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Convenient entry to *N*-pyridinylureas with pharmaceutically privileged oxadiazole substituents *via* the acid-catalyzed C—H activation of *N*-oxides

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ABSTRACT

Pyridine-*N*-oxides bearing a pharmacophoric oxadiazole moiety could be C—H functionalized *via* the acid-catalyzed reaction with dialkylcyanamides, providing access to hitherto undescribed pyridine-2-yl substituted ureas, which have potential as "lead-like" scaffolds for medicinal chemistry. Atom-economy, environmental friendliness (no halide-containing or toxic reagents), simple work-up, as well as scalability are the main advantages of the employed procedure.

Introduction

The capability of *N*-pyridinylureas to participate in hydrogen bonding or anion binding with target enzymes makes them an *engaging* pharmacophoric motif for drug discovery. This moiety is incorporated into small molecules exhibiting various biological activities, which are associated with therapeutic areas such as oncology [1–7], age-related degenerative diseases [8–10], and metabolic disorders including type II diabetes [11–15]. Moreover *N*-pyridinylureas were recognized as antischistosomal [16] and antibacterial [17,18] agents, as well as inhibitors of fatty acid amide hydrolase (FAAH) – the validated target for the treatment of a number of pathologies, including chronic pain, inflammation, psychoses, depression, nausea, and vomiting [19–23].

Disappointingly, the most straightforward route to ureas involves the reaction of amines with toxic reagents such as phosgene [24] or isocyanates [25], that leads to large amounts of harmful halide-containing wastes. As a "greener" alternative, Prokhorov and co-workers described the acid-catalyzed reaction of 4-arylpyrimidine-1-oxides with cyanamide, which resulted in the formation of the respective ureas [26]. Later Rassadin and co-workers found that the same process occurs between 2-methylpyridine-*N*-oxide and dimethylcyanimide [27,28]. However, only *N*-oxides bearing a simple substituent (Me, MeO, halogen, NO_2, CN, COOMe) on the pyridine ring were investigated, whereas medicinal chemistry is concerned with more complicated molecules, particularly with a different heterocyclic periphery. The 1,2,4-oxadiazole ring represents one such privileged motif, as reflected in numerous published medicinal chemistry studies [29–34] and newly-marketed drugs (Translarna® [35], Edarbi® [36]). 1,3,4-Oxadiazoles also have broad pharmaceutical applications [37–39], and in some ADMET parameters, is even more attractive than the 1,2,4-isomer [40,41]. In light of this, we studied the acid-catalyzed C—H functionalization of pyridine-*N*-oxides bearing 1,2,4- and 1,3,4-oxadiazole substituents by dialkylcyanamides.

Results and discussion

Because the synthesis of the required oxadiazole-substituted pyridine-*N*-oxides was not described, we first had to develop an appropriate procedure for their preparation. Since strong acylation (acyl chlorides, anhydrides) or dehydration (phosphorus oxychloride, thionyl chloride) reagents, which are commonly used for oxadiazole ring formation [42–44], can react with highly reactive *N*-oxides [45–47], we decide to initially assemble the oxadiazole

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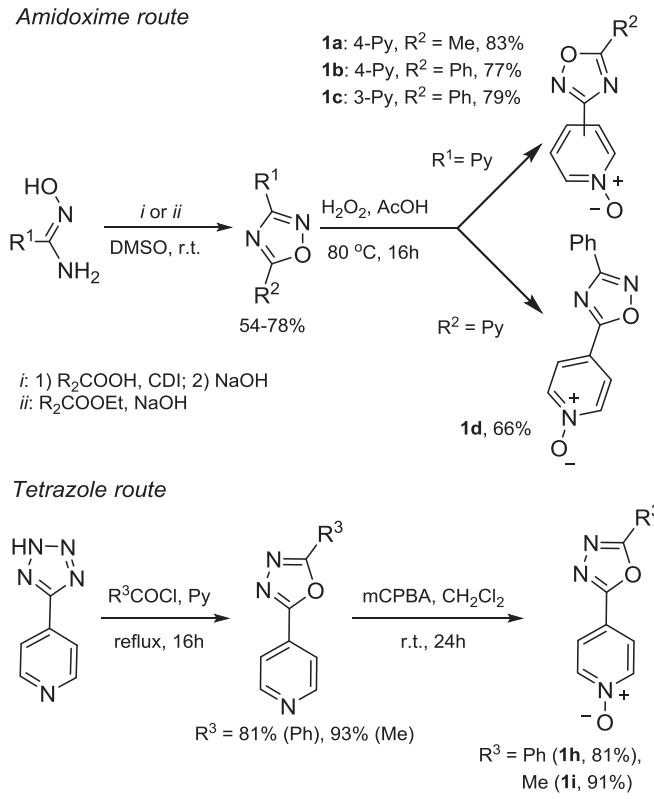
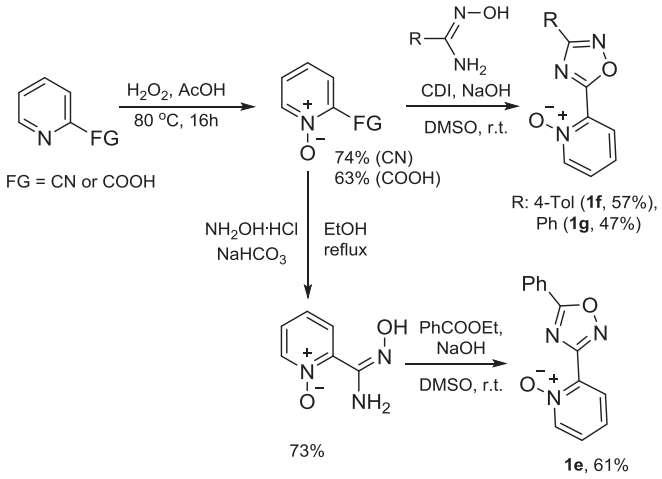


Scheme 1. Synthesis of the starting pyridine-*N*-oxides.

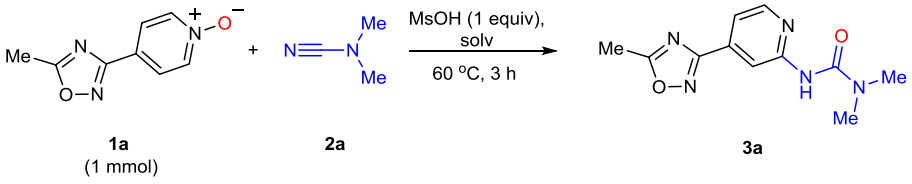


Scheme 2. Synthesis of the 2-substituted pyridine-*N*-oxides.

core from pyridine containing precursors followed by *N*-oxidation. The *amidoxime* [48,49] and *tetrazole* [50] routes were chosen for the preparation of the 1,2,4-oxadiazole and 1,3,4-oxadiazole series, respectively (Scheme 1).

Most of the 1,2,4-oxadiazole bearing pyridines were successfully converted into the corresponding *N*-oxides using H_2O_2 in AcOH according to the literature method [28], while 1,3,4-oxadiazoles decomposed under these conditions. Therefore, *m*-CPBA in CH_2Cl_2 was utilised for the *N*-oxidation of the 1,3,4-oxadiazole series. The synthesis of pyridine-*N*-oxides containing a 1,2,4-oxadiazole moiety in the 2-position was the most difficult task. These

Table 1
Optimization of the reaction conditions.



1a (1 mmol) **2a** **3a**

Entry	**2a** (equiv.)	Solvent (20 equiv.)	Conversion of **1a**[a], % (isolated yield of **3a**, %)
1	1.5	–	21
2	10.0	–	98 (68)
3	1.5	MeCN	66
4	1.5	MeCN	56[b]
5	1.5	acetone	53
6	1.5	DMF	13
7	1.5	EtOH	35
8	1.5	AcOH	24
9	1.5	MeCN	98 (49)[c]
10	1.5	MeCN	91 (54)[d]
11	2.0	MeCN	80
12	3.0	MeCN	83
13	**2.0**	**MeCN**	**98 (76)**[e]
14	2.0	MeCN	98 (59)[f]
15	2.0	MeCN	85[g]

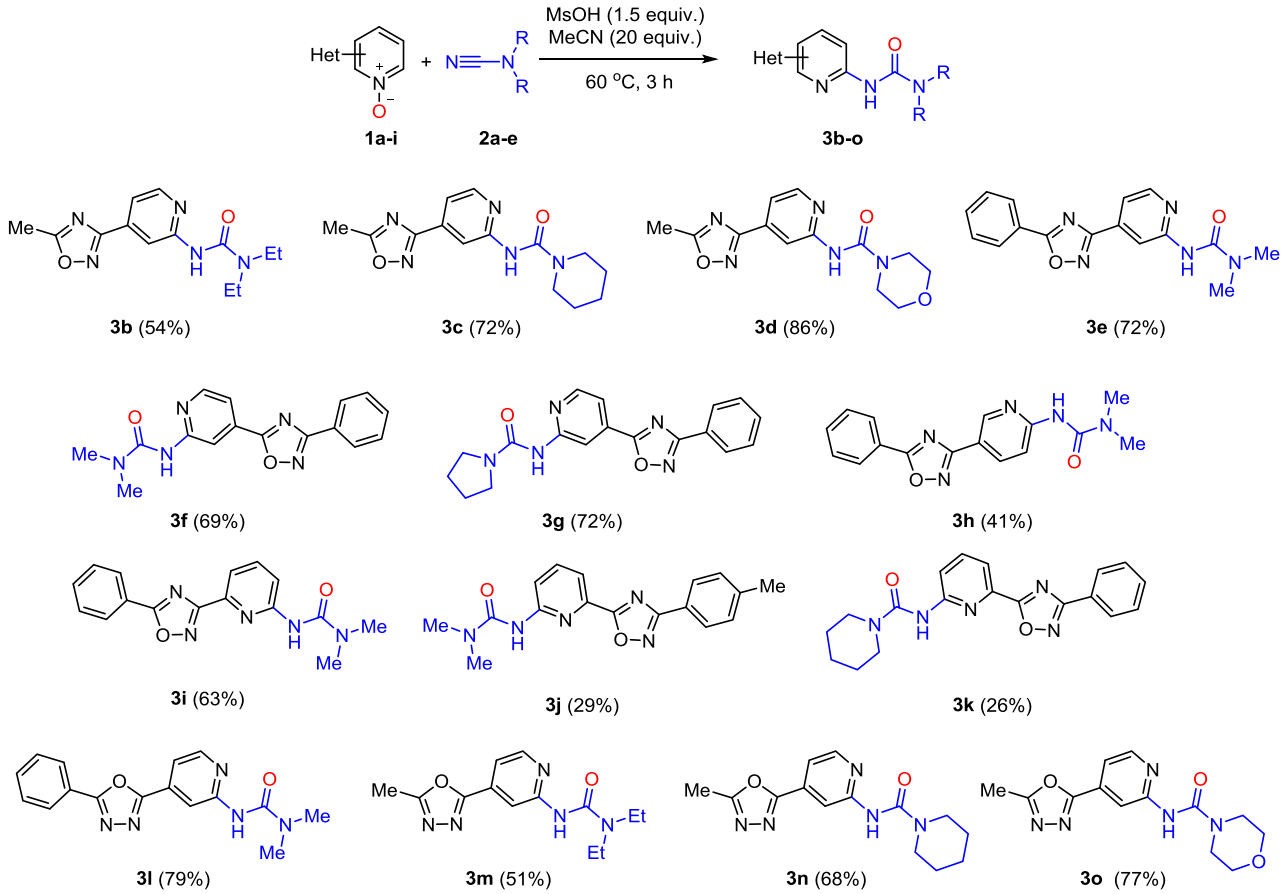
[a] Conversion of the *N*-oxide was estimated by ^{1}H NMR spectroscopy.
[b] 10 equiv. of MeCN was used.
[c] Reaction mixture was heated at 80 °C.
[d] Reaction time was 18 h.
[e] 1.5 equiv. of MsOH was used.
[f] The reaction was performed with 4.5 mmol of **1a** and 6.8 mmol of MsOH.
[g] Microwave heating was used.

compounds decomposed in H_2O_2/AcOH and were also not oxidized by *m*-CPBA in CH_2Cl_2. Therefore, the reaction sequence was altered and the *N*-oxidation was performed at the first stage. The subsequent oxadiazole core assembly was carried out under mild conditions according to our previous developed procedures (Scheme 2) [51–53].

Disappointedly, the acid-catalyzed C—H functionalization with dialkylcyanamides reported by Rassadin and co-workers [28] was not suitable in the case of oxadiazoles due to decomposition. Therefore, we had to develop a new methodology. Initial optimization showed that the reaction needed to be carried out in a solvent (Table 1, entries 1–8). Although complete conversion of the *N*-oxide and isolation of the corresponding urea **3a** was achieved in moderate yield (68%) using excess dimethylcyanamide (DMCA, Table 1, entry 2) as the solvent, this method is too wasteful and expensive for the practical use. MeCN was recognized as the most appropriate solvent for this reaction. However, due to moderate *N*-oxide conversion (66%), the optimization was continued by studying the effect of the temperature and the reaction time (Table 1, entries 9 and 10). Unfortunately, these efforts did not provide the desired result despite a significant increase in the conversion of the starting *N*-oxide. Increasing the temperature (up to 80 °C), as well as prolonging the reaction time, led to an increase in the impurities formed. According to our observations, the latter are formed due to 1,2,4-oxadiazole ring decomposition. Then we tried to vary the DMCA and MsOH amounts (Table 1, entries 11–13). An increase in the DMCA amount used to 2 equiv. led to an increased conversion (Table 1, entry 11), while the use of a larger excess had no effect (Table 1, entry 12). The reaction with 1.5 equiv. of MsOH resulted in full (98%) conversion of *N*-oxide **1a** and the desired urea **3a** was obtained in an improved isolated yield (76%) (Table 1, entry 13). The structure of product **3a** was established by NMR spectroscopy and HRMS. In addition to **3a** the *N*-oxide reduction product – 5-methyl-3-(pyridin-4-yl)-1,2,4-oxadiazole – was isolated (~10%) from the reaction mixture. We then studied the possibility of increasing the reaction scale under these conditions. An increase in the reagent amounts by 4.5 times (Table 1, entry 14) did not lead to a noticeable decrease in the product yield, which indicates the scalability of the proposed procedure.

Finally, we examined the possibility of using microwave heating and found that this does not noticeably affect the speed of the process (Table 1, entry 15).

Used these optimized conditions, we verified the scope of the reaction using a number of 1,2,4- and 1,3,4-oxadiazolyl-substituted *N*-oxides **1** as well as selected commercially available dialkylcyanamides **2** (Scheme 3). Firstly, we replaced DMCA **2a** with other dialkylcyanamides (diethylcyanamide **2b**, 1-piperidinecarbonitrile **2c**, 4-morpholinecarbonitrile **2d**) in the reaction with **1a**. In the case of diethylcyanamide the yield of product **3b** decreased, whereas ureas (**3c**,**d**) were obtained in good yields. Further, a number of structural variations related to the 1,2,4-oxadiazole ring, including replacement of the substituents at positions 3 and 5 of the heterocycle, were explored. There were no observed effects from the replacement of Me with Ph (**3e**), as well as the relocation of substituents (**3f**,**g**). In contrast, the change from 4-pyridine to 3-pyridine led to a lower yield (**3h**). However, the most intriguing results were obtained in cases of 2-pyridine substrates. 2-(5-Phenyl-1,2,4-oxadiazol-3-yl)pyridine 1-oxide **1e** reacted with cyanamide **2a** providing the desired urea **3i** in moderate yield (63%), whereas *N*-oxides **1f** and **1g**, bearing a 1,2,4-oxadi-



Scheme 3. Reaction scope with various *N*-oxides **1** and dialkylcyanamides **2**.

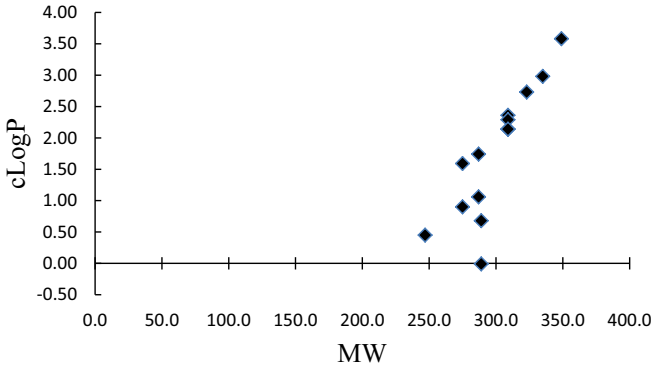


Fig. 1. Molecular weights and cLogP parameters of compounds **3a–o**. cLogP values were calculated using publicly available software (www.molinspiration.com).

azol-5-yl moiety, gave complex mixtures of three different products. These mixtures were separated by column chromatography and one of the components was isolated as an individual compound in each case and determined to be the desired urea (**3j** and **3k**) after comprehensive spectroscopic investigations. Unfortunately, the structures of the other components have not been determined to date, and to fill this gap is the goal for our future research.

Two 1,3,4-oxadiazole derivatives (**1h** and **1i**) were also tested in the reaction with different dialkylcyanamides, and they displayed the same reactivity as similar 1,2,4-oxadiazoles (Scheme 3). In all cases, the desired ureas **3l-o** were obtained in moderate to good isolated yields.

Since we regarded the obtained ureas as suitable scaffolds for medicinal chemistry, it was of interest to evaluate their compliance to "lead-like" criteria [54], i.e. low molecular weight (MW <300) and low lipophilicity characteristics (cLogP < 3). Indeed, although some of the synthesized compounds exceed the lead-likeness threshold of molecular weights, all of them have cLogP lower than 3 (Fig. 1). Thus, the structural motif described in this work could be utilized in drug optimization to increase water solubility.

Conclusion

We have reported the first example of using pyridine-*N*-oxides bearing 1,2,4- or 1,3,4-oxadiazole moieties as substrates for the acid-catalyzed reaction with dialkylcyanamides. This method represents an atom-economical, convenient, and simple route to previously undescribed *N*-hetaryl-substituted ureas, which predominantly conform to the criteria of lead-likeness and can be suitable starting points for medicinal chemistry optimization.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2019.151108.

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