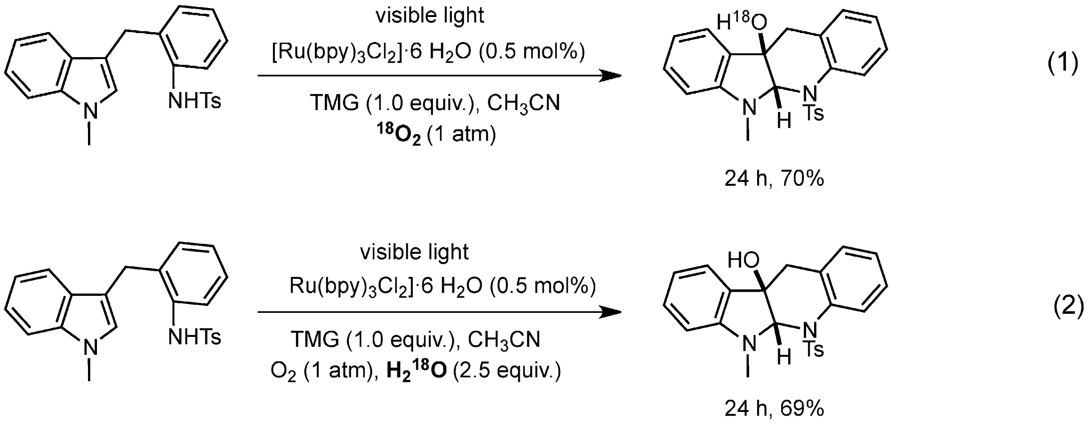
[a] *Reaction conditions:* **4** (0.25 mmol), [Ru(bpy)$_3$Cl$_2$]·6H$_2$O (0.5 mol%), TMG (1.0 equiv.), CH$_3$CN (2.5 mL), 36 W fluorescent lamp irradiation, air.

[b] Yield of isolated product.

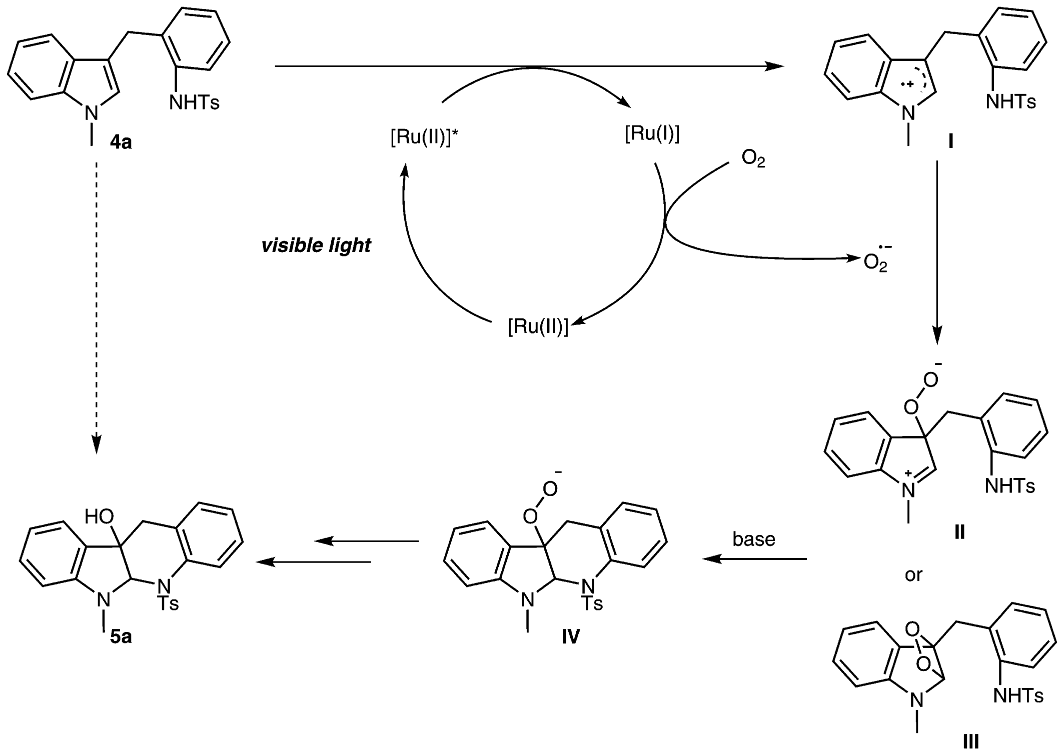
[c] Determined by using ^{1}H NMR methods.

[d] DMF was used instead of CH$_3$CN.

[e] The structure of **5s** was further confirmed by X-ray analysis; see ref.[14]



Scheme 1. ^{18}O labeling experiments.



Scheme 2. Proposed mechanism for the oxyamidation of indoles.

formation of **4a** to **5a** takes place in up to 64% yield even in the presence of 2.0 equivalents of amines. These findings suggest that the current photosensitized oxyamidation process probably involves a radical mechanism, although the singlet oxygen-mediated process cannot be ruled out. Further investigations on the details of the mechanism are ongoing.

In conclusion, the studies described above have resulted in the development of a visible light-induced oxyamidation reaction of indoles using molecular oxygen in air as the oxidant. The process produces a variety of densely functionalized tetrahydro-5*H*-indolo[2,3-*b*]quinolinols in moderate to good yields. Further investigations of the mechanism as well as synthetic applications of this reaction are currently underway in our laboratory.

Experimental Section

General Procedure for Visible Light-Induced Aerobic Oxyamidation of Indoles to Tetrahydro-5*H*-indolo[2,3-*b*]quinolinols

To a mixture of 4-methyl-*N*-{2-[(1-methyl-1*H*-indol-3-yl)methyl]phenyl}benzenesulfonamide **4a** (0.25 mmol, 97.6 mg), Ru(bpy)$_3$Cl$_2$·6H$_2$O (0.00125 mmol, 0.9358 mg) in CH$_3$CN (2.5 mL) was added TMG (0.25 mmol, 28.8 mg). The solution was stirred at room temperature under visible light irradiation in the open air (without bubbling air). After **4a** had been completely consumed (monitored by TLC analysis), the solvent was evaporated under vacuum. The product was then purified using flash column chromatography on silica gel (petroleum ether/ethyl acetate = 5/1 as eluant) to afford the desired product **5a** as a white solid; yield: 72.2 mg (71%).

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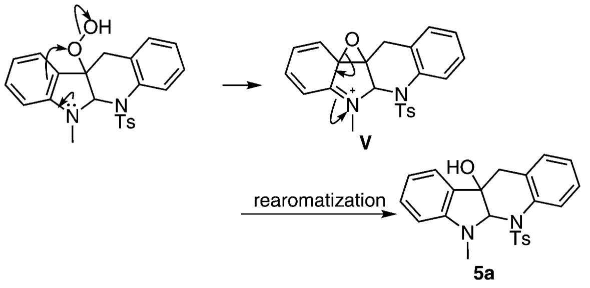

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COMMUNICATIONS

8 Visible Light-Induced Aerobic Oxyamidation of Indoles: A Photocatalytic Strategy for the Preparation of Tetrahydro-5*H*-indolo[2,3-*b*]quinolinols

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