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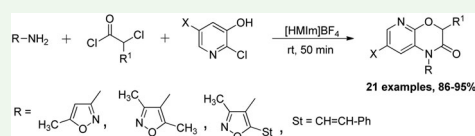
A fast and highly efficient one-pot synthesis of novel isoxazolyl pyrido[2,3-*b*][1,4]oxazin-2(3*H*)-ones via Smiles rearrangement using task-specific ionic liquid [HmIm]BF₄ as green solvent

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ABSTRACT

A facile and convenient procedure for the synthesis of isoxazolyl pyrido[2,3-*b*][1,4]oxazin-2(3*H*)-ones via Smiles rearrangement from isoxazole amine, chloroacetyl chloride and 2-chloro-3-hydroxypyridine using [HmIm]BF₄ as task-specific ionic liquid has been described. The protocol proves to be efficient and environmentally benign in terms of high yields, eco-friendly solvent, ease of recovery, and reusability of reaction medium.



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Isoxazolyl pyrido[2,3-*b*][1,4]oxazin-2(3*H*)-ones; Smiles rearrangement; ionic liquid; green chemistry


Introduction

Environment protection laws and corporate pressure to minimize the amount of toxic waste arising from chemical syntheses have motivated the development of innovative and eco-friendly chemical technologies. In this context, room-temperature ionic liquids, especially those based on 1-methyl imidazolium cation, have given a promise as attractive alternative to conventional solvents (1,2). The use of room-temperature ionic liquids have made significant advancement in the development of clean chemical processes in organic synthesis targeted to avoid or at least minimize the use of toxic or waste-generating reagents or solvents. The potential of ionic liquid (IL) as green solvent has already been established in several chemical transformations (3,4). The dual role of Brønsted acidic ionic liquid [HmIm]BF₄ as green solvent and catalyst was proved in a variety of organic chemical reactions (5–7).

Nitrogen heterocycles are of special interest because they constitute an important class of natural and non-natural products, many of which exhibit useful biological activities. Investigation of the oxazine or pyrido oxazine heterocycles has shown that they possess varied biological properties such as analgesic, anticonvulsant,

antitubercular, antibacterial, and anticancer activity (8–11). Particular attention has been paid to these compounds since the discovery of the non-nucleoside reverse transcriptase inhibitor trifluoromethyl-1,4-oxazine-2-one, which shows high activity against a variety of HIV-1 mutant strains (12). The fluorescent properties of 1,4-oxazinones are valuable as laser dyes (13–15) and coupling agent for oxidative hair dyes (16,17). In addition, pyrido 1,4-oxazine derivatives have exhibited therapeutic potential for the treatment of Parkinson's disease (18–22). However, very few methods are reported for the synthesis of pyrido-1,4-oxazine derivatives (23–28); isoxazole amines are not utilized for the synthesis of the title compounds. Recently, Anvar Mirzaei reported one-pot synthesis of pyrido-1,4-oxazin derivatives from 2-amino-3-hydroxypyridine and activated acetylenic compound under MeOH solvent (29). However, these reported methods suffer from many disadvantages such as low yields, multistep synthesis, hazardous organic solvents, long reaction time, harsh reaction conditions, difficulties in work-up, and the use of stoichiometric amounts and/or relatively expensive reagents. These may not be the preferred choices in the view of the green chemistry.

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Hence, an efficient and mild synthesis is needed for contemporary chemical synthesis of pyrido-1,4-oxazines. In our continuing efforts to utilize ionic liquids as environmentally attractive media, for the synthesis and catalytic processes, we have some interest in the synthesis of different heterocycles carrying isoxazole moiety (30–32). As a sequel to our investigations on multi-component synthesis (33–36), as well as green synthesis, we, herein, report for the first time a simple and efficient one-pot synthesis of isoxazolyl pyrido-1,4-oxazinones from isoxazole amine, chloroacetyl chloride, and 2-chloro-3-hydroxypyridine via Smiles rearrangement promoted by the Brønsted acidic IL, 1-methyl imidazolium tetrafluoroborate [HmIm]BF₄, as a solvent and catalyst, under mild conditions. In view of this, our method has a merit, whereby the title compounds are being synthesized in a one-pot green method under mild conditions at ambient temperature in excellent isolated yields in short reaction time.

Results and discussion

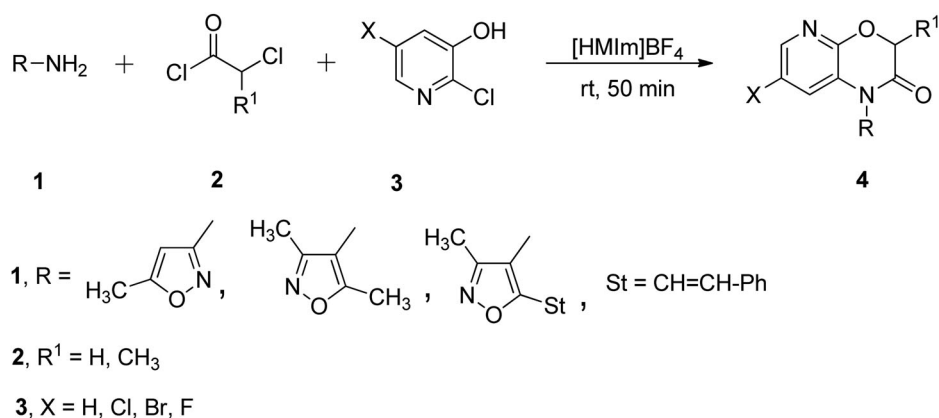
The concept of our process, illustrated in Scheme 1, is based on using simple starting materials, such as different isoxazole amines (**1**), substituted 2-chloroacetyl chloride (**2**) and 2-chloro-3-hydroxy pyridine (**3**), to form pyrido [2,3-*b*][1,4]oxazinone ring system (**4**) at ambient temperature. The commercial availability of starting materials makes this approach sufficient diversity oriented, thus fulfilling the recent demand for the generation of large combinatorial chemical libraries. Moreover, this novel one-pot three-step reaction provides a quick and efficient entry to functionalized pyrido[2,3-*b*][1,4]oxazinone derivatives of biological interest.

We first examined the reaction of 3-amino-5-methylisoxazole **1a**, with chloroacetyl chloride **2a**, and 2-chloro-3-hydroxy pyridine **3a**, in IL [HmIm]BF₄ at ambient

temperature for 50 min to afford the isoxazolyl pyrido [2,3-*b*][1,4]oxazinone **4a** in 95% yield (Table 1, entry 1). This result encouraged us to explore the reactivity of other 2-chloro-3-hydroxypyridine substrates **3b–3d** with 3-amino-5-methylisoxazole **1a**, independently in the presence of different chloroacetyl chloride **2a** and **2b** in IL [HmIm]BF₄ at ambient temperature to afford the corresponding isoxazolyl pyrido[2,3-*b*][1,4]oxazinones **4b–4g** in good yields (Table 1, Entry 2, 3–7).

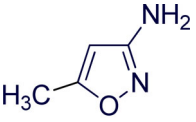
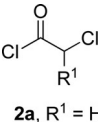
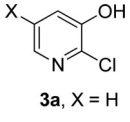
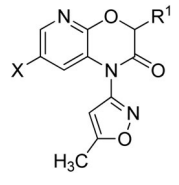
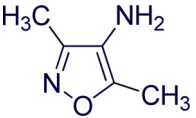
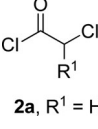
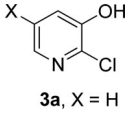
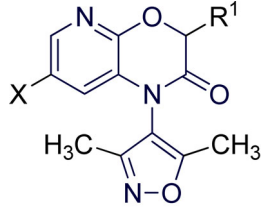
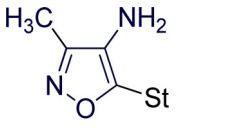
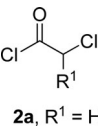
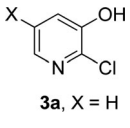
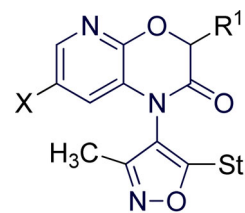
After this success, a few other isoxazole amines – viz., 4-amino-3,5-dimethylisoxazole **1b** and 4-amino-3-methyl-5-styrylisoxazole **1c** – were also treated with different chloroacetyl chloride **2a** and **2b**, and substituted 2-chloro-3-hydroxy pyridine **3a–3d** in the presence of IL [HmIm]BF₄ at ambient temperature and it was found that the transformation proceeded well to give the desired products **4h** to **4u** in good yields (entry 8–21). The structures of the products **4a–u** were confirmed on the basis of IR, ¹H NMR, ¹³C NMR, and MS spectral data. All the products **4a–u** reported are new.

To determine whether the ionic liquid [HmIm]BF₄ was an essential factor to realize the conversion of this reaction, the same reaction was carried out in DMF, Toluene, CH₃CN and methanol in the absence of ionic liquid; the results are ineffective. To know the influence of Brønsted acidic IL [HmIm]BF₄ in this reaction, the reaction was also carried out with other ionic liquids such as [bmIm]BF₄, [bmIm]Br, and [bmIm]OH. In these reactions, the reaction is sluggish and the conversion only reached 45–60%, and the reaction required more time (1.5–4 h) (Table 2). It is noteworthy that [HmIm]BF₄ is highly influencing the reaction by acting as Brønsted acid catalyst as well as effective solvent media for the synthesis of title compounds. Having established that, the best solvent and catalyst is [HmIm]BF₄ for this transformation; another advantage is, it can be easily recovered after the completion of



Scheme 1. One-pot synthesis of isoxazolyl pyrido[2,3-*b*][1,4]oxazinones.

Table 1. One-pot synthesis of isoxazolyl pyrido[2,3-*b*][1,4]oxazin-2(3*H*)-ones via Smiles rearrangement^a.

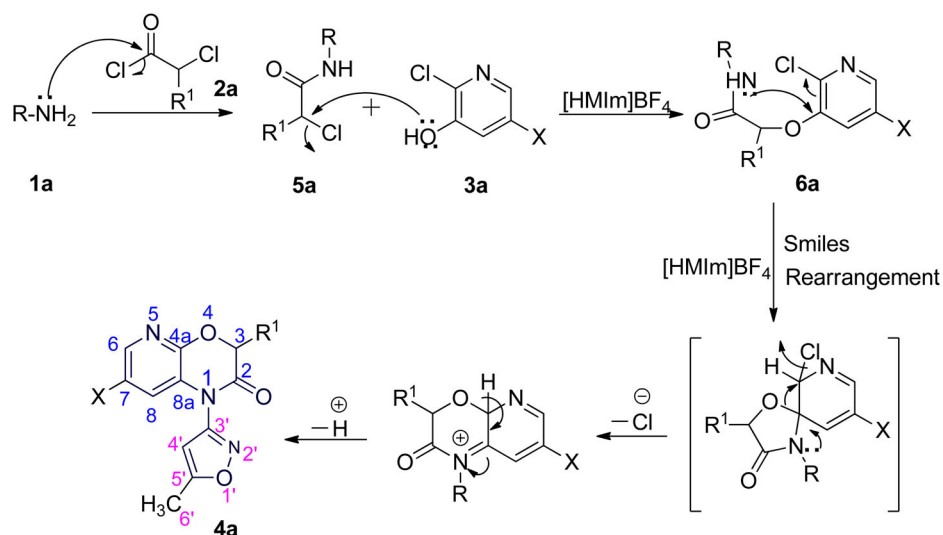
| Entry | Amine | Chloroacetyl chloride | Pyridine | Product ^b | Time (min) | Yield ^c (%) |
|-------|---|---|--|---|------------|------------------------|
| 1 |  1a |  2a , R ¹ = H |  3a , X = H |  | 50 | 95 |
| 2 | 1a | 2a , R ¹ = H | 3b , X = Cl | 4b , X = Cl; R ¹ = H | 45 | 90 |
| 3 | 1a | 2b , R ¹ = CH ₃ | 3b , X = Cl | 4c , X = Cl; R ¹ = CH ₃ | 50 | 92 |
| 4 | 1a | 2a , R ¹ = H | 3c , X = Br | 4d , X = Br; R ¹ = H | 40 | 91 |
| 5 | 1a | 2b , R ¹ = CH ₃ | 3c , X = Br | 4e , X = Br; R ¹ = CH ₃ | 50 | 93 |
| 6 | 1a | 2a , R ¹ = H | 3d , X = F | 4f , X = F; R ¹ = H | 45 | 89 |
| 7 | 1a | 2b , R ¹ = CH ₃ | 3d , X = F | 4g , X = F; R ¹ = CH ₃ | 50 | 92 |
| 8 |  1b |  2a , R ¹ = H |  3a , X = H |  4h , X = H; R ¹ = H | 48 | 90 |
| 9 | 1b | 2a , R ¹ = H | 3b , X = Cl | 4i , X = Cl; R ¹ = H | 46 | 89 |
| 10 | 1b | 2b , R ¹ = CH ₃ | 3b , X = Cl | 4j , X = Cl; R ¹ = CH ₃ | 40 | 91 |
| 11 | 1b | 2a , R ¹ = H | 3c , X = Br | 4k , X = Br; R ¹ = H | 45 | 86 |
| 12 | 1b | 2b , R ¹ = CH ₃ | 3c , X = Br | 4l , X = Br; R ¹ = CH ₃ | 50 | 90 |
| 13 | 1b | 2a , R ¹ = H | 3d , X = F | 4m , X = F; R ¹ = H | 44 | 92 |
| 14 | 1b | 2b , R ¹ = CH ₃ | 3d , X = F | 4n , X = F; R ¹ = CH ₃ | 50 | 88 |
| 15 |  1c St = CH=CH-Ph |  2a , R ¹ = H |  3a , X = H |  4o , X = H; R ¹ = H | 40 | 91 |
| 16 | 1c | 2a , R ¹ = H | 3b , X = Cl | 4p , X = Cl; R ¹ = H | 50 | 90 |
| 17 | 1c | 2b , R ¹ = CH ₃ | 3b , X = Cl | 4q , X = Cl; R ¹ = CH ₃ | 45 | 92 |
| 18 | 1c | 2a , R ¹ = H | 3c , X = Br | 4r , X = Br; R ¹ = H | 47 | 89 |
| 19 | 1c | 2b , R ¹ = CH ₃ | 3c , X = Br | 4s , X = Br; R ¹ = CH ₃ | 45 | 90 |
| 20 | 1c | 2a , R ¹ = H | 3d , X = F | 4t , X = F; R ¹ = H | 50 | 88 |
| 21 | 1c | 2b , R ¹ = CH ₃ | 3d , X = F | 4u , X = F; R ¹ = CH ₃ | 50 | 91 |

^aReaction conditions: Isoxazole amine (1 mmol), chloroacetyl chloride (1 mmol), 2-chloro-3-hydroxypyridine (1 mmol), IL (10 mL).^bThe products were characterized by ¹H NMR, ¹³C NMR, and MS spectra.^cIsolated yields.**Table 2.** Optimization of reaction conditions by using different ILs.

| Entry | IL | Time (h) | Yield (%) |
|-------|-----------------------|----------|-----------|
| 1 | [HmIm]BF ₄ | 0.5 | 95 |
| 2 | [bmIm]OH | 2.5 | 55 |
| 3 | [bmIm]Br | 4.0 | 45 |
| 4 | [bmIm]BF ₄ | 1.5 | 60 |

Table 3. Recycling of IL [HmIm]BF₄.

| Run-IL recycled (in ml) | Yield (%) |
|-------------------------|-----------|
| 1–5 | 95 |
| 2–4.7 | 94 |
| 3–4.4 | 94 |
| 4–4.1 | 92 |
| 5–4.0 | 90 |



Scheme 2. Plausible mechanism for the synthesis of isoxazolyl pyrido[2,3-*b*][1,4]oxazinones.

the reaction and can be reused in subsequent runs (five runs) without much loss of efficiency and with negligible loss of the IL (Table 3).

A mechanistic rationalization for this reaction is explained by the cyclization of *O*-alkylated product by Smiles rearrangement. The spiro-intermediate was expected to be formed in the first step, and then it was rearranged with the expulsion of HCl producing the final compound **4** (Scheme 2).

In order to support the mechanism, the intermediates – namely, *N*-isoxazolyl chloroacetamide (**5a**), and *N*-isoxazolyl-2-(2-chloro-3-hydroxy-4-oxo-1,2,3,4-tetrahydropyridin-3-yl)acetamide (**6a**) – were isolated and it was shown that under the reaction conditions **6a** converted to the compound **4a**. The role of ionic liquid as Brønsted acid catalyst is well known, and in the mechanism it activates the oxygen of compound (**6a**) for readily displacement during the conversion of (**6a**) to (**4a**).

Conclusions

In conclusion, we have demonstrated a simple, convenient, efficient, and environmental friendly protocol for the synthesis of isoxazolyl pyrido[2,3-*b*][1,4]oxazinones by one-pot three-component reaction employing Brønsted acidic ionic liquid [HMIm]BF₄ as a novel solvent and catalyst at ambient temperature via Smiles rearrangement. The procedure offers several advantages such as mild and neutral conditions, excellent yield of products, operational simplicity, and minimum environmental impact, making the technology practical, easy to perform, and facile. In view of the potential activity of isoxazole, and pyrido[1,4]oxazinones, we predict that the newly synthesized isoxazolyl pyrido

[2,3-*b*][1,4]oxazinones may be drug candidates, and the activity data will be published elsewhere.

Experimental

Commercial-grade reagents were used as supplied. Reaction progress and purity of the compounds were checked by thin-layer chromatography (TLC) on the pre-coated silica gel F₂₅₄ plates from Merck, and visualization was done by exposing to the iodine vapor. All the melting points were determined on a Fischer-Johns apparatus and are uncorrected. IR spectra were recorded on KBr discs on a PerkinElmer Bx series FT-IR spectrophotometer. ¹H NMR spectra were recorded as a Varian Gemini 300 MHz spectrometer. ¹³C NMR spectra were recorded on a Bruker 75 MHz spectrometer. Chemical shift values are given in ppm (δ) with tetramethyl silane as internal standard. Mass spectral measurements were carried out by EI method on a Jeol JMC-300 spectrometer at 70 eV. Elemental analyses were determined by a PerkinElmer 240 CHN elemental analyzer.

The ILs [HMIm]BF₄, [bmIm]BF₄, [bmIm]Br, and [bmIm]OH were prepared according to the reported procedures (5,37–39).

General procedure for the synthesis of isoxazolyl pyrido[2,3-*b*][1,4]oxazin-2(3H)-ones (4a–u)

A mixture of isoxazole amine **1** (1.20 g, 12.24 mmol), chloroacetyl chloride **2** (1 mL, 12.24 mmol) and 2-chloro-3-hydroxypyridine **3** (1.58 g, 12.24 mmol) was stirred in the presence of [HMIm]BF₄ (10 mL) at room temperature for 50 min. The reaction was monitored by TLC. After completion of the reaction, the reaction

mixture was poured into ice-cold water and extracted with diethyl ether (3 × 15 mL). The combined organic layers were dried over anhydrous Na₂SO₄. The solvent was distilled off under reduced pressure and the crude reaction mass was purified by column chromatography (eluent: 15% EtOAc in hexane) to afford the final product.

Spectral data of synthesized compounds

1-(5-Methylisoxazol-3-yl)-1H-pyrido[2,3-b][1,4]oxazin-2 (3H)-one (4a).

Yield: 95%, mp 110–112°C. IR (KBr): 3078, 2952, 1684, 1580, 1080, cm⁻¹, ¹H NMR (300 MHz, CDCl₃): δ 2.20 (s, 3H, isoxazole-CH₃), 4.50 (s, 2H, OCH₂), 6.70 (s, 1H, isoxazole-H), 7.06 (dd, 1H, H₇, J = 8.0, 4.6 Hz), 7.42 (dd, 1H, H₈, J = 8.0, 1.5 Hz), 8.02 (dd, 1H, H₆, J = 4.8, 1.2 Hz); ¹³C NMR (75 MHz, CDCl₃): δ 10.24 (C-6'), 80.62 (C-3), 116.70 (C-4'), 121.78 (C-8a), 126.68 (C-7), 138.50 (C-8), 141.40 (C-6), 152.20 (C-4a), 156.40 (C-3'), 164.30 (C-5'), 168.42 (C-2). EI-MS m/z: 232 (M + H)⁺. Anal. Calcd for C₁₁H₉N₃O₃: C, 57.14; H, 3.92; N, 18.17%. Found: C, 57.10; H, 3.95; N, 18.15%.

Disclosure statement

No potential conflict of interest was reported by the authors.

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Notes on contributors

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Mr. Pittala Praveen Kumar obtained his M.Sc. degree in Organic Chemistry from Osmania University (INDIA). He is currently a Ph.D. student under the supervision of Dr. M. Nagi Reddy at CSIR-IICT Hyderabad, India. His research focuses on the development of new isoxazole derivatives and their biological activity.

Prof. Eligeti Rajanarendar received his Ph.D. degree in Organic Chemistry from Kakatiya University, India and then joined as a professor at Department of Chemistry Kakatiya University, Warangal, India. His research interests synthetic methodology and their applications in the expeditious syntheses of organic functional materials.

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