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Selectfluor-mediated regioselective nucleophilic functionalization of N-heterocycles under metal- and base-free conditions†

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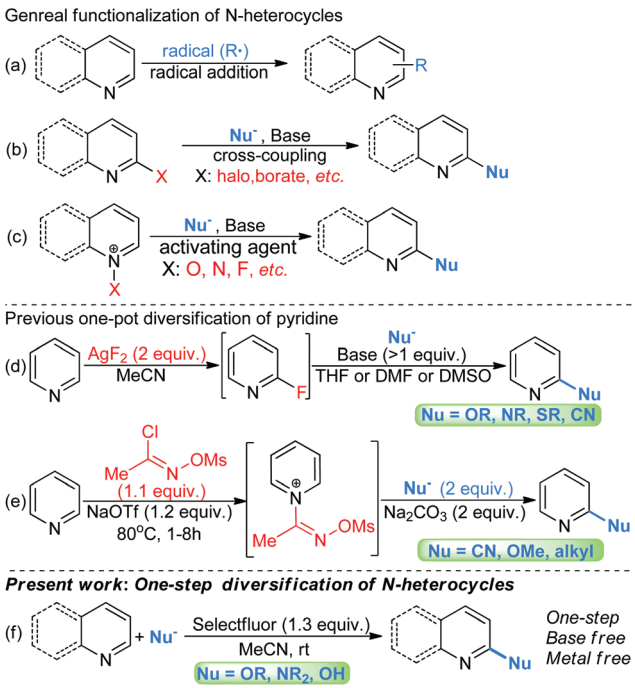
A practical and environmentally attractive methodology for the direct diversification of N-heterocycles at ambient temperature under open-air conditions was developed. The obvious advantage of the process is that no toxic reagent, transition metal, base or other additive is employed, thus greatly reducing costs, facilitating post-reaction neutralization and purification and minimizing the environmental impact.

Introduction

The development of an eco-friendly chemical process to produce diverse value-added chemicals from the nature-abundant and low-cost raw material is a particularly attractive concept, but represents a considerable challenge in green organic synthesis. N-Heterocycles and their derivatives are the extremely important motifs in bioactive natural products and synthetic pharmaceuticals.[1] It is well-known that the heterocycle core and its modified functional group(s) cooperatively affected the activity of the molecule, not just the heterocycle itself. Thus, the research of structure–function relationships requires the heteroaromatic derivatives containing a set of distinct functional group(s) attached to heterocycle cores. Generally, structural modification of N-heterocycles can be divided into two categories according to the reaction mechanism: (1) Minisci-type radical substitution (Scheme 1a),[2] and (2) the nucleophilic reaction of pre-functionalized N-heterocycle substrates, including substituted N-heterocycles (Scheme 1b)[3] and electrophilically activated N-heterocycles (Scheme 1c).[4] The major drawback in process (1) is that regioselectivity is usually difficult to control and regioisomeric mixtures were produced. In particular, the regioisomers of N-heteroaromatic molecules often have contrasting indications or even opposing biological activities. The approach (2) presents the following disadvantages:[5] (a) the requirement of a directing group significantly limits their applications, especially for the late-stage structural modification, because the installation and removal of such groups require additional steps; and (b) additional synthesis procedure and isolation steps of N-activated heterocycles are often unavoidable.



Scheme 1 Direct preparation of functionalized N-heterocycles.

Although various functional groups can be introduced in N-heterocycles, most of the functionalization processes incorporate only one type of functional group. To the best of our knowledge, only two examples of diverse nucleophilic

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functionalization of N-heterocycles have been reported. In 2014, Hartwig and colleagues have pioneered the direct structural modification of pyridines *via* AgF_2-mediated sequential C–H fluorination and nucleophilic substitution (Scheme 1d).[6] Very recently, Fier has designed and applied *N*-((methylsulfonyl)oxy)acetimidoyl chlorides to achieve diverse functionalization of pyridines (Scheme 1e).[7] Although these methods exhibited highly advantageous features, from an eco-friendly and practical point of view,[8] both approaches require two-step operation procedures and superstoichiometric amounts of bases to facilitate the reaction,[9] which leads to not only high cost but also environmental problems. In addition, the difficulties to remove the trace transition-metal contamination from the final products, particularly for the late-stage modification of medicine, restrict their applications. It is therefore highly desired to develop an efficient, practical and eco-friendly process that can achieve direct diversification of N-heterocycles employing abundant and readily available materials under metal- and base-free conditions.

We were aware that *N*-fluoropyridiniums\quinoliniums were employed as the synthetic precursors for the functionalization of N-heteroaromatic substrates, given their direct C–H bond activation at the 2-position.[10] For example, Strekowsli has reported one-pot sequential *N*-fluorination and alkoxylation of quinoline at ultra-low temperature by using F_2 as a fluorinating reagent.[11] However, the high toxicity, explosivity and corrosion of fluorine gas severely restrict the strategy for general applications. In light of the unique pharmacological activities of diverse heteroaromatic derivatives and also in continuation of our efforts in green chemistry,[12] herein, we report a direct synthesis of diverse heteroaromatic derivatives through Selectfluor-mediated regioselective nucleophilic functionalization of N-heterocycles under metal- and base-free and open air conditions (Scheme 1f).

Results and discussion

To test the possibility of our envisioned process, we chose commercially available Selectfluor as a potential fluorinating reagent due to its air and moisture stability yet is an extraordinary powerful donor of an electron-deficient fluorine atom to a wide number of nucleophiles. To our delight, the desired 2-(1*H*-benzo[*d*][1,2,3]triazol-1-yl)quinoline (**3aa**) was obtained in 82% NMR yield by using quinoline (**1a**) as the model substrate and 1*H*-benzotriazole (**2a**) as the nucleophilic reagent in MeCN at ambient temperature for 7 h (entry 1). As anticipated, the property of the fluorinating reagent played a vital role in the transformation outcome, and other related fluorides did not promote C–H functionalization (entries 2–6). Significantly, the employment of a slight excess of Selectfluor (1.3 equiv.) was found to provide the desirable **3aa** in excellent yield (entries 7 and 8). In screening different solvents, the reaction in $MeNO_2$ gave the markedly inferior yield of **3a** (entry 9); no desired products were obtained with THF, DCE, DMF, DMSO or acetone (entries 10–13). Conducting the reaction at a quinoline concentration of 0.1 M did not affect the reaction yield; however, the higher concentration of 0.25 M (entry 14) was selected to provide conditions that minimize solvent waste and shorten the reaction time (5 h). Elevated reaction temperature did not improve the reaction outcome (entry 15) (Table 1).

Using the optimal reaction conditions, the scope of the transformation was explored with respect to both N-heterocycles and nucleophiles. To our satisfaction, the quinolines containing steric-hindered, electron-donating and electron-withdrawing substitute groups all underwent coupling reactions smoothly at the 2-position (**3aa–3ja**). It is noteworthy that the halo-substituents (–Cl, Br and F) on the quinoline ring of the substrates were well tolerated (**3da**, **3ea** and **3ja**), providing a handle for further transformations. The current method is also applicable for the efficient synthesis of highly functionalized isoquinoline (**3ka**), pyridine (**3la–3ua**), pyrrole (**3va**) and *N*-methylpyrrole (**3wa**). However, when 3-bromopyridine, 4-chloropyridine and 4-methoxycarbonylpyridine were used as substrates, only a trace amount of the desired product could be detected. In addition, a range of nitrogen-containing heterocycle coupling partners are tolerated, including 1*H*-benzo[*d*]imidazole (**3ab**), various substituted 1*H*-benzotriazole (**3ac–3af**), pyrazole (**3ag–3ah**), triazole (**3ai–3aj**) and 1*H*-tetrazole (**3ak**). Encouraged by the above results, we set out to investigate C–H alkoxylation and hydroxylation. The alcohols with diverse chain lengths and isomeric structures did not affect the outcome of the transformation (**4aa–4ae**). Neither the steric hindrance (**4bb**) nor the electronic effect (**4bc–4bh**) of quinolone rings significantly influenced the reaction. Moreover, 58–67% conversion of quinolines **1** and near quanti-

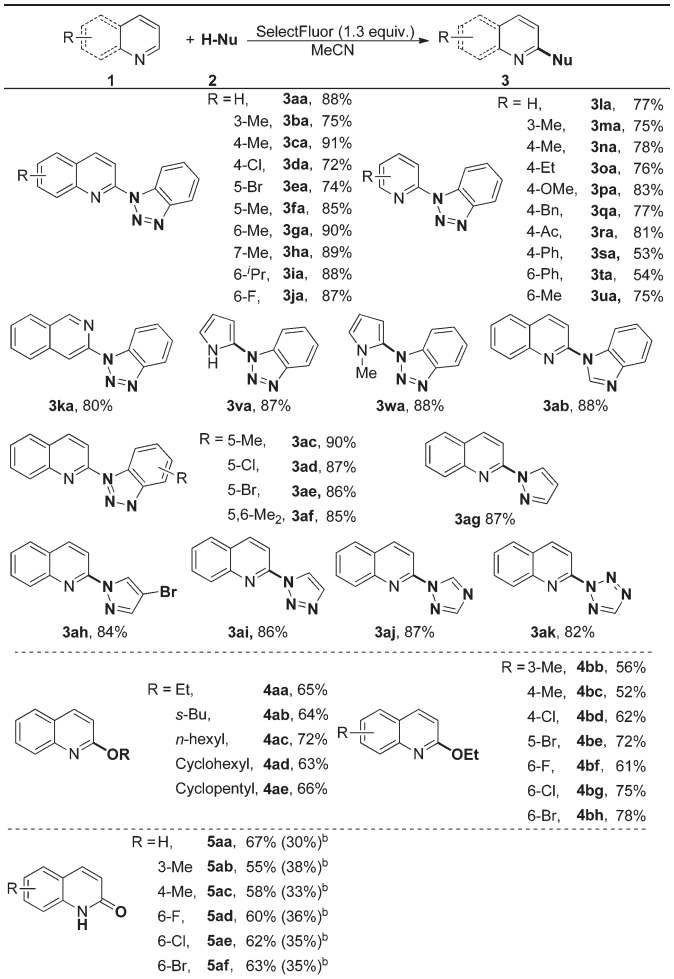
Table 1 Optimization of reaction conditions[a]

1a 2a 3aa

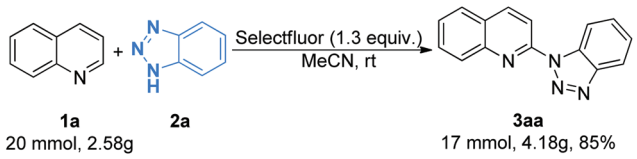
Entry	[F]	Solvent	Temp.	Yield[b]
1	Selectfluor (1 equiv.)	MeCN	r.t.	82
2	NFSI (1 equiv.)	MeCN	r.t.	N.R.
3	DAST (1 equiv.)	MeCN	r.t.	N.R.
4	BAST (1 equiv.)	MeCN	r.t.	N.R.
5	TBAF (1 equiv.)	MeCN	r.t.	N.R.
6	$Et_3N{\cdot}HF$ (1 equiv.)	MeCN	r.t.	N.R.
7	Selectfluor (1.3 equiv.)	MeCN	r.t.	93
8	Selectfluor (1.5 equiv.)	MeCN	r.t.	93
9	Selectfluor (1.3 equiv.)	$MeNO_2$	r.t.	85
10	Selectfluor (1.3 equiv.)	THF	r.t.	N.R.
11	Selectfluor (1.3 equiv.)	DCE	r.t.	N.R.
12	Selectfluor (1.3 equiv.)	DMF	r.t.	Trace
13	Selectfluor (1.3 equiv.)	Acetone	r.t.	Trace
14[c]	Selectfluor (1.3 equiv.)	MeCN	r.t.	92%
15	Selectfluor (1.3 equiv.)	MeCN	60 °C	92%

[a] Unless otherwise specified, the reactions were carried out in a vial in the presence of **1a** (0.1 mmol), **2a** (0.15 mmol), a fluoride reagent and a solvent (1 ml). [b] Estimated by ^1H NMR using diethyl phthalate as an internal reference. [c] Molar concentration: 0.25 mmol ml^{-1}.

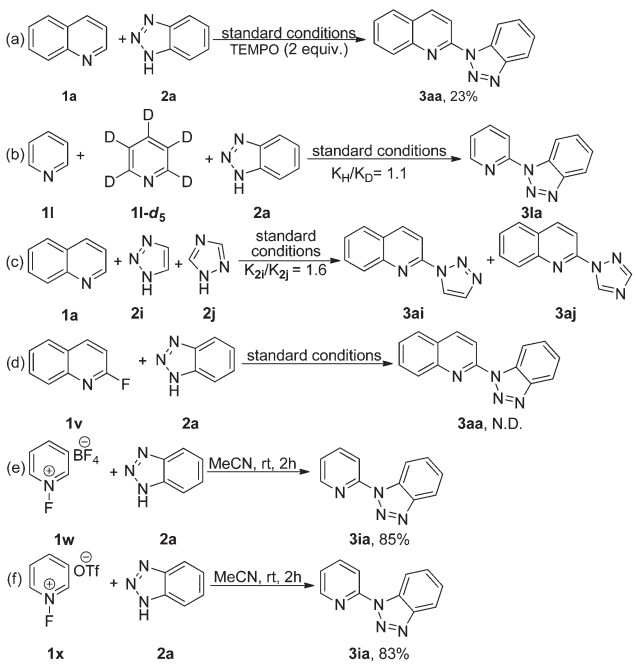
Table 2 Reaction scope[a]



[a] All reactions were carried out in a vial in the presence of **1** (0.5 mmol), **2** (0.75 mmol), Selectfluor (0.65 mmol) and MeCN (2 ml); aminations were performed at room temperature. Alkoxylation and hydroxylation were performed at 60 °C; isolated yields are reported. [b] The percentage of quinoline **1** was recovered.



Scheme 2 Gram-scale synthesis of **3aa**.



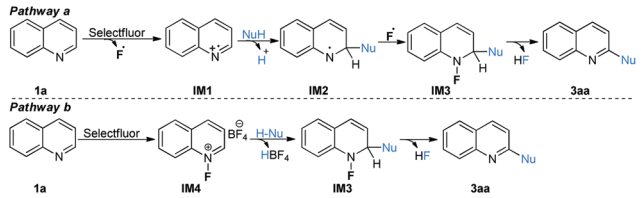
Scheme 3 Control experiments.

tative selectivity for quinolin-2(1*H*)-ones could be achieved using water as the nucleophile (**5aa–5af**). However, no reaction occurred when aniline, benzamide, morpholine, sodium cyanide or 1-hexanethiol was used as the nucleophile (Table 2).

The practicability of this transformation was explored by employing **1a** (20 mmol, 2.58 g) with **2a** under the standard reaction conditions (Scheme 2). As anticipated, the desired **3aa** was formed in 85% yield, which suggested that the methodology is a practical process for the preparation of various functionalized N-heterocycles.

To gain some insights into the mechanism, several controlled experiments were conducted (Scheme 3). When the reaction was performed in the presence of 2 equiv. of 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) as an additive under standard reaction conditions, **3aa** was formed in 23% NMR yield (Scheme 3a). The poor yield of **3aa** suggested that the radical process was likely the main reaction pathway. Secondly, the intermolecular kinetic isotope effect (KIE) was studied with respect to the heterocyclic C(sp_2)–H/D bonds. A clear KIE value of 1.1 was obtained in a 1 : 1 mixture of pyridine (**1l**) and pyridine-d5 (**1l-d5**) (Scheme 3b). Besides this, K_{2i}/K_{2j} was determined to be around 1.6 when a mixture of 1*H*-1,2,3-triazole (**2i**) and 1*H*-1,2,4-triazole (**2j**) was subjected to the standard conditions (Scheme 3c). Taken together, these observations revealed that the scission of the heterocyclic C(sp_2)–H bond might not be in the rate-limiting step and the nucleophilic attack on heterocycles would be rate-limiting in the overall process. Considering that this category of cross-coupling reactions may involve the haloquinoline intermediate, *in situ* generated from the halogenation of quinoline, we attempted to treat 2-fluoroquinoline **1v** with **2a** under the standard reaction conditions. However, no **3aa** was observed (Scheme 3d). The treatment of 1-fluoropyridinium tetrafluoroborate (**1w**) or 1-fluoropyridinium triflate (**1x**) with **2a** in MeCN at ambient temperature gave the target product **3aa** in good yields (Scheme 3e and f). Taken together, these results suggest that *N*-fluoroquinoliniums might be the reaction intermediate.

Although various mechanistic details of these reactions are not fully understood, two tentative mechanisms for the reac-



Scheme 4 Possible mechanisms.

tion are depicted in Scheme 4 according to the above mentioned observations and previous literature reports. Initially, quinoline **1a** is easily oxidized by Selectfluor *via* a single-electron-transfer process, producing the radical cation **IM1**[13] along with a fluorine radical (Scheme 4a). Then the nucleophile first attacks the α-carbon of the intermediate **IM1** to give the intermediate **IM2**.[4a,c] Finally, the fluorine radical adds to the nitrogen atom of **IM2** to form the intermediate **IM3**, which undergoes deprotonation/aromatization to deliver the desired product **3aa**. Alternatively, another process with *N*-fluoroquinoliniums as the reactive intermediate could not be excluded (Scheme 4b).[11] The coupling reaction may follow a mechanism similar to that of the F_2-mediated alkoxylation of quinoline with EtOH as the nucleophile reported by Strekowsli, with the major difference being Selectfluor as the fluorinating reagent instead of hazardous F_2. First, quinoline **1a** is activated by Selectfluor to form the active *N*-fluoroquinolinium intermediate **IM4**. Subsequently, **IM4** is attacked by the nucleophile to generate the intermediate **IM3**.

Conclusions

This work for the first time provides a facile, practical and environment-friendly methodology for the one-step diversification of N-heterocycles. This protocol occurs to provide N-heteroaryl- and alkoxy-substituted heterocycles as well as quinolin-2(1*H*)-ones without any transition-metal or base, which is inaccessible through conventional methods. Noteworthy advantages of the present reaction are as follows: (a) the abundance and accessibility of inexpensive N-heterocycles, nucleophilic reagents and Selectfluor; (b) operational simplicity and mild reaction conditions with good functional-group tolerance; (c) a broad range of N-heterocycle substrates: quinoline, isoquinoline, and pyridine as well as pyrrole; and (d) ease of scale-up. Preliminary mechanistic investigation suggested that a radical process and a nucleophilic attack pathway may be involved in the direct C–H functionalization reaction.

Conflicts of interest

There are no conflicts to declare.

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