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Blue LED-driven C-N bond formation for synthesis of imidazopyridines

Amardeep R Jadhao[a], Shankesh C Zyate[b]& Sanjay S Gaikwad*[c]

[a] Department of Chemistry, Late Pushpadevi Patil Arts and Science College, Risod, Dist. Washim 444 506, Maharashtra, India

[b] Department of Chemistry, Shri R. L. T. College, Akola 444 001, Maharashtra, India

[c] Department of Chemistry, MES, Abasaheb Garware College, Pune 411 004, Maharashtra, India

E-mail: sanjchem99@gmail.com

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The formation of C-N bonds has emerged as a powerful key for structural modifications in organic synthesis. Although transition metal-free procedures provide a viable protocol to construct hetero molecules, the use of costly catalysts and high temperatures has restricted universal applicability. Moreover, radical-based C-N bond formation without the assistance of a transition metal catalyst has been a great challenge. Herein we report the blue LED-driven transition metal-free strategy for C-N bond formation. This reaction features easy-to-handle, mild reaction conditions, reactants and good functional group tolerance. The mechanistic investigation suggests that this strategy proceeds through phenacyl bromide radical precursor and NBS reaction mediated by H_2O to construct IPs.

Keywords: Blue LED irradiation (BLI), C-N bond formation, Transition metal-free, IPs

The formation of C-N bonds has emerged as a powerful key for structural modifications in organic synthesis, heterochemistry, and medicinal chemistry[1-6]. Many researcher efforts have been devoted to developing new and efficient methodologies in this field. Among such strategies, multicomponent reaction, hydroamination, tandem methods, and hydroamination have recently emerged as a particular, efficient, and powerful approach for C-N bond formation[7-9].

It has been acknowledged that the advancement of visible-light-mediated synthesis represents an excellent route for a variety of organic transformations[10]. Various methods involving transition metals, certain organic dyes, and nanoparticles as photo catalysts have been reported in the literature[11,12]. However, there are several disadvantages associated with transition-metal photocatalysts, since they have disadvantageous inherent malignancy and high cost. Photochemistry comes with a green approach so the selection of reagents and solvents becomes more valuable. In this scenario, a catalyst-free synthesis that uses a green solvent medium in a variety of synthetic processes has become an important tool.

Imidazopyridines (IPs), nitrogen-fused hetero-cycles, are regarded as essential structural units because of their wide range of applications in material science, pharmaceutical chemistry, and organo-metallics[13-16]. Moreover, Ips are important scaffolds due to their presence in various top commercial drugs such as necopidem, sariplдem, zolpidem, alpidem, and miroprofen (Fig. 1)[17]. The Ips nucleus is present in several biologically active natural products. IPs are of special interest since they exhibit a wide range of biological activities and are often chemically produced from natural sources[18,19]. Because of their powerful inhibitory activity against various cancer cell growths

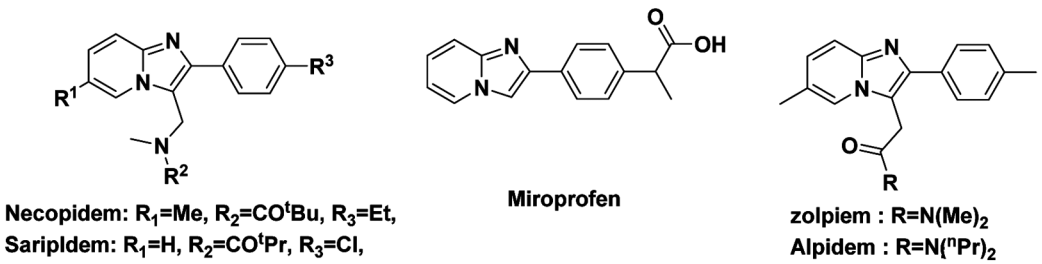


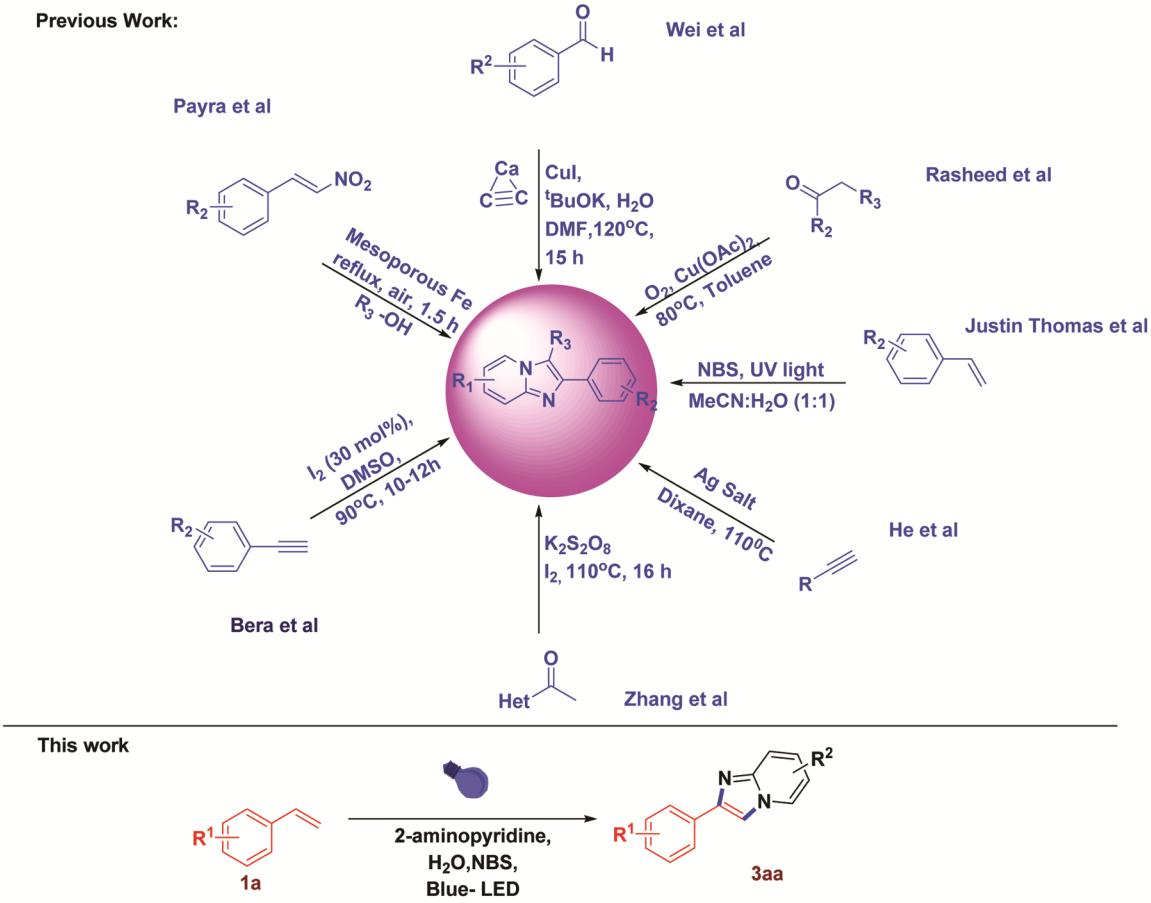
Fig. 1 — Examples of imidazo[1,2-a]pyridine-based drugs

and migration, these chemicals have recently caught the interest of researchers as potential anticancer therapies[20].

In recent advances, Wei *et al*. (2022) reported an oxidative cyclization method for the synthesis of 3-Methyl-2-arylimidazo[1,2-*a*]pyridines by using calcium carbide with a substituted aldehyde in the presence of organic solvents and strong bases which involves the heating for a prolonged period[21]. Zhang *et al*. (2021) reported a three-component reaction for the synthesis of 3-aroylimidazo[1,2-*α*]-*N*-heterocycles using I_2 catalyst in the presence of dimethyl sulfoxide as a methylene donor, this method also suffers from prolonged heating[22]. Rasheed *et al*. reported the synthesis of substituted imidazopyridines by oxidative cyclization in the presence of $Cu(OAc)_2$. Similarly, Bera *et al*. developed a transition metal-free, chemoselective synthesis of imidazo[1,2-*a*]pyridine derivatives[23]. Payra and co-workers reported the synthesis of 2-alkoxyimidazo[1,2-a]pyridines in the presence of Fe–SBA-15 catalyst[24]. Sivappa *et al*. have developed a one-pot synthesis of 3-substituted imidazo[1,2-a] pyridines[25a]. Thomas *et al*. in 2020 reported the synthesis of imidazopyridines from styrene, NBS, and 2-aminopyridines in the presence of a UV-fluorescent lamp. This route provides a good option for obtaining imidazopyridines but the presence of UV-fluorescent light causes a serious disadvantage due to its mutation-causing property on over-exposure[25b]. So, there is still a great need to develop a new eco-friendly and advantageous route towards the synthesis of imidazopyridines.

Over the last decade, the photochemical reaction has attracted much attention due to environmental friendliness, improved selectivity, and increased reaction rate[26-30]. Among them, the photocatalytic formation of C-N bonds is an efficient and valuable tool for heterochemistry. Photolytic C-N bond formation of cyclic compounds especially IPs has been neglected.

In continuation to our previous research[31,32],we hereby report a simple and potent blue LED-driven transition metal-free, C-N bond formation strategy for one-pot synthesis of imidazopyridines (IPs) with mild reaction condition reactants and good functional group tolerance (Scheme 1). Notably, the reaction pathway



Scheme 1

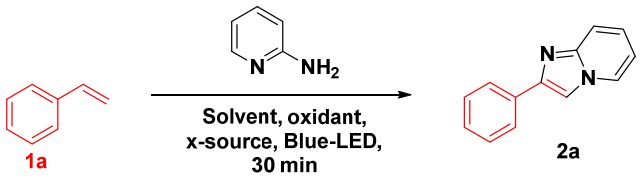
involves a blue LED, phenacyl bromide radical precursor, and NBS in green solvent H_2O to construct IPs.

Results and Discussion

To the best of our knowledge and literature survey, radical-based synthesis of IP has not been reported with styrene. Therefore, by considering the pharmaceutical importance of IPs, we focused our attention on the development of a transition metal-free, one-pot route from styrene to Ips in the presence of NBS and water as solvent relying on an environmentally more benign initiation process than the one used traditionally.

Optimization of the reaction was thus carried out by varying the x-source, oxidant, and solvent (Table 1). Simple styrene (**1a**) with iodine in the presence of *tert*-butyl hydroperoxide (TBHP, entry 1) or 2-iodoxybenzoic acid (IBX, entry 2 and 3) as an oxidant in DCE and DMSO as a solvent on irradiation with blue LED gratifyingly, observed the formation of intended IPs, **2a** in trace yields of 10%, 13%, and 16% respectively, confirmed by isolation and NMR analysis.

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



Entry	Solvent	X source	Oxidant	Yield of **2a** (%)[b]
1	DCE	I_2	TBHP	10
2	DCE	I_2	IBX	13
3	DMSO	I_2	IBX	16
4	DMSO	NIS	IBX	28
5	DCM	NIS	IBX	25
6	DEE	NIS	IBX	23
7	THF	NIS	IBX	22
8	DMSO	KI	IBX	trace
9	DMSO:H_2O	KI	Oxone	00
10	ACN:H_2O	KI	$K_2S_2O_8$	33
11	Dioxane:H_2O	NBS	–	38
12	H_2O	Br_2	air	00
13	EtOH:H_2O (1:1)	NBS	–	72
14 [c]	EtOH:H_2O (1:1)	NBS	–	60
15	H_2O	TBAB	air	00
16	H_2O	NCS	–	00
17	H_2O	NIS	–	25

[a]Condition: Styrene (1 mmol), X-source (2 mmol), oxidant (1 mmol), solvent (2 mL), and then 2-aminopyridine (2 mmol), in blue LED for 30 min. [b]Isolated yields.[c]NBS was added in two portions after the first step.

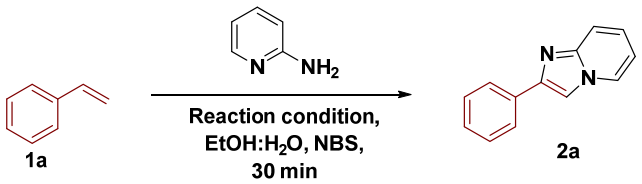
When styrene was treated with another iodine source *i.e.*, *N*-iodosuccinimide (NIS), using IBX as an oxidant in DMSO, DCM, DEE, THF as a solvent, and blue LEDs irradiation was used to produce the desired product **2a** in 22-28% yield (entry 3-7) indicating X-source was required for the formation of radical phenacyl iodide with polar solvent favours the reaction. Interestingly, we noticed that the use of KI in the presence of oxone failed to produce the intended product (entry 9), whereas KI in the presence of $K_2S_2O_8$ produced the desired product **2a** in 33% yield (entry 10). To analyze the role of oxidant we tried to run a reaction without oxidant in NBS using dioxane: H_2O solvent-cosolvent system which surprisingly furnished desirable product **2a** in moderate yield (entry 11) indicating the prominent role of air as an oxidant. We also tested additional bromine sources, such as molecular bromine in water (Br_2, entry 12) and tetrabutylammonium bromide (TBAB, entry 15), with air as the oxidant, but no product formation was observed. Furthermore, the scope of the reaction was then established using optimized conditions (entry 13), the intended product, **2a** was obtained by blue LED-assisted irradiation of styrene, using EtOH:H_2O (1:1) and NBS (2.0 mol equiv.) with 2-aminopyridine, in a good yield of 73%. At last, when NBS was substituted with NCS, the reaction failed to produce the desired product (entry 15), whereas, on using NIS the IP **2a** was obtained in low yield (Entry 16). After 24 h irradiation, the styrene could not be fully consumed.

After the realization of the best solvent system and oxidant for the synthesis of IPs, we further investigated different sources and irradiation with visible light. It was found that when we performed the reaction in daylight with standard reaction conditions, the product formed readily (Table 2, entry 1, 46% yield) however, in the absence of light the rate of product formation was significantly reduced (Table 2, entry 2). We furthermore performed the reaction in the compact fluorescent lamp (CFL of 18W, 20W, and 24W) the product was obtained in 38%, 52%, and 56% yields respectively (Table 2, entry 3-6). Furthermore, with the specific intensity of the green light, we get desired product in 43% yield (Table 2, entry 7). However, with the blue intensity of light, the rate of reaction enhances to furnish product by 72% (Table 2, entry 8). Once the optimal conditions had been finalized, the scope of the synthetic strategy was explored by a series of different derivatives of styrenes (**1**), and 2-aminopyridines (**2**).

Substrate scope using a series of styrenes:

With these optimized reaction conditions in hand, the substrate scopes of the reaction were explored (Scheme 2). It was found that styrenes with different substitutions reacted quite well under the optimal reaction conditions (**2a-2h**). The incorporation of electron-donating groups (*e.g.* -Me) or electron-withdrawing groups (*e.g.* -Br, -Cl) did not influence the reaction efficiency, affording the corresponding products in moderate to good yields.

Table 2 — Optimization of source



Entry	Source of radiation	Time (min)	Yield[b] (%)
1	Daylight	30	46
2	No light	30	trace
3	7W, White LED	30	16
4	18W, CFL	30	38
5	20W, CFL	30	52
6	24W CFL	30	56
7	Green LED	30	43
8	Blue LED	30	72

[a] Reaction conditions: **1a** (1.0 mmol), NBS (2.0 mmol) in ethanol: water irradiated using CFL under open air at room temperature. [b] Isolated yield of the product (%). NR=No reaction.

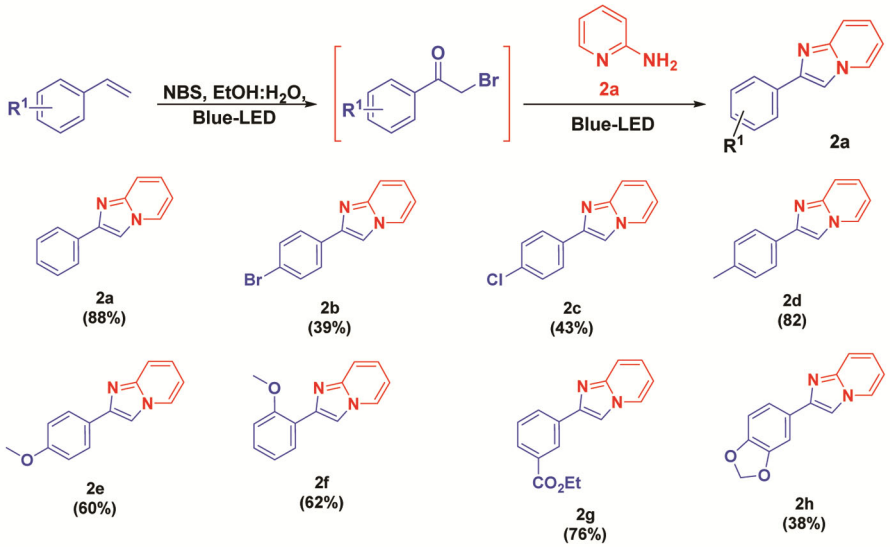
Note that, halo-substituted styrene also produced the expected products **2b**, and **2c** in 39% and 43% yield respectively. Next, we turned our attention to investigating the reactivity of methoxy-substituted styrenes. Generally, a wide range of IPs can be achieved with moderate to good yields (**2e-2h**).

To show the scalability of this Blue LED-promoted catalyst-free cyclization process, the reaction of **1a** with 2-aminopyridine was performed at a 1.5 mmol scale, and product **2f** could be obtained in 62% yield. It should be pointed out that a series of important functional groups, such as –Br (**2b**), 2-OMe (**2f**), COOEt (**2g**), and 1,3-dioxol (**2h**), are well tolerated. The relatively low yield of the desired product in some cases might be attributed to the poor solubility of the corresponding styrene starting materials in water.

Substrate scope using a series of 2-aminopyridines:

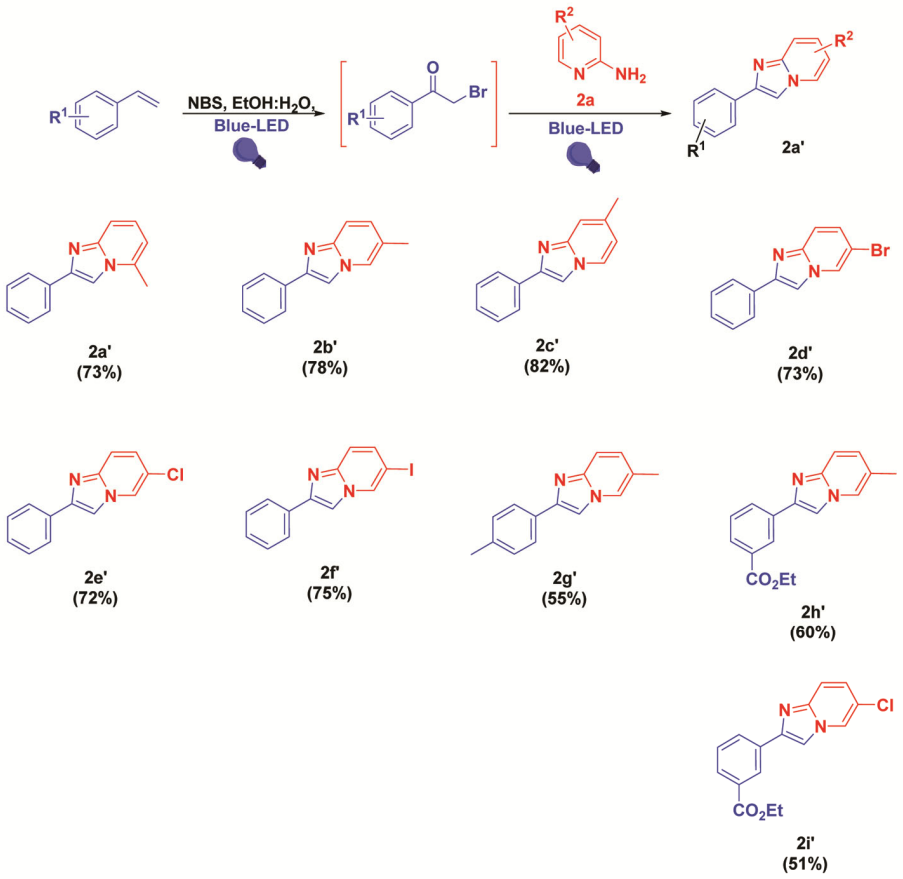
To expand the potential utility of this protocol for synthetic organic chemistry, we turn our attention toward substituted 2-aminopyridine (Scheme 3). The scope of 2-aminopyridinewith simple, non-substituted styrene was further investigated.

A range of substituted 2-aminopyridines *i.e.*, 5-Me, 6-Me, 7-Me,6-Br, 6-Cl, 6-I proceeds finely to provide the corresponding imidazo[1,2-α]pyridine **2a'-2f'** in very good yields between 77-86% (Scheme 3). 6-Methyl, 2-aminopyridine with tolyl styrene and carboxylate styrene proceeded smoothly to deliver the



Reaction Conditions: Styrene (1 mmol), NBS (2 mmol), EtOH:H_2O (1:1 v/v 2 mL), and then 2-aminopyridine (2 mmol), in blue LED for 30 min

Scheme 2 — The reaction substrate scope of the styrene



Reaction Conditions: Styrene (1 mmol), NBS (2 mmol), EtOH:H_2O (1:1 v/v 2 mL), and then 2-aminopyridine derivative (2 mmol), in blue LED for 30 min

Scheme 3 — The reaction substrate scope of the 2 aminopyridines

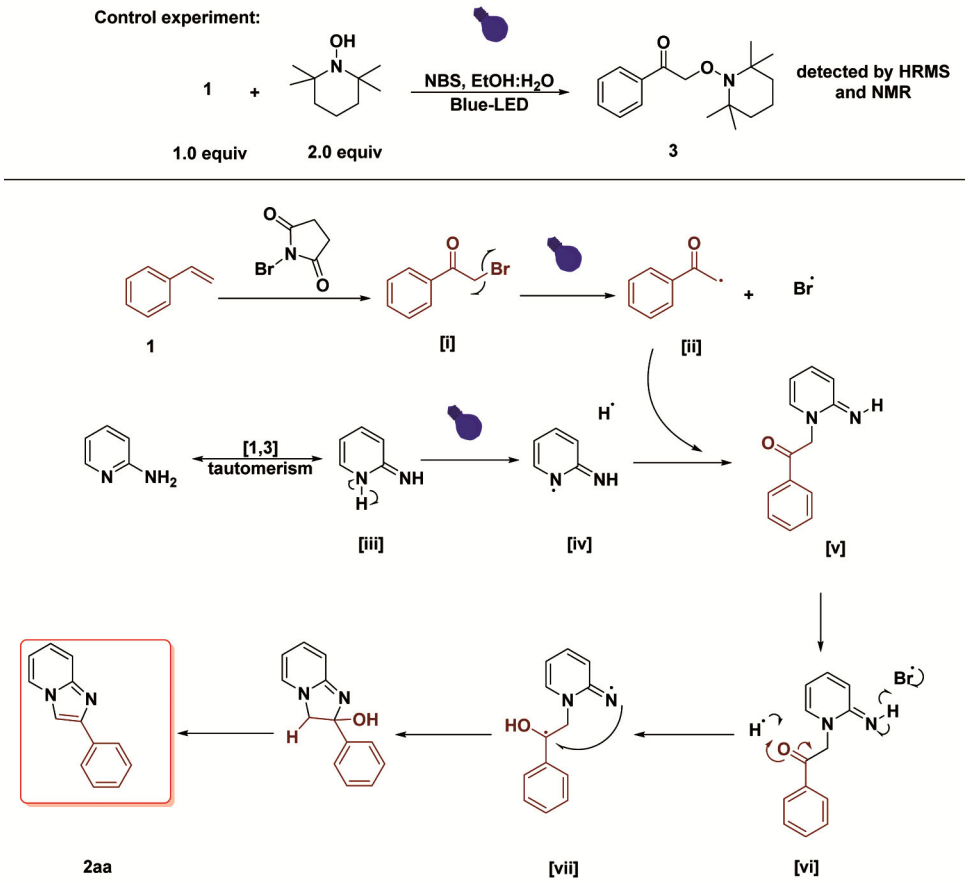
products **2g'-2h'** in 55-60% yields. Interestingly, 6-chloro-2-aminopyridine afforded the targeted products **2i'** in 51% yield.

It has been observed that simple styrene provided a desirable product with good yield compared to substituted styrene due to its good agreement with an electronic influence. Also, substitution on 2-amino pyridines does not influence the yield.

Reaction with either electron donating (-OMe) or electron-withdrawing (-X) substrate proceeded successfully to give moderate to a good yield of the desired product. -Br, -Cl substituted styrene, shows moderate yield due to electron withdrawing nature. -OMe, -Me substituted styrene shows a good yield of product, which might be attributed to its electron-donating nature. Higher yields are obtained with electron-donating substituents on the styrene, illustrating the influence of electronic effects on the reaction.

To demonstrate the versatility of this method, to our delight when the styrene was reacted with sodium sulfinate salt (**3a**) under the same reaction conditions, the expected *β*-keto sulfones (**4a**) formation was observed in 83% yield. This result encouraged us to further study the substrate scope of the sulfonation reaction the reaction was thoroughly investigated under the previously optimized conditions using varying combinations of styrene (**1**) and sodium sulfinate salts (**3**) as shown in Scheme 4.

Halo-substituted styrenes and aryl sodium sulfinate salts react nicely under optimized conditions to furnish consistent *β*-keto sulfone. *para*-Bromo and chloro-substituted styrenes react with sodium 4-methyl sulfinate to yield *β*-keto sulfone **4b** and **4c** in 81% and 80% yields respectively. Whereas, *ortho*-chlorostyrene furnishes the product **4d** in 75% yield. *para*-Substituted benzyl sodium sulfinate salts react nicely under given conditions with styrene **1a** to furnish the products **4m**, and **4n** in good yields of 82% and 83% respectively. Alkyl-substituted styrenes and aryl sodium sulfinate salts also react well to furnish the corresponding *β*-keto sulfone **4e**, **4f**, **4k,**



Scheme 4 — Control experiment and plausible reaction mechanism

and **4l** in good to very good yields. *para*-Substituted alkyl group on either of the reacting species like *p*-Me styrene and *p*-^{t}Bu sodium aryl sulfinate give considerably enhanced yields than the *m*-Me styrene and *o*-Me sodium aryl sulfinate salts. *p*-OMe styrene provides an 87% yield of the product **4g**, and *p*-OMe substituted sulfinate salt also furnishes a comparative yield of product **4o** in 85%. *m*-ester substituted styrene gives declined yields of corresponding sulfone **4h** (65%), this might be due to decreased nucleophilicity of styrene during phenacyl bromide formation. The same trend is observed on the heterocyclic styrene **4i**. *p*-NO_2 substituted sulfinate salt gives lesser yields of the product (51%, **4p**). Naphthyl sulfinate salts react nicely under the provided conditions to give *β*-keto sulfone **4q** in a very good yield of 84%. When the aliphatic sodium sulfinate salt was used instead of aromatic sulfinate salt, unfortunately, the *β*-keto sulfone **4r** formation was not observed under the developed conditions.

A plausible mechanism based on the results of control studies and literature survey[33-35] is depicted the Scheme 4. The reaction involves the NBS/H_2O-promoted synthesis of bromohydrin, followed by the NBS-mediated oxidation of secondary benzylic alcohol for the *in-situ* generation of phenacyl bromide [**i**]. On other hand, the reaction was initiated by [1,3] tautomerism in 2-aminopyridines to imino compound [**ii**], After this radical [ii] combines with [**iv**] to give compound [**v**] as depicted in **Scheme 4**. The homolytic cleavage of the N-H bond supported by free radical bromine takes place. Simultaneously, the carbonyl group gets cleaved to convert into a hydroxyl group to afford [**vi**]. Finally [**vii**] coalesces to form the desired product *via* cyclization followed by re-aromatization. However, the generation of [**i**] was shown by a radical trapping experiment using TEMPO, which led to the formation of **3**, which was also confirmed by the NMR spectrum (see supporting information).

Conclusion

In summary, we reported a straightforward transition-metal-free, strategy for constructing imidazopyridines and *β*-keto sulfones *via* C-N and C-S

bond formation **in** a one-pot, blue LED mediated addition of styrenes and 2 aminopyridines/ sodium sulfinate salts. Although the overall yield is moderate, this offers access to useful intermediate and a new way to generate synthons which is still a challenging task in organic synthesis. The reaction proceeds at a mild temperature, making the reaction conditions easy to operate. Our current protocol tolerates various functional groups and can be applied to strained imidazopyridines/*β*-keto sulfones like scaffolds. More importantly, this one-pot protocol can be applied to the synthesis of pharmaceutical imidazopyridines/*β*-keto sulfones-based drug molecules. Application of this methodology to the synthesis of bioactive targets is under progress in our research group.

Supplementary Information

Supplementary information is available in the website http://nopr.niscpr.res.in/handle/123456789/58776.

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