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Electrochemical-induced regioselective C-3 thiocyanation of imidazoheterocycles with hydrogen evolution

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ABSTRACT

A direct and efficient protocol for thiocyanation of imidazoheterocycles accompanying with the hydrogen evolution under electrochemical oxidation has been described. Various important thiocyanated and selenocyanated imidazoheterocycles have been constructed through this method in moderate to excellent yields and can easily be scaled up. Further mechanistic studies suggest that aryl radical cation is the key intermediate in this transformation.

Introduction

The direct and efficient construction of diverse organic thiocyanates is of great importance since thiocyanated compounds are widely exist in natural products [1], and are widely used in pharmaceutical ingredients and material science as well [2]. Aryl and hetero-aryl thiocyanates can be converted to various sulfur-containing derivatives including thioheterocycles [3], thioethers [4], sulfur carbamates [5] conveniently. Therefore, since their potentially wide applications, it's reasonable that further research works aiming at furnishing thiocyanated arenes and heterocycles have been conducted. However, many reported reactions still suffer from some drawbacks, including the need of metal catalysts/promoters (such as Mn [6], CAN [7], Cu [8], Pd [9]) or stoichiometric oxidants (such as iodonium reagent [10], NCS [11], oxone [12], $K_2S_2O_8$ [13]). In recent years, some visible light-induced thiocyanation reactions have been reported [14], which still used some unrecoverable photocatalysts and oxidants. On the other hand, imidazo [1, 2-*a*]pyridine scaffolds is an important synthesis units which were widely found in natural products, biological activities and optoelectronic material [15]. The most important is that some commercial available drugs, such as zolpidem, contain the imidazo[1, 2-*a*]pyridine skeletons. Therefore, the synthesis of thiocyanated imidazo[1,2-*a*] pyridines has drawn us much extensive attention. In this context, Wang and coworkers [13a] reported the regioselective C-3 thiocyanation of imidazopyridines under catalyst-free conditions using 1.5 equiv of $K_2S_2O_8$ as an oxidant (Scheme 1a). In the same year, Hajra and coworkers [14b] described the thiocyanation of imidazoheterocycles through visible light photoredox catalysis using eosin Y as the photocatalyst and oxygen as an oxidant (Scheme 1a). Nevertheless, these protocols suffer from the use of excess amounts of oxidant, which lead to some limitations for its applications.

The past decade has witnessed an increasing interest in organic electrosynthesis, which expanded the toolbox of organic chemists for green synthesis [16]. After years of development, electrochemical anodic oxidation has emerged as a versatile, environmentally friendly and powerful method for the C—C [17], C—N [18], C—O [19], C—S [20], C-P [21] bonds formation, which make it more economical and practical to thiocyanate C(sp^2)-H bonds through an electrochemical oxidative way. However, these reported examples mainly focus on indoles [22] and other active compounds [23]. In addition, Petrosyan and coworkers [23b] performed the thiocyanation of pyrazolo[1,5–a]pyrimidines in three-electrode undivided

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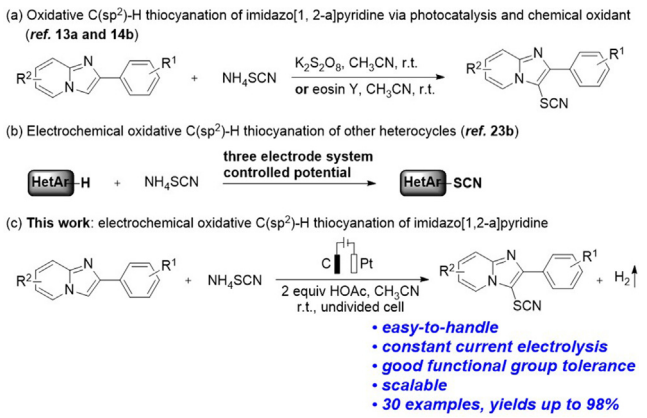
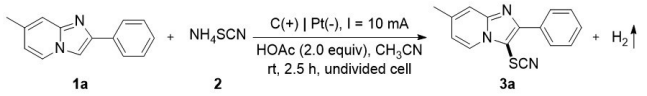


Scheme 1. Oxidative C(sp^2)-H thiocyanation.

cell under controlled potential conditions, which was not easy-operating, especially for industrial preparation (Scheme 1b). Herein, with the aid of anodic oxidation and cathode hydrogen evolution, the 3-thiocyanatoimidazopyridines can be furnished under mild conditions without the usage of redox catalysts and excess oxidants in a simple undivided cell (direct electrolysis) (Scheme 1c). During the preparation of this manuscript, Wang and coworkers have described selected examples of this transformation [15m].

To optimize the reaction conditions, 7-methyl-2-phenylimidazo [1,2–a]pyridine **1a** and ammonium thiocyanate (NH_4SCN) **2** were used as the model substrates in this study (Table 1). The desired product **3a** was obtained in 95% yield as the best reaction result after investigating a series of the key reaction factors. At the same time, H_2 can be detected by GC- TCD. The optimal condition was conducting the electrolysis at a constant current of 10 mA·cm^{-2} at room temperature, using an undivided cell equipped with a platinum plate cathode and a graphite rod anode (Table 1, entry 1). Neither decreasing nor increasing the current would improve the reaction yields in the condition of the same faraday efficiency (Table 1, entries 2–3). The influence of the electrode materials was investigated as well. From our experiment result, it seems that the replacement of anode from graphite rod to platinum plate leads to a tiny decrease of reaction yield (Table 1, entry 4). However, the absence of acetic acid in the reaction system lead to a severe decrease of the efficiency of thiocyanation (Table 1, entry 5).

Table 1
Optimization of conditions.[a]



Entry	Variation from the standard conditions	Yield (%)[b]
1	none	95
2	8 mA instead of 10 mA, 3 h	90
3	12 mA instead of 10 mA, 2.2	93
4	Pt(+) |Pt(-) instead of C(+) | Pt(-)	90
5	no HOAc	80
6	KSCN (2 equiv) instead of NH_4SCN (2 equiv)	85
7	NH_4SCN (4 equiv) instead of NH_4SCN (2 equiv)	96
8	without current	n.d.
9	reaction in the air	95
10	without current in air	n.d.

[a] Standard conditions: C anode, Pt cathode, constant current = 10 mA, **1a** (0.2 mmol), **2** (0.4 mmol), HOAc (0.4 mmol), CH_3CN (10.0 mL), room temperature, 2.5 h; H_2 was detected by gas chromatography. n.d. = not detected. [b]Isolated yields were showed.

The replacement of ammonium thiocyanate with potassium thiocyanate resulted in a slight decrease in yield due to its poor solubility in organic solvent (Table 1, entry 6). Further increasing the equivalent of ammonium thiocyanate did not observably improve the yield (Table 1, entry 7). Furthermore, the results of some controlled experiments showed that the reaction did not proceed without current in air or nitrogen atmosphere (Table 1, entries 8, 10). Notably, the efficiency and yield of the reaction was not affected in air under the constant current electrolysis conditions (Table 1, entry 9).

With the optimized conditions in hand, we next turned to explore the functional group tolerances for the thiocyanation of imidazopyridines (Table 2). Significantly, good functional group tolerance of imidazopyridines could be achieved. For instance, electron effect did not affect the reactivity so much. Imidazopyridines containing electron-neutral or electron-rich substituents at any aryl ring were transformed to the corresponding products in good to excellent yields (Tables 2, 3a–e, p). Electron-poor functional groups, such as $MeSO_2$-, CN–, CF_3- and CH_3OOC–, could also be tolerated and showed moderate to good reactivity under the present reaction conditions (Tables 2, 3f, g, h, i, q). Additionally, the results of **3b** and **3c** showed that the steric effect of the thiocyanation reaction was not obvious. Substrates with halogen groups, such as fluoro, chloro or bromo, furnished the desired products in good yields, which could be further converted to other useful functional groups (Tables 2, 3j, k, l, 3r, s, t). The naphthyl and thienyl substituted substrates were abided smoothly with excellent yields under this transformation (Tables 2, 3m and n). Interestingly, the styryl substituted imidazopyridine also got an excellent result (Tablse 2, 3o), while the di-substituted substrate was effective for thiocyanation as well (Tables 2, 3u). Moreover, 2-phenylimidazo[1,2–a]pyrimidine was also tested and could be transformed into the thiocyanated product with 93% yield (Tables 2, 3v). Unfortunately, when using imidazo[1,2–a]pyridine as the substrate, no desired product **3w** was obtained under standard conditions. A reasonable explanation is that the imidazo[1,2-*a*]pyridine is not easily oxidized because of its relatively high oxidation potential. Notably, the selenocyanated products **3x** and **3y** were obtained in moderate yield, which extended the scope of the reaction to selenocyanation (Scheme 2). The structure of **3b** and **3r** were confirmed by X-ray crystallographic analysis (see Supporting Information for details).

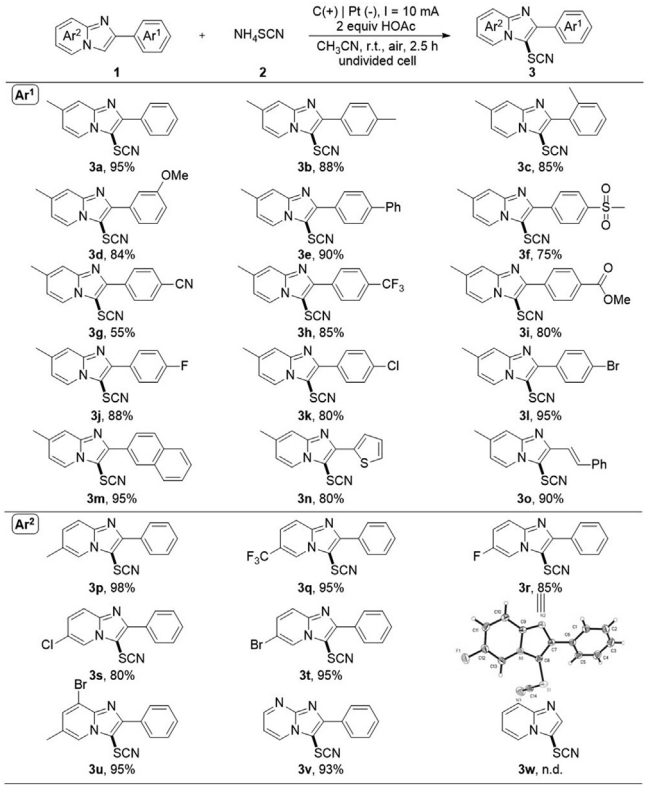
To explore the synthetic potential of this electrochemical oxidative C(sp^2)–H dehydrogenative thiocyanation, some imidazo[2,1-*b*] thiazoles, such as **4a-4f**, were synthesized and explored the reactivity under standard conditions (Table 3). Satisfactorily, corresponding thiocyanated products **5a-5f** could be successfully obtained in 60–95% yield.

Then, this thiocyanation reaction on a gram scale was also carried out. When 5 mmol of **1a** reacted with 10 mmol of **2**, the desired products of **3a** were obtained in 85% isolated yields, with the generation of H_2 at the same time (Scheme 3). Thus, this result exhibited a great potential of this electrochemical synthesis in the construction of various biologically active imidazopyridines.

In order to gain more insight of the reaction mechanism, some controlled experiments were designed and practiced in Scheme 4. Interestingly, both TEMPO (2,2,6,6-tetramethylpiperidin-1-yl)oxy and triethyl phosphite $P(OEt)_3$ [21b] could fully inhibit the reaction, which suggested that the reaction probably proceeded through a radical process.

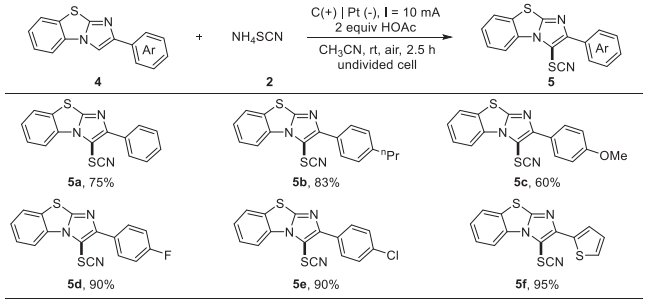
At last, to study the redox potential (Fig. 1), cyclic voltammetry (CV) experiments of **1a** and **2** were performed. Obviously, the oxidation peaks of **1a** and **2** were observed at 1.72 V Vs Ag/AgCl and 1.96 V Vs Ag/AgCl respectively. The onset potential of two substrates are almost the same, which indicated that both **1a** and **2** might be oxidized at anode under standard conditions.

Table 2
Substrates scope of the synthesis of 3-thiocyanatoimidazopyridines.[a]

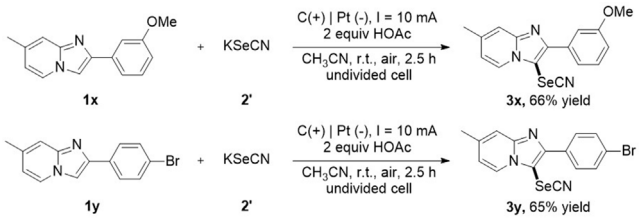


[a] Standard conditions: C anode, Pt cathode, constant current = 10 mA, **1** (0.2 mmol), **2** (0.4 mmol), HOAc (0.4 mmol), CH_3CN (10 mL), room temperature, 2.5 h; isolated yields were showed.

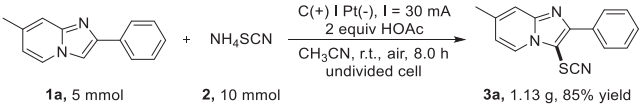
Table 3
Substrates scope of benzo[*d*]imidazo[2,1-b]thiazole.[a]



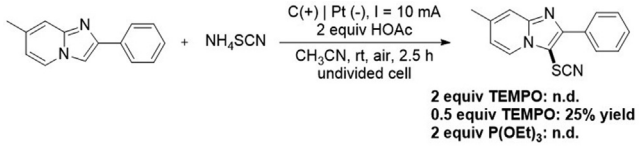
[a] Standard conditions: C anode, Pt cathode, constant current = 10 mA, 4 (0.2 mmol), 2 (0.4 mmol), HOAc (0.4 mmol), CH3CN (10 mL), room temperature, 2.5 h; isolated yields were showed.



Scheme 2. Selenocyanation of imidazo[1,2-*a*]pyridine.



Scheme 3. Gram-scale synthesis.



Scheme 4. Radical quenching experiments.

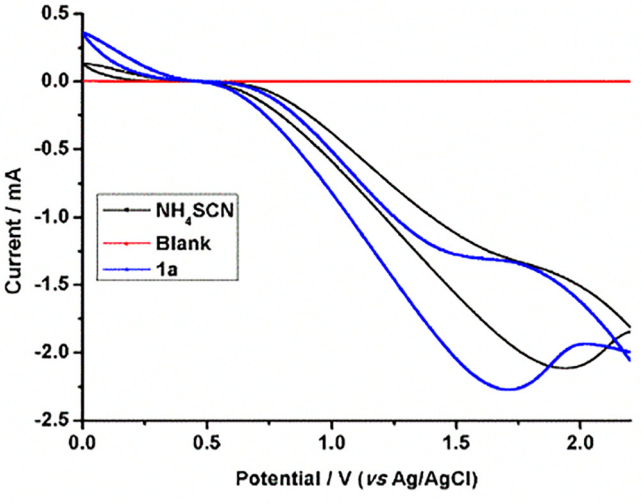
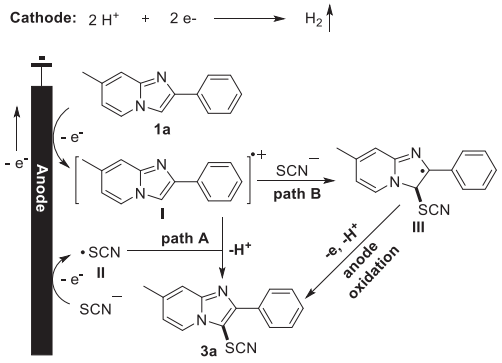


Fig. 1. Cyclic voltammetry of **1a** (0.1 mmol, 0.01 M) or **2** (0.2 mmol, 0.02 M) in 10 mL CH_3CN with 24 uL HOAc under air atmosphere. A glassy carbon working electrode (diameter (d) = 2 mm), Ag/AgCl reference electrode and a platinum wire counter electrode were used. The scan rate was 100 mV/s.

In terms of above results as well as previous reports [22,23b,23c,23d], two plausible mechanisms (path A and path B) for this electrochemical oxidative C(sp^2)–H thiocyanation of 7-methyl-2-phenylimidazo[1,2-*a*]pyridine with NH_4SCN are described in Scheme 5. Firstly, 7-methyl-2-phenylimidazo[1,2-*a*] pyridine **1a** is oxidized to form a radical cation **I** through the anodic oxidation. Meanwhile, thiocyanate anion could also be oxidized to thiocyanyl radical **II** on the surface of anode, which subsequently react with **I**. The intermediate then quickly deprotonized to generate the desired product **3a** (path A). Additionally, the radical cation intermediate **I** have a low SOMO, while thiocyanate anion has a HOMO at higher energy level. It can be predicted that those two species have a strong SOMO-HOMO interaction, which leads to the combination and directly form a radical intermediate **III,** which then rapidly give out an electron to anode and deprotonate to furnish the product **3a** (path B). At the same time, concomitant cathodic reduction of H^+ lead to the hydrogen gas release.

In conclusion, we have developed a catalyst- and oxidant-free protocol of electrochemical oxidative thiocyanation of imidazopyridines and imidazo[2,1-*b*]thiazoles, accompanying with the hydrogen evolution. Synthetically, this straightforward, efficient and easy-operating method exhibits a wide range of substrates scope and excellent tolerance towards functional groups. More importantly, some drug candidate products such as 3-thiocyanatozolimidines can be constructed in moderate to excellent yields and



Scheme 5. Proposed mechanism.

can easily be scaled up. Besides, this electro-oxidative method can also be applied for the selenocyanation. Mechanistically, detailed mechanistic studies strongly suggested that the reaction was triggered by an aryl cation radical created by anodic oxidation. Further applications of this reaction to synthesize other bioactive compounds are underway in our laboratory.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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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.2020.152755.

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