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An Electrochemical Way to Generate Amphiphiles from Hydrazones for the Synthesis of 1,2,4-Triazole Scaffold Cyclic Compounds

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An electro-oxidative cyclization pathway in which hydrazones are selected as starting materials to generate amphiphiles by reacting with benzylamines and benzamides was reported. This strategy successfully prepared a series of 1,2,4-triazoles in satisfactory yields. Moreover, the use of cheap stainless steel as

the anode, the feasibility to conduct the transformation as a one-pot reaction and the proof that scaling-up these reactions is possible make this transformation attractive for potential application in industry.

Introduction

As the most widespread scaffolds, cyclic skeletons, ubiquitously existing in plenty of functional materials and pharmaceuticals, have drawn rather considerable interest in recent years.^[1] Undoubtedly, a cyclization has been considered as the most appealing strategy for the construction of highly functionalized ring molecules, because at least two new bonds are formed in one step by using acyclic coupling partners.^[2] Therefore, realizing the efficient synthesis of diverse cyclic molecules from simple and easily available starting materials through cyclization pathways has always been a research hotspot at home and abroad.^[3] Notably, novel strategies and great breakthroughs have been continuously achieved in this field with the aid of transition metal catalysts,^[4] organic catalysts such as amines^[5] and phosphines,^[6] and inorganic catalysts like iodine.^[7] However, transition metal salts, harsh conditions and strong chemical oxidants are always indispensable for these transformation, while the toxicity of transition metals, over-oxidation and high temperatures are sometimes destructive to biomedical fields. With the development of more green and sustainable chemistry approaches, it is of great significance to explore an

alternative atom- and step-economic and eco-friendly annulation avenue to furnish cyclic compounds.

1,2,4-Triazoles are significant skeletons in numerous pharmaceuticals and biological active compounds. Therefore, many methods for synthesis of 1,2,4-triazoles have been reported in recent years.

For example, in 2012, Punniyamurthy and co-workers reported the copper(II)-catalyzed aerobic oxidative synthesis of 1,2,4-triazoles from bisarylhydrazones (Scheme 1a).^[8] In 2016, Ren and co-workers developed an I₂-catalyzed oxidative coupling reactions hydrazones and amines (Scheme 1b).^[9] More recently, organic electro-synthesis is regarded as an environmentally friendly and gentle method of inducing free radicals and is gradually being used in cyclization reactions, such as the synthesis of 1,2,4-triazoles. In 2018, Zhang's group reported the electrochemical synthesis of tetrazoles through a [3 + 2] cyclo-addition of azides with hydrazones (Scheme 1c).^[10] In 2018, Zhang and co-workers also developed an electrochemical synthesis of 1,2,4-triazole-fused heterocycles (Scheme 1d).^[11] In 2018, Yuan's group reported a multicomponent electro-synthesis

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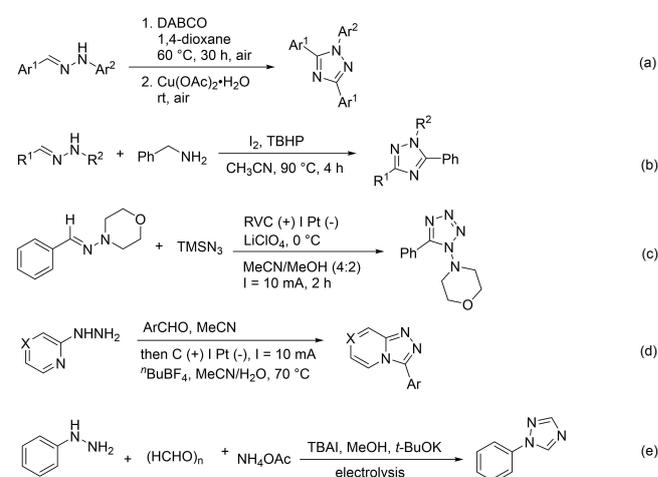
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Scheme 1. Different methods for the synthesis of 1,2,4-triazoles.

of 1,5-disubstituted and 1-aryl-1,2,4-triazoles (Scheme 1e).^[12] Although there was much progress in the synthesis of multi-substituted 1,2,4-triazoles, developing a simple and efficient method for the synthesis of 1,2,4-triazoles is still necessary.

Results and Discussion

In 2019, inspired by the mechanism of difunctionalization of alkenes,^[13] our group has attempted to find or synthesize some molecules, herein named bifunctional reagents, which can serve as both radical precursors to electrochemically generate radicals **M** (Scheme 2a) to react with alkenes and nucleophiles to subsequently couple with cationic species to finish the intramolecular cyclization. Based on this idea, we have successfully achieved the preparation of a series of heterocyclic compounds, including dihydrofurans, isoxazolines, oxazines and pyrroles by using 1,3-dicarbonyl compounds as bifunctional reagents.^[14] We wondered whether the radical species, formed from the corresponding bifunctional reagent, could undergo further oxidation to release an amphiphile to couple with another molecule which simultaneously possesses electrophilic sites and nucleophilic sites, eventually giving cyclic compounds (Scheme 2b). Because of the electron-withdrawing effects of the carbonyl groups, the acetylacetone-derived C-centered radical is difficult to be oxidized in order to give the corresponding cationic species. After further analogical reasoning, hydrazones were eventually selected as the desired materials because of the low oxidative potential of the corresponding benzylic radical (Scheme 2c).^[15] In view of our interest in heterocyclic compounds and the significance of 1,2,4-triazole units in heterocycles,^[16] we herein aim to verify this concept by achieving an electro-oxidative [3+2] annulation between hydrazones and amines to synthesize 1,2,4-triazole derivatives.

To realize the desired transformation, we initially chose (*E*)-1-benzylidene-2-phenylhydrazine **1a** and benzylamine **2a** as the model substrates to optimize the reaction conditions in

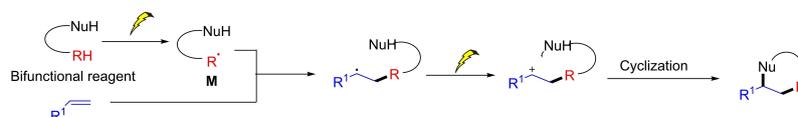
Table 1. Optimization of reaction conditions.^[a]

Entry	Variation from the standard conditions	yield ^[b] (%)
1	none	81
2	10 mA instead of 8 mA, 9 h	75
3	6 mA instead of 8 mA, 15 h	73
4	^t Bu ₄ NBF ₄ instead of ^t Bu ₄ NPF ₆	75
5	^t Bu ₄ NOAc instead of ^t Bu ₄ NPF ₆	58
6	without NaOAc	20
7	HOAc instead of NaOAc	68
8	Na ₂ CO ₃ , K ₂ CO ₃ , or NaHCO ₃ instead of NaOAc	63, 60 or 63
9	DMF, MeCN or DCM instead of DCE	48, 68 or 71
10	45 °C instead of 55 °C	66
11	65 °C instead of 55 °C	55
12	C rod (+) or C felt (+) instead of C cloth (+)	66 or 68
13	Pt (-) or Ni (-) instead of SSC (-)	75 or 62
14	without Cp ₂ Fe	70
15	TEMPO, KI or DDQ instead of Cp ₂ Fe	75, 80 or 81
16	under air	72
17	without electricity	N.D. ^[c]

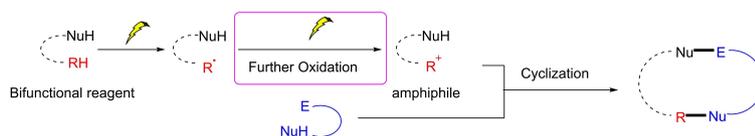
[a] Reaction conditions: carbon cloth (15 × 15 mm), SSC = stainless steel cathode (10 × 10 × 0.3 mm), constant current = 8 mA, **1a** (0.5 mmol), **2a** (1.5 mmol), ^tBu₄NPF₆ (0.5 mmol), NaOAc (0.5 mmol), Cp₂Fe (0.05 mmol), DCE (10 mL), argon, 55 °C, 11.5 h, undivided cell; [b] isolated yield; [c] N.D. = not detected.

Table 1. The best isolated yield (81 %) of **3a** was obtained when the reaction was conducted under the optimal conditions (entry 1). Both elevating and lowering the reaction current were found to slightly reduce the yields (entries 2 and 3). Moreover, other electrolytes like ^tBu₄NBF₄ and ^tBu₄NOAc gave inferior yields (entries 4 and 5). Removing the base NaOAc from the reaction system led to a sharply decreased yield (entry 6), implying the base to facilitate the removal of protons to some

a) Our previous method for preparing cyclic compounds via electrocatalysis



b) **This pathway:** electrochemically generating amphiphilic reagents for preparing cyclic compounds



c) **This work:** electrochemically generating the amphiphiles from hydrazones for the synthesis of 1,2,4-triazoles



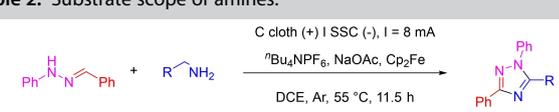
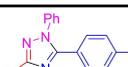
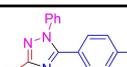
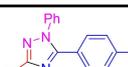
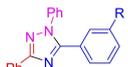
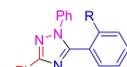
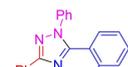
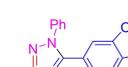
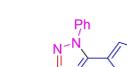
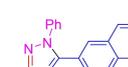
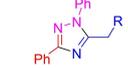
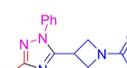
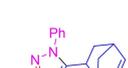
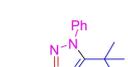
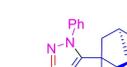
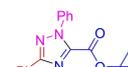
Scheme 2. Our strategies for the construction of ring compounds.

extent. Subsequently, some acids or other bases as additives were examined, also resulting in declined yields (entries 7 and 8). Furthermore, other common solvents like DMF, MeCN or DCM all negatively impacted the reactions (entry 9). We also found that increasing or decreasing the temperature led to disappointing experimental results (entries 10 and 11). In addition, unsatisfactory reaction efficiencies were observed when using carbon rod and carbon felt as anodes or palladium and nickel as cathodes (entries 12 and 13). In order to promote electro-oxidative efficiency, we screened some common mediators^[17] and observed that KI, DDQ (4,5-dichloro-3,6-dioxocyclohexa-1,4-diene-1,2-dicarbonitrile) and Cp₂Fe gave similar yields, about 80% (entries 14 and 15). When the reaction was carried out under air, the yield slightly decreased to 72% (entry 16). Expectedly, no target product was formed without current passing through the reaction system (entry 17).

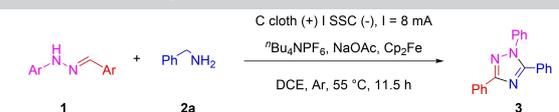
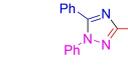
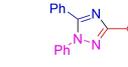
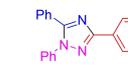
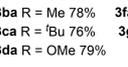
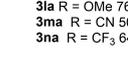
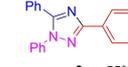
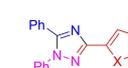
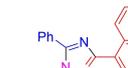
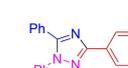
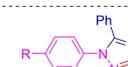
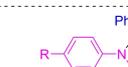
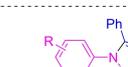
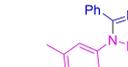
With the optimized conditions in hand, the substrate scope of reaction in regard to the amines **2** was explored in Table 2. Varieties of *para*-substituted benzylamines, including alkyl, alkoxy, amino, aryl, halo, trifluoromethyl counterparts, were extremely amenable to this transformation, leading to a series of target products **3aa–3al** in 51–81% yield. Moreover, *ortho*- and *meta*-substituted benzylamines, with either electron-donat-

ing or -withdrawing groups on the aromatic ring, were also smoothly converted into the corresponding products **3am–3av** in considerable yields, especially the desired product **3av**, indicating the electric effect and steric hindrance effect of the substituents on the benzene ring had little influence on the reaction yields. Moreover, disubstituted benzylamine was also compatible with this protocol, leading to **3aw** in moderate yield. Of course, the reactivity of other aryl-methylamines like furan-2-ylmethanamine was also investigated, and the target products **3ax–3az** all were isolated in about 80% yield. Subsequently, we found that alkylamines are also suitable substrates in this transformation, furnishing the products **3aa'–3ag'** in moderate to high yields. Finally a glycine-derived substrate was subjected to the reaction and 1,2,4-triazole **3ah'** was formed in satisfactory yield.

Then, we focused on the applicability of the different hydrazone derivatives. As can be seen from Table 3, the reaction proceeded smoothly when a variety of hydrazones derived from diversely substituted benzaldehydes were used, and a series of 1,2,4-triazoles **3aa–3oa** were generated in 15–81% isolated yield. Gratifyingly, a large variety of substituent groups were tolerated, including alkyl, alkoxy, cyano, halo,

Table 2. Substrate scope of amines. ^[a]		
		
 3aa R = H 81% 3ab R = Me 80% 3ac R = ⁱ Pr 75% 3ad R = ^t Bu 79%	 3ae R = OMe 76% 3af R = OPh 80% 3ag R = N(Me) ₂ 62% 3ah R = Ph 73%	 3ai R = F 80% 3aj R = Cl 77% 3ak R = Br 51% 3al R = CF ₃ 70%
 3am R = Me 80% 3an R = CN 71% 3ao R = COOMe 70% 3ap R = CF ₃ 73%	 3aq R = Me 78% 3ar R = OMe 78% 3as R = F 80% 3at R = Cl 70% 3au R = Br 62%	 3av 70%
 3aw 52%	 3ax X = O 78% 3ay X = S 81%	 3az 80%
 3aa' R = Me 70% 3ab' R = ⁿ C ₁₀ H ₂₂ 84% 3ac' R = Ph 45%	 3ad' 64%	 3ae' 58%
 3af' 38%	 3ag' 40%	 3ah' 73%

[a] Reaction conditions: carbon cloth (15 × 15 mm), SSC = stainless steel cathode (10 × 10 × 0.3 mm), constant current = 8 mA, **1a** (0.5 mmol), **2a** (1.5 mmol), ⁿBu₄NPF₆ (0.5 mmol), NaOAc (0.5 mmol), Cp₂Fe (0.05 mmol), DCE (10 mL), argon, 55 °C, 11.5 h, undivided cell, isolated yield.

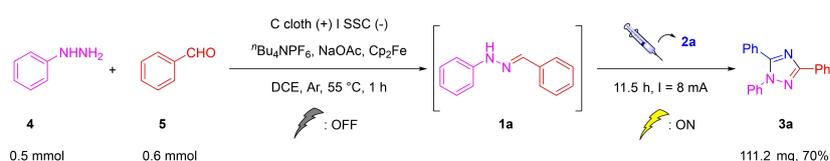
Table 3. Substrate scope of hydrazones. ^[a]		
		
 3aa R = H 81% 3ba R = Me 78% 3ca R = ^t Bu 76% 3da R = OMe 79%	 3ae R = OPh 79% 3fa R = OAc 15% 3ga R = CF ₃ 56%	 3ha R = F 78% 3ia R = Cl 60% 3ja R = Br 23% ^b
 3ka R = Me 65% 3la R = OMe 76% 3ma R = CN 50% 3na R = CF ₃ 64%	 3oa 52%	 3pa 71%  3qa 55%
 3ra X = S 51% ^[b] 3sa X = O 32%	 3ta 35%	 3ua 57%
 3va R = Me 66% 3wa R = ^t Bu 62% 3xa R = CF ₃ 61% 3ya R = CN 31%	 3za R = F 76% 3za' R = Cl 74% 3ba' R = Br 77%	 3ca' R = 3-Br 66% 3da' R = 3-OCF ₃ 54% 3ea' R = 2-Br 61%
 3fa 42%	 3ga 66%	 3ha 65%

[a] Reaction conditions: carbon cloth (15 × 15 mm), stainless steel cathode (10 × 10 × 0.3 mm), constant current = 8 mA, **1a** (0.5 mmol), **2a** (1.5 mmol), ⁿBu₄NPF₆ (0.5 mmol), NaOAc (0.5 mmol), Cp₂Fe (0.05 mmol), DCE (10 mL), argon, 55 °C, 11.5 h, undivided cell, isolated yield; [b] 70 °C instead of 55 °C.

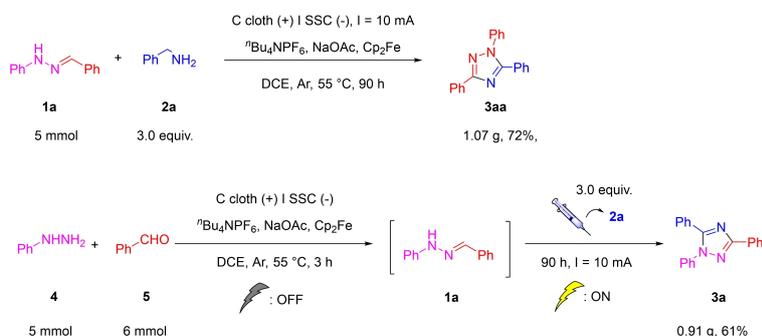
trifluoromethyl and ester groups. We speculate that the nucleophilic addition of benzylamine to the acetyl group of **1f** was the main reason why the yield of **3fa** was only 15%. In addition, hydrazones **1p** and **1q** were well compatible with the reaction conditions, delivering the desired products in 71% and 55% isolated yield, respectively. Moreover, several hydrazones derived from other aromatic aldehydes, such as furan-2-carbaldehyde, thiophene-2-carbaldehyde and naphthaldehyde, were also effective substrates for this transformation, releasing the corresponding products **3ra–3ua** in acceptable yields. By comparing yields of **3ta** and **3ua**, we came to the conclusion that the steric hindrance of the substituents should be taken into consideration. Subsequently, several hydrazone compounds **1v–1h'** were prepared through the condensation of substituted phenylhydrazines with benzaldehyde. To our delight, all these hydrazones, including both mono- and di-substituted derivatives, showed satisfactory reactivity and were successfully converted into the target 1,2,4-triazole products **3va–3h'a** in moderate isolated yields. Unfortunately, the desired alkyl product was not detected due to its lower reactivity.

Step-economic reactions have always been the pursuit of chemists. As, compared with hydrazones, phenylhydrazines and benzaldehydes are more commercially accessible, the one-pot reaction without intermediate treatment for the construction of 1,2,4-triazole compounds by using phenylhydrazines, benzaldehydes and benzylamines as starting materials is appealing and would be very practical. By turning the current on and off, we have achieved the one-pot transformation to synthesize **3a** as depicted in Scheme 3. Satisfactory isolated yields 72% and 61% have been obtained when the scale-up experiments of the model reaction and one-pot reaction were carried out on a 5 mmol scale, demonstrating the potential application of these transformations in the field of industry and medicine.

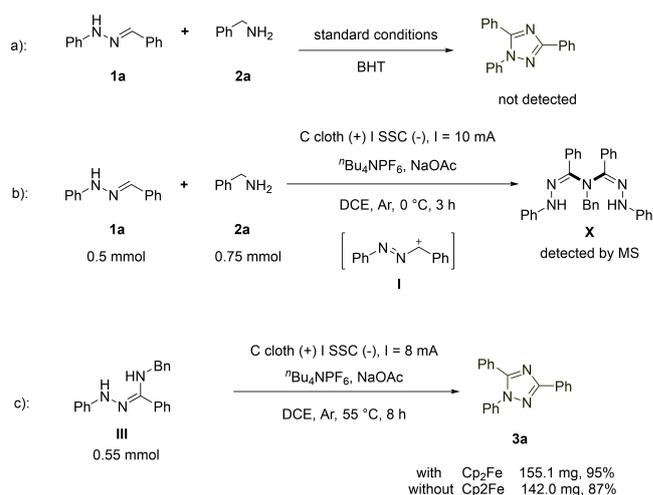
a) One-pot reactions by turning the current on and off



b) Scale-up experiments



Scheme 3. One-pot reaction and scale-up experiments (SSC = stainless steel cathode).



Scheme 4. Control experiments (SSC = stainless steel cathode, BHT = 3,5-di-*tert*-butyl-4-hydroxytoluol).

To shed light on the reaction mechanism, several control experiments were performed (Scheme 4). Adding radical inhibitor BHT (2,6-di-*tert*-butyl-4-methylphenol) led to compound **3a** not being detected. When the model reaction was conducted under similar conditions at 0 °C, compound **X** was detected by Mass Spectrometry, indicating cationic species **I** was formed in the reaction to a great extent (Scheme 4b). Moreover, compound **III** was synthesized as starting material and subjected solely into the reaction in the presence or absence of Cp_2Fe , and the final product **3a** was obtained in 95% or 87% yield, respectively. This result suggested compound **III** might be formed as the intermediate in the reaction, and on the other hand, Cp_2Fe might facilitate the dehydrogenation and aromatization. Furthermore, cyclic voltammetry (CV) experiments

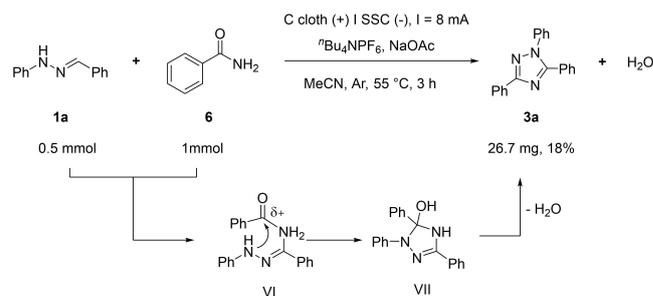
(Supporting Information, Figure S1) were also performed. Compared with benzylamine **2a**, a more obvious initial oxidative peak of hydrazone **1a** could be observed at 0.85 V (vs. Ag/AgCl). When the mixture of **1a** and **2a** was examined and an lower oxidative peak was observed at 0.50 V (vs. Ag/AgCl), indicating that hydrazone **1a** might undergo two consecutive oxidations, promoted by benzylamine **2a**, to form intermediate **I**.

Based on these results, a mechanism was proposed as shown in Scheme 5. Initially, with the aid of the base NaOAc and benzylamine **2a**, hydrazone **1a** is quickly oxidized at the anode to release the radical intermediate, promptly followed by the next oxidation process to give cationic species **I**, which then undergoes nucleophilic addition to form species **II**. The latter species can further tautomerize to **III**, which undergoes electrochemical oxidation to liberate the electrophilic species **IV**. Subsequently, intramolecular cyclization occurs, generating the ring compound **V**. Further electro-oxidative aromatization of **V** leads to the final 1,2,4-triazole product **3a**. At the same time, acetic acid can capture electrons at the cathode to release hydrogen, maintaining the electrochemical balance.

To test the validity of our proposed cyclization approach – electrochemically generating an amphiphile from hydrazones for the synthesis of cyclic compounds – instead of benzylamines **2**, benzamide **6** was chosen as substrate to react with the amphiphile formed from **1a** to give 1,2,4-triazole product **3a**. By this, we sought to test our hypothesis that the amino group of benzamide could couple with cationic species **I** to give compound **VI** and the carbonyl group of **VI** could serve as electrophilic site to be attacked to form intermediate **VII**. Finally, the release of one water molecule to generate the target product **3a**. With this idea in mind, we only did a preliminary experiment as shown in Scheme 6. To our delight, the desired product was obtained in 18% yield, proving the practicability of this strategy to a large degree. Follow-up experiments of this transformation are currently investigated in our laboratories.

Conclusion

In summary, we have firstly proposed an electrochemical cyclization pathway for the construction of ring compounds in which a bifunctional reagent can serve as both electrophile and nucleophile under electrolytic conditions. Based on this concept and by analogy with 1,3-dicarbonyl compounds, hydrazones

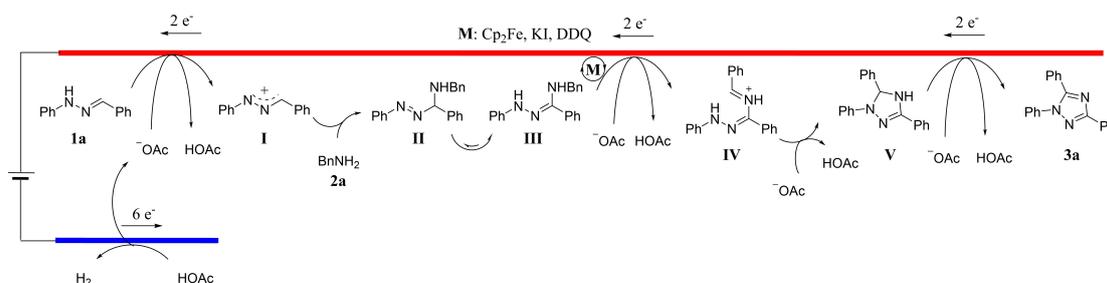


Scheme 6. Further confirmatory experiment of our proposed strategy (SSC = stainless steel cathode).

have been chosen as starting materials, which can electrochemically generate carbon-centered cations as the electrophilic site as well as a nitrogen-centered nucleophilic site to react with benzylamine derivatives for the construction of 1,2,4-triazole skeletons under mild and eco-friendly conditions. A series of functional groups are tolerated in this transformation and a great number of 1,2,4-triazoles are obtained in moderate to high yields. Moreover, the control experiments and CV experiments prove the reasonability of the proposed mechanism. More importantly, a preliminary and tentative annulation between hydrazones and benzamide has been successfully achieved, indicating the practicability of the proposed strategy to a great extent. We firmly believe that the reaction protocol might have instructive significance in further studies on the synthesis of ring compounds in organic electrochemistry.

Experimental Section

Experimental Procedures. To an oven-dried undivided three-necked bottle (20 mL) with a magnetic stir bar were added hydrazones **1** (0.5 mmol), Bu_4NPF_6 (0.5 mmol), NaOAc (0.5 mmol), Cp_2Fe (10%, 0.05 mmol). The bottle was equipped with a carbon cloth (15×15 mm) anode and a stainless steel (10×10×0.3 mm) cathode and was then flushed with argon. To the argon-filled bottle was successively added DCE (10 mL) and amines **2** (1.5 mmol). The reaction mixture was stirred and electrolyzed at a constant current of 8 mA at 55 °C for 11.5 h. After completion of the reaction, the solvent was removed under reduced pressure to afford the residue, which was purified by flash column chromatography on a silica gel using a mixture of petroleum ether and ethyl acetate to give the products **3**.



Scheme 5. Proposed mechanism for the transformation (DDQ = 2,3-Dichloro-5,6-dicyano-1,4-benzoquinone).

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Research data are not shared.

Keywords: amphiphiles · cyclization · electrooxidation · one-pot reaction · 1,2,4-triazole

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