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# Effective synthesis of symmetrical oxygen- and nitrogen-heterocycles from electron-deficient alkynes via the catalysis of electrogenerated base and $Fe^{3+}$ ions

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ABSTRACT

An efficient method has been developed for the synthesis of oxygen- and nitrogen-heterocycles from electron-deficient alkynes and alcohol or amines via electrochemically induced two-step process. The first step involves that the electrogenerated base (EGB) catalyzes hydroalcoholization or hydroamination process. In the second step, $Fe^{3+}$ ions and strong bases electrogenerated can synergically catalyze the cyclization process to give symmetrical heterocycles. Some key factors were investigated and the reaction mechanism was discussed.

## 1. Introduction

Oxygen- and nitrogen-heterocycles are fundamental structural units of a variety of natural products that have shown a broad range of biological activities, such as antiallergic and antiasthamatic properties,[1,2] and many drugs contain heterocyclic skeletons.[3–12] The search for efficient methods to synthesize the heterocycles continues to attract the great interest of synthetic chemists. By now, several methodologies have been developed for the construction of heterocyclic skeleton with metal catalysts (Pd, Ru, and Ag).[13–25] Although these methodologies are efficient, it remains a challenge for the convenient synthesis of diverse heterocycles with a simple catalytic system, particularly under mild conditions. The electrochemical method has an advantage over the classical methodologies because the electrons as a 'reagent' allow the formation of highly reactive intermediates (such as the electrogenerated bases, EGBs). These reactive intermediates are usually produced under mild reaction conditions, avoiding the use of toxic and harmful compounds (i.e., oxidative or reductive agents, acids, and bases as well as related waste byproducts). The electrochemically generated cyanomethyl anion has shown a good activity in many organic reactions. Many target compounds were conveniently synthesized via the use of the electrogenerated bases. For example, Feroci and co-workers have successfully employed cyanomethyl anion to synthesize a series of compounds, such as oxazolidin-2-ones, carbamates, 5-methylene-1,3-oxazolidin-2-ones, and β-lactams.[26–30]

Inspired by the literatures,[26–30] we attempted to synthesize the oxygen- and nitrogen-heterocycles from electron-deficient alkynes and alcohol or amines via the catalysis of electrogenerated bases and metal ions. The results indicated that the electrogenerated bases through pre-electrolyzed TBAB/MeCN solution can effectively catalyze the reaction of diethylbut-2-ynedioate (**1a**) with propan-1-ol (**2a**) to give the adduct (**3aa**) in good yields. Then, with the help of electrogenerated $Fe^{3+}$ ions and strong bases as catalysts, the adduct (**3aa**) could be smoothly cyclized to the symmetrical polysubstituted furan (**4aa**) (Scheme 1). Interestingly, with propan-1-amine to replace propan-1-ol, the symmetrical polysubstituted pyrrole can also be obtained under the similar conditions. To the best of our knowledge, such a synthetic route of the oxygen- and nitrogen-heterocycles has not been ever reported. Compared with the two-step conventionally synthetic process of polysubstituted furans,[23,24] the present approach is very clean and avoids the use of noble metal catalysts or some organic bases.

## 2. Results and discussion

### 2.1. The optimization of reaction conditions for the synthesis of 3aa

Substrates **1a** and **2a** were taken as model compounds to be investigated for the optimization of reaction conditions. The electrolysis of TBAB/MeCN electrolyte, followed by the addition of **1a** and **2a** into the catholyte, the electrolyte became a brown solution rapidly, which indicates the EGBs (i.e., cyanomethyl and other derived anions) can effectively promote the formation of **3aa**. No product (**3aa**) was obtained when the electrolysis was performed in an undivided cell, even if **1a** and **2a** was added at the beginning of the electrolysis. The yield of **3aa** was strongly depended on electricity, solvent, and the supporting electrolyte (Table 1). When the electricity passed through the cell from 1 to 2 F $mol^{-1}$, the yields

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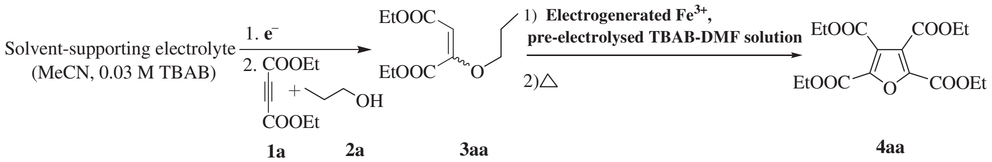


**Scheme 1.** The principle for the synthesis of symmetrical furan from diethylbut-2-ynedioate and propan-1-ol.

**Table 1**
The optimization of reaction conditions for the synthesis of **3aa**[a]

| Entry | Solvent | Supporting electrolyte | Electricity[b] (Q) | Yield of **3aa**[c] (%) |
|---|---|---|---|---|
| 1 | MeCN | TBAB | 1 | 82 |
| 2 | MeCN | TBAB | 2 | 96 |
| 3 | MeCN | TBACl | 2 | 75 |
| 4 | MeCN | TEATFB | 2 | 62 |
| 5 | EtCN | TBAB | 2 | 81 |
| 6 | $MeNO_2$ | TBAB | 2 | 68 |
| 7 | DMSO | TBAB | 2 | 47 |
| 8 | DMF | TBAB | 2 | 10 |

[a] Reaction was performed according to the general procedure.
[b] Number of Faradays per mol of **1a** supplied to the electrodes.
[c] Isolated yields, based on the added **1a**.

increased from 82% to 96% (Table 1, entries 1 and 2). TBACl and TEATFB as supporting electrolyte, the yields decreased (Table 1, entries 3 and 4). When the electrolysis was carried out in EtCN, $MeNO_2$, DMSO, and DMF solvents, the product **3aa**, after the usual work up, was isolated in 81%, 68%, 47%, and 10% yields, respectively (Table 1, entries 5–8). This indicates that the yield of **3aa** is related to the nature of the EGBs in these solvents. Thus, the satisfactory electrolytic conditions are the electrolyte consisting of MeCN and TBAB as well as 2 F mol$^{-1}$ of electricity. It should be pointed out that the adducts resulted from electron-deficient alkynes and amines were also obtained in the absence of the EGBs.

## 2.2. The optimization of reaction conditions for 3aa cyclized to 4aa

Without the pre-electrolysis of TBAB/DMF solution, it was found that the **3aa** was difficult to be transformed into the corresponding furan no matter what the reaction temperature was increased or decreased. When the galvanostatic-controlled pre-electrolysis of TBAB/DMF solution with Fe as anode and Pt as cathode at room temperature, the polysubstituted **4aa** was obtained in 24% yield (Table 2, entry 1). In this case, Fe$^{3+}$ ions could be electrogenerated in situ. So the formation of **4aa** may be related to the catalytic role of the Fe$^{3+}$ ions. However, when the same amount of $FeCl_3$ was directly added to the non-electrolyzed TBAB/DMF solution or $LiClO_4$ (instead of TBAB) was used as supporting electrolyte, **4aa** was not detected (Table 2, entries 2 and 3). The formation of **4aa** might be related to the formation of basic intermediates ($R_3N$ and $R^-$) resulting from the electroreduction of tetrabutylammonium cations ($R_4N^+$) at the cathode in DMF ($R_4N^+ + e^- \rightarrow R_4N\bullet$, $R_4N\bullet \rightarrow R\bullet + R_3N$, and $R\bullet + e^- \rightarrow R^-$).[31] Our experimental results (GC/MS analysis) confirmed the formation of $Bu_3N$. In addition, only addition of $FeCl_3$ and $Bu_3N$ to the non-electrolyzed TBAB/DMF solution, **4aa** was not obtained (Table 2, entry 4). It can be inferred that the electrogenerated alkyl anions $R^-$ may play an important role in the activation of **3aa** into **4aa**. The alkyl anion $R^-$ and Fe$^{3+}$ ions may synergically catalyze the cyclization process. When reaction temperature was increased to 60 °C, the yield of **4aa** could reach 63% (Table 2, entry 5). For some other electrogenerated metal ions (Cu$^{2+}$, Zn$^{2+}$, Al$^{3+}$), the yields (**4aa**) were 52%, 28%, and 13%, respectively (Table 2, entries 6–8). In comparison, Fe$^{3+}$ shows good catalytic activity. It should be mentioned that, under the electrogenerated Fe$^{3+}$ ions and alkyl anions $R^-$ as catalysts, the adducts originated from electron-deficient alkynes and primary amines were transformed into the corresponding pyrroles only when the reaction temperature was increased up to 100 °C. For secondary amines, the reaction required higher temperature (120 °C).

**Table 2**
The optimization of reaction conditions for **3aa** cyclized to **4aa**[a]

| Entry | Catalyst | Temperature (°C) | Yield of **4aa**[b] (%) |
|---|---|---|---|
| 1[c] | Fe$^{3+}$ | rt | 24 |
| 2[d] | $FeCl_3$ | rt | 0 |
| 3[e] | $FeCl_3$ | rt | 0 |
| 4[f] | $FeCl_3$ | rt | 0 |
| 5[g] | Fe$^{3+}$ | 60 | 63 |
| 6 | Cu$^{2+}$ | 60 | 52 |
| 7 | Zn$^{2+}$ | 60 | 28 |
| 8 | Al$^{3+}$ | 60 | 13 |

[a] Reaction was performed according to the general procedure.
[b] Isolated yields, based on the added electron-deficient alkynes.
[c] Fe$^{3+}$ was electrogenerated in situ.
[d] $FeCl_3$ was added to the non-electrolyzed TBAB/DMF solution.
[e] $LiClO_4$ was used as a supporting electrolyte.
[f] $FeCl_3$ and $Bu_3N$ were added to the electrolyte without electrolysis.
[g] Metal ions were generated in an undivided cell containing TBAB/DMF electrolyte with Pt cathode (4 cm$^2$) and Fe anode. After 1 F mol$^{-1}$ of starting electron-deficient alkynes, the electrolysis was stopped.

During the pre-electrolysis of $n$-$Bu_4NBr$/DMF solution, the electroreduction of $Bu_4N^+$ eventually produces $Bu_3N$ and $R^-$. We deduce that $\mathbf{R}^-$ may exist in the form of $RFeBr_2$ because Fe$^{3+}$ ions generated at Fe anode can react with alkyl anions $R^-$ and $Br^-$ ($R^- + Fe^{3+} + 2Br^- = RFeBr_2$) to keep electrical neutrality of the electrolyte. In order to confirm the formation of $R^-$, the pre-electrolyzed TBAB/DMF solution containing the electrogenerated Fe$^{3+}$ was distilled to remove DMF at reduced pressure. It was found that the reaction of the obtained residue with water could produce the gas. Also, the gas was confirmed to be $n$-butane by GC/MS analysis.

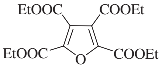
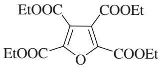
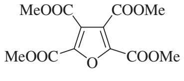
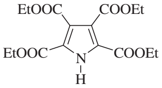
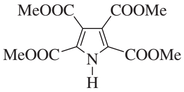
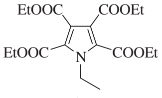
## 2.3. Synthesis of other symmetrical heterocycles

On the basis of the above optimized conditions, the scope of the cyclization reaction was further examined. The reactions of **1a** with propan-1-ol (**2a**) or ethanol (**2b**) could afford tetrasubstituted furan (**4aa**) in 63 and 51% yields, respectively (Table 3, entries 1 and 2). Similarly, the reaction of dimethylbut-2-ynedioate (**1b**) with **2a**, the homogenous product (**4ba**) was obtained in 53% yield (Table 3, entry 3). Interestingly, with **1a** and **2c** (amine) as substrates, the pyrrole product (**4ac**) could be obtained in 40% yield (Table 3, entry 4). The reaction of **1b** with **2c** was examined and the product (**4bc**) was obtained in 42% yield (Table 3, entry 5). With secondary amine (**2d**) and **1a** as substrates, **4ad** is also obtained in 36% yield (Table 3, entry 6).

## 2.4. The reaction mechanism

The reaction mechanism was proposed in Scheme 2. Firstly, the EGB promotes the nucleoaddition of **2a** with **1a** to produce the

**Table 3**
Synthesis of other symmetrical heterocycles[a]

| Entry | Alkyne | Alcohol/amine | Product | Yield[b] (%) |
|---|---|---|---|---|
| 1 | **1a** | $CH_3CH_2CH_2OH$<br>**2a** | <br>**4aa** | 63 |
| 2 | **1a** | $CH_3CH_2OH$<br>**2b** | <br>**4aa** | 51 |
| 3 | **1b** | $CH_3CH_2CH_2OH$<br>**2a** | <br>**4ba** | 53 |
| 4 | **1a** | $CH_3CH_2CH_2NH_2$<br>**2c** | <br>**4ac** | 40 |
| 5 | **1b** | $CH_3CH_2CH_2NH_2$<br>**2c** | <br>**4bc** | 42 |
| 6 | **1a** | $HN(CH_2CH_3)_2$<br>**2d** | <br>**4ad** | 36 |

Compound **1a** is diethylbut-2-ynedioate and **1b** is dimethylbut-2-ynedioate.

[a] Reaction was performed according to the general procedure.

[b] Isolated yields, based on the added electron-deficient alkynes.

adduct **3aa**. Secondly, during the electrogeneration of $Fe^{3+}$ ions in the TBAB/DMF electrolyte, tetrabutylammonium cations $R_4N^+$ at the cathode could accept electrons to produce tributylamine $R_3N$ and the alkyl radical R•. The alkyl radical R• further accepts electrons to produce the alkyl anion $\mathbf{R}^-$ (carbanion, a strong base).[31] When **3aa** was added to the pre-electrolysis of TBAB/DMF solution, **3aa** could be divided into four intermediates (**A**, **B**, **C**, and **D**) on the influence of the alkyl anion $\mathbf{R}^-$ under appropriate condition. **E** is formed via the combination of **A** with **B**. **C** can react with **D** and produce *n*-propyl ether (confirmed by GC/MS analysis). Under the synergic role of $Fe^{3+}$ ions and $\mathbf{R}^-$ alkyl anions, **E** is activated and deprotonated by alkyl anions $\mathbf{R}^-$.[28,31,32] Subsequently, **F** undergoes through an intramolecular ring-closing reaction in itself and produces **G**. With the assistance of $Fe^{3+}$ ions catalyst, **G** further releases a proton to the alkyl anion $\mathbf{R}^-$ and affords the product (**4aa**).

## 3. Experimental section for the synthesis of symmetrical oxygen- and nitrogen-heterocycles from electron-deficient alkynes and alcohol or amines

### 3.1. Chemicals

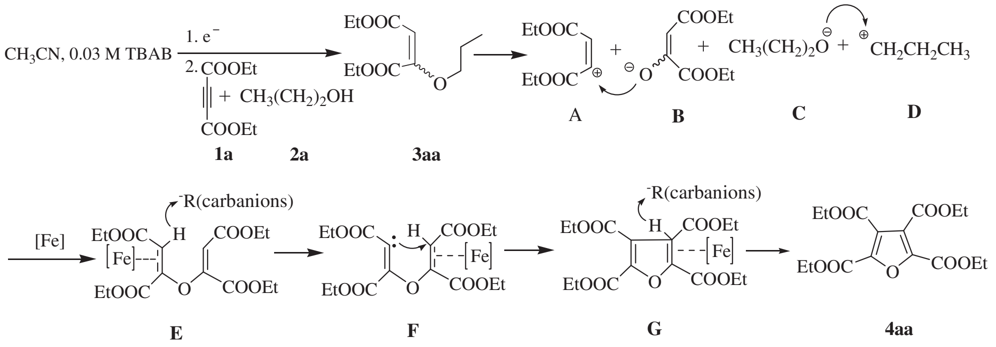
*N*,*N*-Dimethylformamide (DMF) and MeCN were kept over anhydrous $MgSO_4$ for several days. EtCN and $MeNO_2$ were distilled before use; Dimethylsulphoxide (DMSO) was used without any purification. Tetrabutylammonium chloride (TBACl), tetrabutylammonium bromide (TBAB), and tetraethylammonium tetrafluoroborate (TEATFB) were dried at 60 °C under vacuum for 10 h. The electron-deficient alkynes were purchased from Alfa Aesar without further purification.

### 3.2. Instrumentation

FTIR spectra were measured by a TENSOR27 spectrometer. $^1H$ NMR and $^{13}C$ NMR were determined on a Bruker DRX-400 spectrometer with $CDCl_3$ as the solvent in the presence of $SiMe_4$ as an internal standard. Mass spectral analyses were done on a Shimadzu QP5050A spectrometer.

### 3.3. General procedure

Electrolysis was performed in a divided cell fitted with Pt electrodes with the solvent-supporting electrolyte solution (TBAB/MeCN (0.03 M), 30 mL) with $N_2$ protection under galvanostatic control ($J$=15 mA $cm^{-2}$) at room temperature. At the end of the electrolysis, electron-deficient alkyne (1.0 mmol) and alcohol (2.0 mmol) were added to the catholyte. The mixture solution was continuously stirred for 1 h under $N_2$ protection. Then the solvent was evaporated under reduced pressure. The crude adduct was transferred into three-neck round-bottom flask equipped with a magnetic stirrer. Subsequently, TBAB/DMF solution containing $Fe^{3+}$ ions electrogenerated in situ at the iron anode were added. The reaction solution was heated to reflux for 10 h with $N_2$ protection. After the completion of the reaction, the mixtures were poured into water and extracted with diethyl ether (3×30 mL). After the evaporation of ether, the obtained crude product was purified by thin layer chromatography, using petroleum ether/ethyl acetate 10:1 as eluent.



**Scheme 2.** The plausible reaction mechanism for the synthesis of **4aa**.

*3.3.1. (Z) Diethyl 2-propoxymaleate (**3aa**).* IR (KBr): $\nu$ 2990, 1716, 1618, 1463, 1095 cm$^{-1}$. $^1$H NMR (CDCl$_3$, 400 Hz): $\delta$ 5.34 (s, 1H), 4.36–4.43 (m, 4H), 4.06 (m, 2H), 1.62 (m, 2H), 1.25–1.36 (m, 6H), 0.88 (t, $J$=7.2 Hz, 3H). $^{13}$C NMR (CDCl$_3$, 100 Hz): $\delta$ 165.1, 162.7, 159.7, 104.3, 66.3, 61.4, 60.3, 23.4, 13.5, 13.2, 10.8. MS (EI): $m/z$ 230 (M$^+$).

*3.3.2. (E) Diethyl 2-propoxymaleate (**3aa**).* IR (KBr): $\nu$ 2991, 1716, 1620, 1467, 1094 cm$^{-1}$. $^1$H NMR (CDCl$_3$, 400 Hz): $\delta$ 5.36 (s, 1H), 4.38–4.47 (m, 4H), 4.07 (m, 2H), 1.62 (m, 2H), 1.26–1.40 (m, 6H), 0.89 (t, $J$=7.2 Hz, 3H). $^{13}$C NMR (CDCl$_3$, 100 Hz): $\delta$ 165.3, 162.8, 159.1, 104.0, 66.9, 61.6, 60.1, 23.6, 13.5, 13.3, 10.6. MS (EI): $m/z$ 230 (M$^+$).

*3.3.3. Tetraethyl furan-2,3,4,5-tetracarboxylate (**4aa**).* IR (KBr): $\nu$ 2994, 1718, 1622, 1560, 1089 cm$^{-1}$. $^1$H NMR (CDCl$_3$, 400 Hz): $\delta$ 4.36–4.43 (m, 8H), 1.35–1.40 (m, 12H). $^{13}$C NMR (CDCl$_3$, 100 Hz): $\delta$ 160.7, 156.7, 143.7, 124.3, 62.5, 62.2, 14.1, 13.9. MS (EI): $m/z$ 356 (M$^+$).

*3.3.4. Tetramethyl furan-2,3,4,5-tetracarboxylate (**4ba**).* IR (KBr): $\nu$ 2982, 1720, 1601, 1579, 1136 cm$^{-1}$. $^1$H NMR (CDCl$_3$, 400 Hz): $\delta$ 3.93 (s, 6H), 3.96 (s, 6H). $^{13}$C NMR (CDCl$_3$, 100 Hz): $\delta$ 160.1, 156.9, 143.7, 124.3, 52.9, 49.9. MS (EI): $m/z$ 300 (M$^+$).

*3.3.5. Tetraethyl1H-pyrrole-2,3,4,5-tetracarboxylate (**4ac**).* IR (KBr): $\nu$ 2990, 1708, 1612, 1520, 1069 cm$^{-1}$. $^1$H NMR (CDCl$_3$, 400 Hz): $\delta$ 8.08 (s, 1H), 4.27–4.44 (m, 8H), 1.32–1.41 (m, 12H). $^{13}$C NMR (CDCl$_3$, 100 Hz): $\delta$ 166.5, 161.6, 126.7, 123.5, 62.4, 61.2, 14.2, 14.0. MS (EI): $m/z$ 355 (M$^+$).

*3.3.6. Tetramethyl1H-pyrrole-2,3,4,5-tetracarboxylate (**4bc**).* IR (KBr): $\nu$ 2988, 1708, 1610, 1575, 1138 cm$^{-1}$. $^1$H NMR (CDCl$_3$, 400 Hz): $\delta$ 8.78 (s, 1H), 3.98 (s, 6H), 3.95 (s, 6H). $^{13}$C NMR (CDCl$_3$, 100 Hz): $\delta$ 166.1, 160.3, 126.7, 123.3, 52.5, 51.9. MS (EI): $m/z$ 299 (M$^+$).

*3.3.7. Tetraethyl1-ethyl-1H-pyrrole-2,3,4,5-tetracarboxylate (**4ad**).* IR (KBr): $\nu$ 2995, 1718, 1630, 1515, 1065 cm$^{-1}$. $^1$H NMR (CDCl$_3$, 400 Hz): $\delta$ 4.27–4.44 (m, 8H), 3.89 (q, $J$=7.2 Hz, 2H), 1.32–1.41 (m, 12H), 1.52 (t, $J$=7.2 Hz, 3H). $^{13}$C NMR (CDCl$_3$, 100 Hz): $\delta$ 166.2, 160.7, 134.7, 116.7, 64.8, 63.1, 29.1, 16.2, 14.5, 14.1. MS (EI): $m/z$ 383 (M$^+$).

## 4. Conclusions

In conclusion, from the electrochemical point of view, we have explored the domino reactions, to synthesize the oxygen- and nitrogen-heterocycles from electron-deficient alkynes with alcohol or amines. It was found that the synergic effect of electrogenerated Fe$^{3+}$ ions and **R**$^-$ alkyl anions play an important role in the reactions. The present approach represents a valuable and competitive alternative procedure for the synthesis of heterocycles.

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## Supplementary data

Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.tet.2011.04.058. These data include MOL file and InChiKey of the most important compounds described in this article.

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