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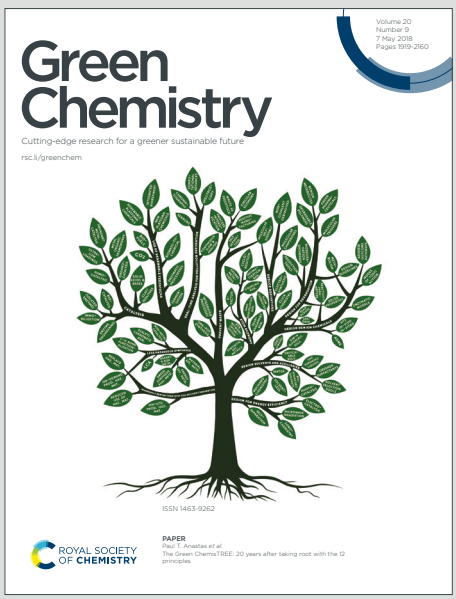

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DOI: 10.1039/C9GC01895F

COMMUNICATION

Intramolecular Electrochemical Dehydrogenative N–N Bond Formation for the Synthesis of 1,2,4-Triazolo[1,5-*a*]pyridines

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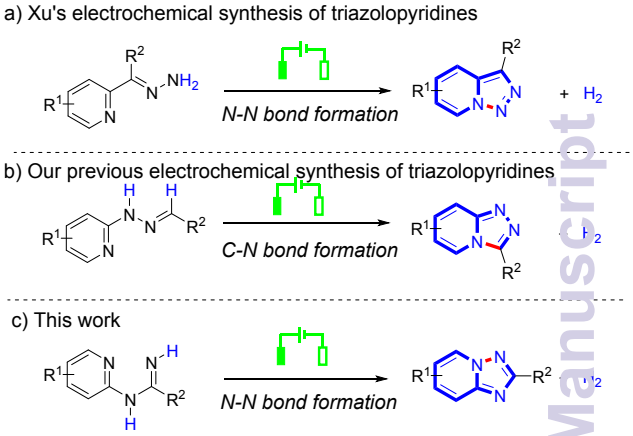
Received 00th January 20xx,
Accepted 00th January 20xx

DOI: 10.1039/x0xx00000x

A metal and oxidant-free intramolecular dehydrogenative N–N bond formation has been developed under mild and scalable electrolytic conditions. Various valuable 1,2,4-Triazolo[1,5-*a*]pyridines were synthesized efficiently from the readily available *N*-(2-pyridyl)amidines. The reactions were conducted in a simple undivided cell under constant current condition with $^{n}Bu_4NBr$ as both the redox mediator and electrolyte. This protocol was applied to the efficient synthesis of key intermediate for anti-diabetic compounds.

Introduction

Due to the special biological activities and electrical properties, nitrogen-containing heterocycles constitute one of the largest and most important groups of organic compounds.[1] Among these, the 1,2,4-triazolo[1,5-*a*]pyridine nucleus is a privileged structural motif widely found in biologically active natural products, pharmaceuticals, agrochemicals and organic materials (Figure 1).[2-8] The importance of this heterocyclic moiety has promoted the development of many practical synthetic routes for the preparation,
transformation and finding of specific properties of 1,2,4-triazolo[1,5-*a*]pyridine derivatives. In 2009, Ueda and Nagasawa reported a copper-catalyzed intermoleular synthetic approach to triazolo[1,5-*a*]pyridine by using 2-aminopyridines and nitriles as starting substrates,[9a] and the majority of synthetic routes rely on the intramolecular oxidative cyclization of aminopyridine derivatives (such as ***N*-Aryl amidines**, guanidines) at high temperature in the presence of either transition metal catalysts [9] or stoichiometric amounts of external oxidants (such as NaClO,[10] $Pb(OAc)_4$,[11] MnO_2,[12] PIFA[13] and I_2[14]). Consequently, more efficient and sustainable synthetic approaches for the access of this heterocyclic scaffold are still required.



Scheme 1 Electrochemical synthesis of triazolopyridines

Electrochemical anodic oxidation, which uses electron to drive the reaction and avoids the use of chemical oxidants, is recognized as an environmentally friendly synthetic methodology.[15] Despite the fact that electrochemical methods have made great progress in the construction of C–C or C–heteroatom bonds, [16] the electrochemical construction of N–N bonds is still very rare. In 2014, Baran and co-workers developed an unique method of electrochemical dimerization of carbazoles and carbolines, which was applied as the pivotal step for the first total synthesis of a rare N–N linked dimeric natural product dixiamycin B.[17] Later, Waldvogel group achieved the novel access to pyrazolidin-3,5-diones and phthalazin-1,4-diones through intramolecular elecrochemical N–N bond formation of dianilides.[18-19] Recently, Xu and co-workers reported an electrochemical intramolecular N–N bond formation for the synthesis of [1,2,3]triazolo[1,5-*a*]pyridines from 2-acylpyridine hydrazines (Scheme 1a).[20] And our group developed an electrochemical synthesis of 1,2,4-triazolo[4,3-*a*]pyridines via a metal- and oxidant-free intramolecular dehydrogenative C–N bond formation (Scheme 1b).[21] With our continued interest in developing novel and efficient methodologies for the synthesis of privileged heterocycles,[21-22] herein we reported an electrochemical intramolecular dehydrogenative N–N bond formation for the synthesis of 1,2,4-triazolo[1,5-*a*]pyridines under metal- and oxidant-free conditions (Scheme 1c).

Results and discussion

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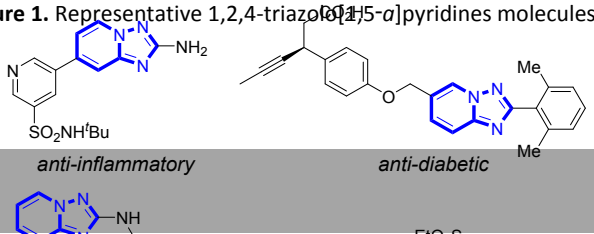
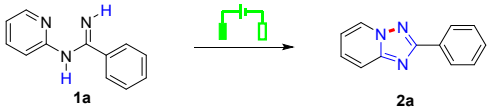
Figure 1. Representative 1,2,4-triazolo[1,5-*a*]pyridines molecules



***N*-(2-pyridyl) amide **1a** was used as the model substrate to test the oxidative cyclization conditions. The electrochemical reaction was conducted in MeCN at room temperature under constant current electrolysis conditions using a three-necked round bottom flask as an undivided cell, which equipped with a reticulated vitreous carbon (RVC) anode and a platinum plate cathode. Initially, different electrolytes were screened. There is no desired product formed with either NH_4Br, $^nBu_4NOAc$ or $^nBu_4NBF_4$ as the electrolyte (Table 1, entries 1-3). It was found the product **2a** was isolated in 35% yield when $^nBu_4NCl$ (2.0 equiv) was used as the electrolyte, which indicate that the halide ion might act as the redox **mediator** and promote the N–N bond formation (Table 1, entry 4).[23] Encouraged by this result, $^nBu_4NI$ and $^nBu_4NBr$ were then tested and **2a** was obtained in fair yields (Table 1, entries 5-6). Increasing the electrolyte's loading to 3.0 equiv., the yield could be improved to 73% yield (Table 1, entries 7-8). Increasing the current from 6 mA to 7 mA the yield was further improved to 86% yield (Table 1, entry 9). However, the yield was decreased slightly when the current was further increased to 8 mA (Table 1, entry 10). Gratifyingly, using graphite rod to replace RVC as anode led to 2a in 92% yield (Table 1, entry 11), which tallies with Waldvogel's observation for the performance of reaction set-up during their electrochemical synthesis of pyrazolidin-3,5-diones.[18] Switching to the other electrodes resulted in much lower yields (Table 1, entries 12-13).

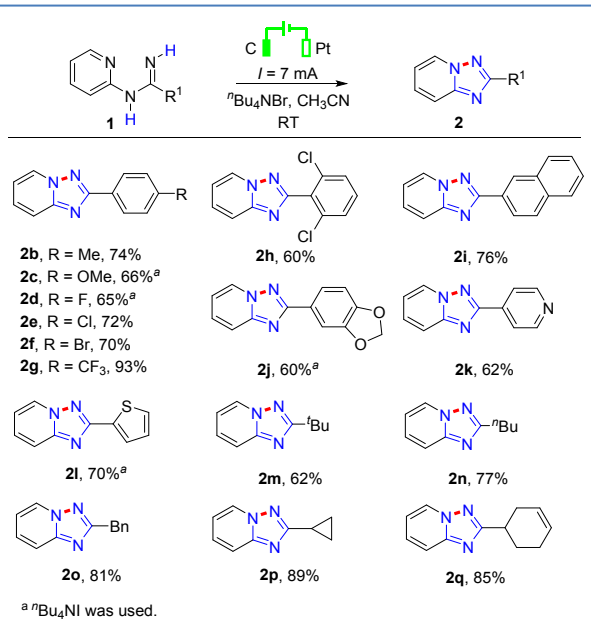
Table 1 Optimization of electrochemical conditions[a]



Entry	Anode/cathode	Electrolyte (equiv)	Current	Yield[b] (%)
1	RVC/Pt	NH_4Br (2.0)	6 mA	n.d.
2	RVC/Pt	$^nBu_4NOAc$ (2.0)	6 mA	n.d.
3	RVC/Pt	$^nBu_4NBF_4$ (2.0)	6 mA	n.d.
4	RVC/Pt	$^nBu_4NCl$ (2.0)	6 mA	35
5	RVC/Pt	$^nBu_4NI$ (2.0)	6 mA	54
6	RVC/Pt	$^nBu_4NBr$ (2.0)	6 mA	55
7	RVC/Pt	$^nBu_4NI$ (3.0)	6 mA	65
8	RVC/Pt	$^nBu_4NBr$ (3.0)	6 mA	73
9	RVC/Pt	$^nBu_4NBr$ (3.0)	7 mA	86
10	RVC/Pt	$^nBu_4NBr$ (3.0)	8 mA	78
11	C/Pt	$^nBu_4NBr$ (3.0)	7 mA	92
12	Pt/Pt	$^nBu_4NBr$ (3.0)	7 mA	66
13	C/C	$^nBu_4NBr$ (3.0)	7 mA	71

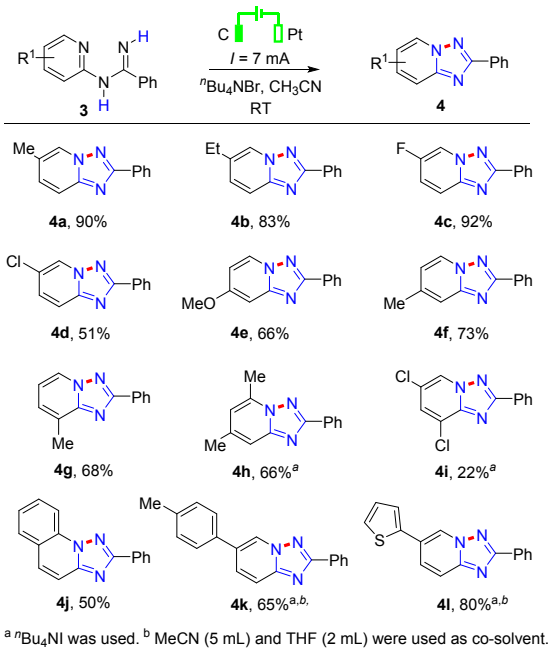
[a] Reaction conditions: Two-electrode undivided cell: RVC anode (100 PPI, 1 cm × 1 cm × 1 cm), Pt cathode (1 cm × 1 cm), **1a** (0.3 mmol), MeCN (7 mL), 2.6 F/mol. [b] Isolated yields. graphite rod (ϕ 6 mm, about 10 mm immersion depth in solution). n.d. = not detected

With the optimized conditions in hand, firstly we prepared the N-(2-pyridyl)benzamidine 1 by condensation of 2-aminopyridine with different nitriles and probed the effect of the substitutes on nitrile side (Scheme 2). Initially, the nitriles with aromatic substituent were tested which all gave the corresponding products in good to excellent yields (2b-2l). Both electron-donating and withdrawing groups were tolerated. Also, the halogen such as F and Cl were compatible with the reaction conditions which allows for further transformations. Naphthyl (2i), pyridine (2k) and thiophene (2l) carbonitriles were effective as well. It is worth mentioning that the methods are not only compatible with aromatic nitriles, but alkyl nitriles are also well tolerated (2m-2q). The nitriles with either linear or cyclic alkyl substituents can afford the desired products with good yields.



Scheme 2 Scope of nitrile derived substrates

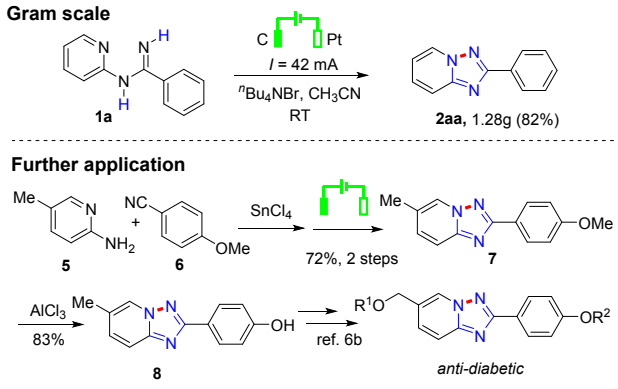
The scope of the aminopyridines was then studied (Scheme 3). The substrates with simple methyl or ethyl substituents gave the corresponding products in good to excellent yields (**4a**, **4b**, **4f**, **4g** and **4h**). Again, both fluorine (**4c**) and chlorine (**4d**) were tolerated under the reaction conditions although two chlorines in the aminopyridine moiety decreased the reaction efficiency for N–N bond formation (**4i**). A moderate yield could be achieved by changing the aminopyridine to aminoquinoline (**4j**). In addition, the substrates bearing tolyl (**4k**) or thienyl (**4l**) substitutents were also effective by employing $^nBu_4NI$ as the electrolyte in a mixture of solvent.



[a] $^{n}Bu_4NI$ was used. [b] MeCN (5 mL) and THF (2 mL) were used as co-solvent.

Scheme 3 Scope of aminopyridine derived substrates

The scalability of this electrochemical dehydrogenative N–N bond formation was then evaluated by performing an 8 mmol scale reaction (Scheme 4). With almost the same ratio of 3 F/mol of electricity, the cyclization of **1a** smoothly furnished the desired product **2a** in 82% yield. This result demonstrates the potential industrial applications of this electrochemical dehydrogenative reaction. Furthermore, we demonstrated that our newly developed electrochemical synthesis can be applied as the key step for the synthesis of a series of compounds having anti-diabetic activity. With pyridinamine **5** and phenylnitrile **6** as the starting materials, the compound could be prepared efficiently through nucleophilic substitution promoted by tin tetrachloride, electrochemical dehydrogenative N–N bond formation, and demethylation with aluminum trichloride, which could be further transformed into various bioactive molecules.[6b]



Scheme 4 Gram scale reaction and further application

To clarify the role of $^{n}Bu_4NBr$, cyclic voltammetry (CV) experiments were performed. As shown in Figure 1, *N*-(2-pyridyl)benzamide **1a** was oxidized to 1.36 V (vs. Ag / AgCl) (curve b). Meanwhile, $^{n}Bu_4NBr$ showed two distinct oxidation peaks (vs Ag / AgCl) and one reduction peak at 1.08 V (vs. Ag / AgCl) at 0.83 V and 1.26 V (curve c). The oxidation potential of *N*-(2-pyridyl)benzamide **1a** was higher than that of bromide ions (1.36 V vs. 1.26 V and 0.83 V). Compared to pure $^{n}Bu_4NBr$, the CV of $^{n}Bu_4NBr$ with *N*-(2-pyridyl)benzamide **1a** exhibited an obvious catalytic current, and the reduction peak disappeared (curve d). This is the typical behaviour of mediator-assisted oxidation and indicates that the oxidized form of $^{n}Bu_4NBr$ reacts with the *N*-(2-pyridyl)benzamide **1a** and therefore cannot be reduced in the second part of the cycle. And these results indicate that the reaction involves indirect electrolysis.[24]

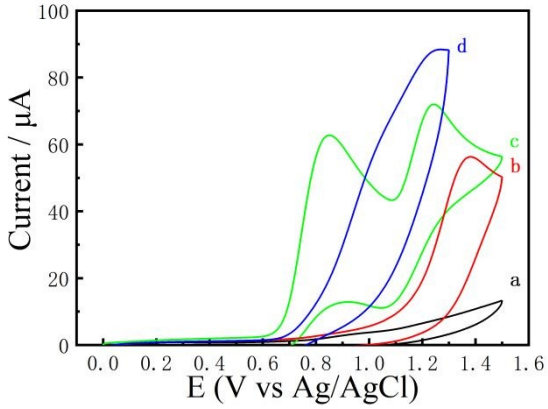
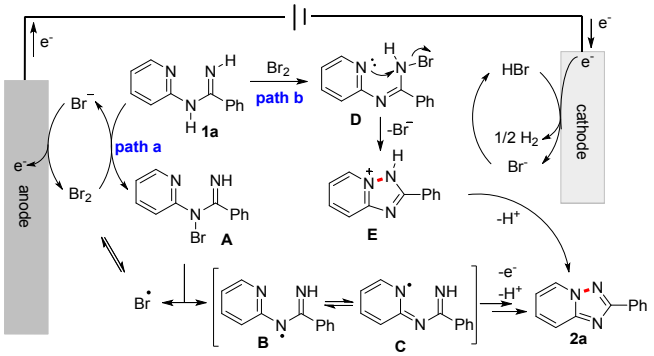


Figure 1 Cyclic voltammograms. a: background; b: **1a** (5 mM); c: $^{n}Bu_4NBr$ (5 mM); d: **1a** + $^{n}Bu_4NBr$ (5 mM).

According to the cyclic voltammograms results and literature reports,[13, 20] a possible mechanism for this dehydrogenative N–N bond formation was proposed as shown in Scheme 5. With $^{n}Bu_4NBr$ as both the redox mediator and electrolyte, there are two possible pathways leading to the product **2a**. The first reaction pathway (path a) could begin with the anodic oxidation of bromide ion to form molecular bromine, which reacts with substrate **1a** to afford the intermediate **A**. A homolytic cleavage might lead to the intermediate **B** which has a resonance structure **C**. Subsequent oxidative cyclization and deprotonation would afford **2a**. The other pathway (path b) **1a** might begin with the bromination of the imine to give the intermediate **D**. A direct intramolecular cyclization might generate the intermediate **E** which would give **2a** after deprotonation.



Scheme 5 Proposed mechanism

Conclusions

In conclusion, we have present an environmentally friendly method to prepare valuable 1,2,4-triazolo[1,5 - *a*]pyridines via dehydrogenative N–N bond formation. The key is the employment of $^{n}Bu_4NBr$ or $^{n}Bu_4NI$ as both the redox **mediator** and electrolyte in an undivided cell. Neither transition metals nor additional oxidants are required for this simple and scalable protocol.

Acknowledgements

This research was supported by the Natural Science Foundation of China grant No. 21871234, Zhejiang Provincial Natural Science Foundation of China for Distinguished Young Scholars under grant No. LR15H300001. We also thank the Zhejiang Provincial Thousand-Talent Program and Zhejiang University of Technology for financial support.

Conflicts of interest

There are no conflicts to declare.

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