

Electrochemical Synthesis Of Quinoxalinediones: 1,4-di(2-Pyridylmethyl)-1,2,3,4-Tetrahydroquinoxaline-6,7-Dione

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Received: 26 July 2012 / Accepted: 31 August 2012 / Published: 1 October 2012

1,4-Di(2-pyridylmethyl)-1,2,3,4-tetrahydroquinoxaline-6,7-dione (**6a**) was synthesized via the electrooxidation of catechol (**1a**) in the presence of *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**) in aqueous solution. A reaction mechanism was proposed base on the result of cyclic voltammetry and controlled-potential coulometry. The electrochemical synthesis of compound (**6a**) was performed successfully at a carbon rod electrode in an undivided cell in good yield and with high product purity.

Keywords: Catechol; Michael reaction; *N*¹,*N*²-di(2-pyridylmethyl)ethylenediamine; Schiff base; Nitrogen-containing heterocycles.

1. INTRODUCTION

Nitrogen-containing heterocycles are abundant in nature and exhibit diverse and important biological properties [1-5]. Functionalized quinoxalines represent an important class of nitrogen-containing heterocycles because they play a significant role as a basic skeleton for the design of a number of antibiotics, such as echinomycin, actinomycin, and leromycin [6,7]. Quinoxaline derivatives are known to have an affinity for quisqualate receptors, and because of this affinity, they are suitable as pharmaceutical agents for the treatment of diseases of the central nervous system [8]. The quinoxaline ring is also a constituent of many pharmacologically and biologically active compounds, such as insecticides, fungicides, herbicides, and anthelmintics [9,10]. In addition, they exhibit well-known biological activities, including anti-viral, anti-bacterial, anti-inflammatory, anti-protozoal, anthelmintic, and anti-cancer activities, and they are also known as kinase inhibitors [11,12]. Consequently, these compounds are useful intermediates in organic syntheses [13]. Quinoxaline

derivatives are used application in dyes [14], organic semiconductors [15], electron luminescent materials [16], and chemically controllable switches [17]; they are also useful as building blocks for the synthesis of anion receptors [18], cavitands [19], dehydroannulenes [20], and DNA cleaving agents [21]. Furthermore, they serve as useful rigid subunits in macrocyclic receptors or in molecular recognition [22]. The development of an effective method for the electrochemical synthesis of quinoxalines is still an important challenge. A number of synthetic strategies have been developed for the preparation of substituted quinoxalines [23-28].

To the best of our knowledge, the literature contains no report of the synthesis of these compounds via an electrochemical synthesis method.

2. EXPERIMENTAL PART

2.1. Apparatus

Cyclic voltammetry and controlled-potential coulometry were performed using an Autolab model PGSTAT 302N potentiostat/galvanostat. The working electrode used in the voltammetry experiments was a glassy carbon disc (1.8 mm in diameter), and platinum wire was used as a counter electrode (CE). The working electrode potentials were measured versus a saturated calomel electrode (SCE). The glassy carbon was polished with a polishing cloth before each measurement. All electrodes for the CV experiments were acquired from France Radiometer Analytical. The working electrode used in the controlled-potential coulometry was an assembly of 12 carbon rods (6 mm in diameter and 110 mm in length), and a large piece of platinum gauze (25 mm in width and 50 mm in length) constituted the CE. A magnetic stirrer was used during electrolysis. The cell used was a simple and undivided cell [3].

2.2. Reagents

The catechols (catechol, 4-*tert*-butylcatechol) were reagent-grade materials from Aldrich. The KH_2PO_4 , K_2HPO_4 and other acids and bases were of pro-analysis grade from E. Merck. 2-Pyridinecarboxaldehyde and ethylenediamine were purchased from Aldrich. All of these chemicals were used without further purification.

2.3. Organic synthesis of N^1, N^2 -di(2-pyridylmethyl)-ethylenediamine (3)

2-Pyridinecarboxaldehyde (2.14 g, 20 mmol) and ethylenediamine (0.60 g, 10 mmol) were mixed in a beaker with 100 ml of MeOH. The obtained mixture was then stirred overnight at room temperature, and sodium borohydride (3.02 g, 80 mmol) was subsequently added. The mixture was refluxed for 3 hours, was allowed to cool and was poured into 250 ml of H_2O . The solution was filtered and evaporated to dryness. The residue was then extracted with water-chloroform (1:3). The organic layer was dried over anhydrous Na_2SO_4 and was subsequently evaporated to yield a brownish-

yellow oil [29-34]. The product (**3**) was characterized as a pure compound using ^1H NMR, ^{13}C NMR, ESI-MS² and IR.

2.4. Electro-organic synthesis of **6a**

An aqueous solution of phosphate buffer (pH 7.0, 0.20 M) in water that contained 2 mmol of catechol (**1a**) and N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) (2 mmol) was electrolyzed at 0.3 V versus SCE in an undivided cell equipped with a graphite anode (an assembly of twelve rods, 6 mm in diameter and 11 cm in length) and a large platinum gauze cathode at room temperature under a constant current density of 2 mA/cm². The process was interrupted during the electrolysis, and the graphite anode was reactivated by being washed in acetone. At the end of the electrolysis, the solution was extracted with dichloromethane and was dried with sodium sulfate. The crude product was recrystallized from a mixture of dichloromethane and *n*-hexane. After purification, the product was characterized by ^1H NMR, ^{13}C NMR, ESI-MS² and IR.

2.5. Characterization of products (**3** and **6a**)

2.5.1. N^1,N^2 -Di(2-pyridylmethyl)-ethylenediamine ($\text{C}_{14}\text{H}_{18}\text{N}_4$) (**3**)

Isolated yield = 77.6%, ^1H NMR, δ ppm (600 MHz CDCl_3): 2.68 (t, 4H), 3.78 (s, 2H), 4.76 (s, 2H), 7.02 (t, 2H), 7.18 (d, 2H), 7.75 (t, 2H), 8.41 (m, 2H). ^{13}C NMR, δ ppm (150 MHz CDCl_3): 48.8, 54.9, 121.8, 122.2, 136.4, 149.1, 159.7. ESI-MS²: m/z , 243.1 ($\text{M}^+ + 1$). IR_(KBr): 3394, 2937, 2845, 2202, 1655, 1594, 1570, 1475, 1436, 1358, 1304, 1150, 1113, 1050, 1001, 762 and 631 cm^{-1} .

2.5.2. 1,4-Di(2-pyridylmethyl)-1,2,3,4-tetrahydroquinoxaline-6,7-dione ($\text{C}_{20}\text{H}_{18}\text{N}_4\text{O}_2$) (**6a**)

Isolated yield = 89.3%, ^1H NMR, δ ppm (600 MHz CDCl_3): 3.56 (s, 4H), 4.55 (s, 4H), 5.60 (s, 2H), 7.25 (t, 4H), 7.33 (t, 2H), 7.39 (t, 2H). ^{13}C NMR, δ ppm (150 MHz DMSO-d_6): 47.9, 56.0, 98.8, 127.2, 127.9, 129.2, 135.9, 149.7, 178.3. ESI-MS²: m/z , 345.1 ($\text{M}^+ - 1$). IR_(KBr): 3435, 3025, 2929, 1596, 1542, 1495, 1451, 1442, 1391, 1365, 1323, 1301, 1246, 1195, 1091, 1077, 1027, 917, 786, 748, 735, 703 and 464 cm^{-1} .

3. RESULTS AND DISCUSSION

3.1. Chemical synthesis of N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**)

The chemical synthesis of the nucleophile (**3**) was achieved using a Schiff base chemical reaction between 2-pyridinecarboxaldehyde and ethylenediamine in methanol solution under reflux conditions. The synthesis of Schiff bases is often performed using a catalyst and is typically conducted by refluxing a mixture of aldehyde (or ketone) and amine [35,36].

The Schiff base reaction for the preparation of N^1, N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) can be represented with a two-step mechanism. The first step in the reaction is reversible and progresses through a carbinolamine intermediate. This step requires the removal of water, often by azeotropic distillation with benzene, to achieve high yields. The reaction is acid-catalyzed, but catalysts are not typically required when aliphatic amines are involved. The second step of the reaction involves the reduction of the resulting compound, which contains two azomethine groups (C=N), to nucleophile **3** using sodium borohydride (NaBH_4) [37-44]. According to our results, the analytical and spectral data are consistent with the proposed formulation.

3.2. Electrochemical oxidation of catechol (**1a**) in the presence of (**3**)

3.2.1. Diagnostic criteria

A cyclic voltammogram of an aqueous solution of 2.0 mM catechol (**1a**) that contained phosphate buffer (pH 7.0, $c = 0.2$ M) showed an anodic peak (A_1) in the positive scan and a cathodic counterpart peak (C_1) in the negative scan.

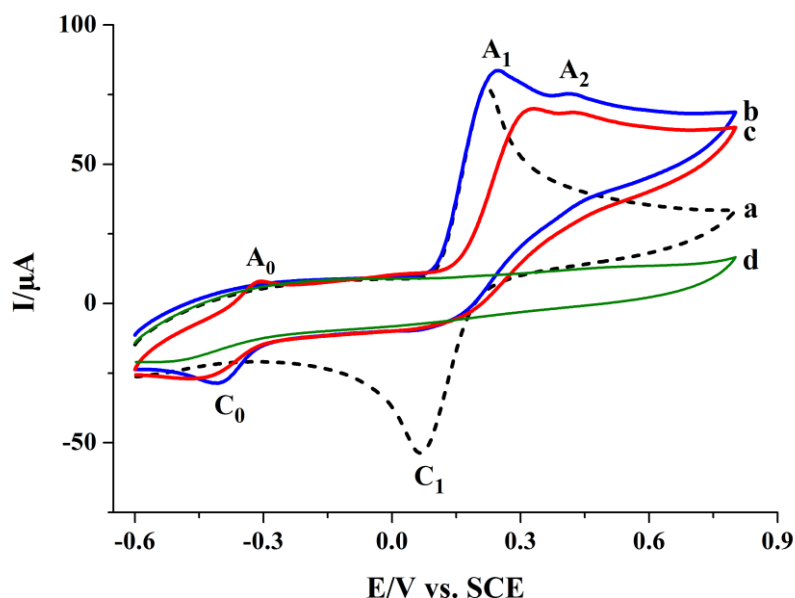


Figure 1. Cyclic voltammograms of: (a) 2.0 mM catechol (**1a**); (b and c) first and second cycles of 2.0 mM catechol (**1a**) in the presence of 2.0 mM N^1, N^2 -di(2-pyridylmethyl)-ethylenediamine (DP MEDA) (**3**), respectively; (d) 2.0 mM N^1, N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**), at a glassy carbon electrode (1.8 mm in diameter) in the 0.2 M phosphate buffer solution ($\text{KH}_2\text{PO}_4/\text{K}_2\text{HPO}_4$) pH=7. Scan rate: 50 mVs^{-1} ; $T=25 \pm 1$ °C.

These peaks correspond to the transformation of catechol (**1a**) to *o*-benzoquinone (**2a**) and vice-versa within a quasi-reversible two-electron process (Fig. 1, curve (a)) [45,46], i.e., any hydroxylation [47] or dimerization [48] reactions are too slow to be observed on the time scale of cyclic voltammetry. The oxidation of catechol (**1a**) in the presence of N^1, N^2 -di(2-pyridylmethyl)-

ethylenediamine (**3**) as a nucleophile was studied in some detail. Figure 1 (curve b) shows the cyclic voltammogram obtained for a 2 mM solution of catechol **1a** in the presence of N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) (2 mM) in an aqueous solution that contained 0.2 M phosphate buffer (pH 7.0). As evident in Figure 1, the cathodic peak C_1 disappeared completely, and a new anodic peak (A_2) appeared at more positive potentials. Additionally, the voltammogram from the negative scan, exhibits a new cathodic peak (C_0) at -0.40 V vs. SCE. In the second cycle, a new anodic peak (A_0) appeared with an E_p value of -0.31 V vs. SCE. This peak is related to the oxidation of intermediate **5a**. Furthermore, the height of the C_1 peak increases proportionally with the augmentation of the potential sweep rate (Fig. 2).

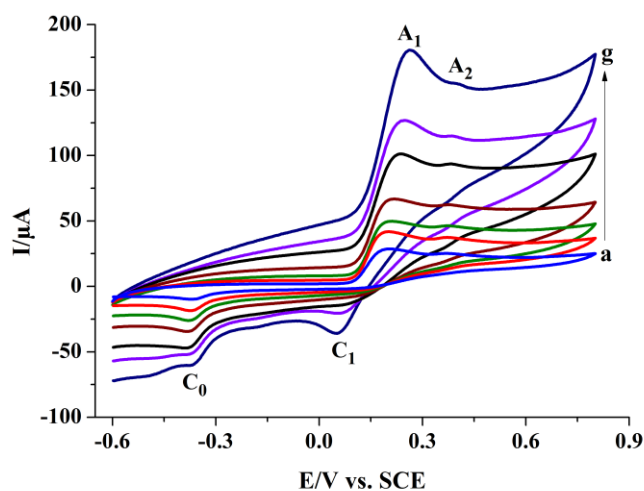


Figure 2. Typical cyclic voltammograms of 1.0 mM catechol (**1a**) in the presence of 1.0 mM N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) in water that contained 0.2 M phosphates ($\text{KH}_2\text{PO}_4/\text{K}_2\text{HPO}_4$) as the buffer and supporting electrolyte (pH=7); at a glassy carbon electrode (1.8 mm in diameter) and at various scan rates. Scan rate from (a) to (g) are 10, 25, 50, 100, 250, 500 and 1000 mVs^{-1} , respectively. $T=25 \pm 1^\circ\text{C}$

A similar situation was observed when the ratio between N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) and **1a** was decreased, i.e., the variation of the peak current ratio (I_P^{C1} / I_P^{A1}) versus the scan rate for a mixture of catechol (**1a**) and N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) confirms the reactivity of **2a** toward N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine. This reactivity appeared as an increase in the (I_P^{C1} / I_P^{A1}) ratio at higher scan rates.

3.2.2. Effect of pH

The cyclic voltammograms of 1.0 mM catechol (**1a**) in the presence of 1.0 mM N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) at various pH levels are shown in Fig. 3. As shown, as the pH was increased, the height of the anodic peak (A_1) increased, the height of the cathodic peak (C_1) decreased and a new cathodic peak (C_2) appeared at more negative potentials.

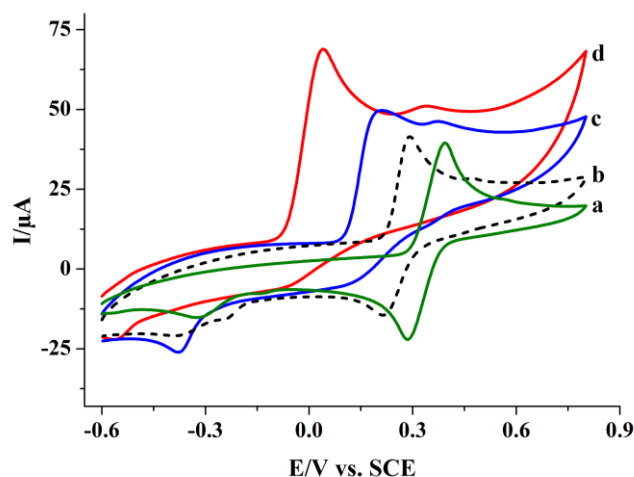


Figure 3. Cyclic voltammograms of 1.0 mM catechol in the presence of 1.0 mM N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine in buffered solutions with various pH levels. pH from (a) to (d) are 3, 5, 7 and 10, respectively. Ionic strength; 0.2 M. Scan rate: 50 mVs^{-1} ; $T=25 \pm 1 \text{ }^\circ\text{C}$.

This behavior is related to the participation of the produced **2a** in the Michael addition reaction with DPMEDA (**3**) that led to formation of the product at more negative oxidation potentials. The rate of this intermolecular Michael addition increased with increasing pH. As shown in Fig. 3, the desired reaction was adequately fast at neutral pH levels. The height of the cathodic peaks also showed that the reaction occurred in the transition region between the diffusion and kinetic situations and was suitable for voltammetric study. The solution that contained phosphate buffer (pH 7.0, $c = 0.2 \text{ M}$) was selected as a medium for a detailed electrochemical study.

3.2.3. Coulometry

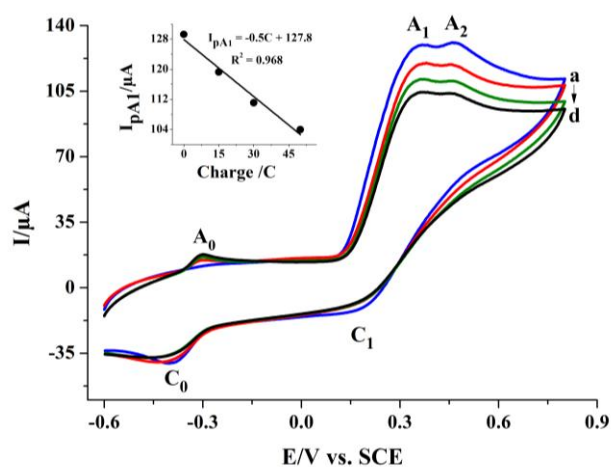
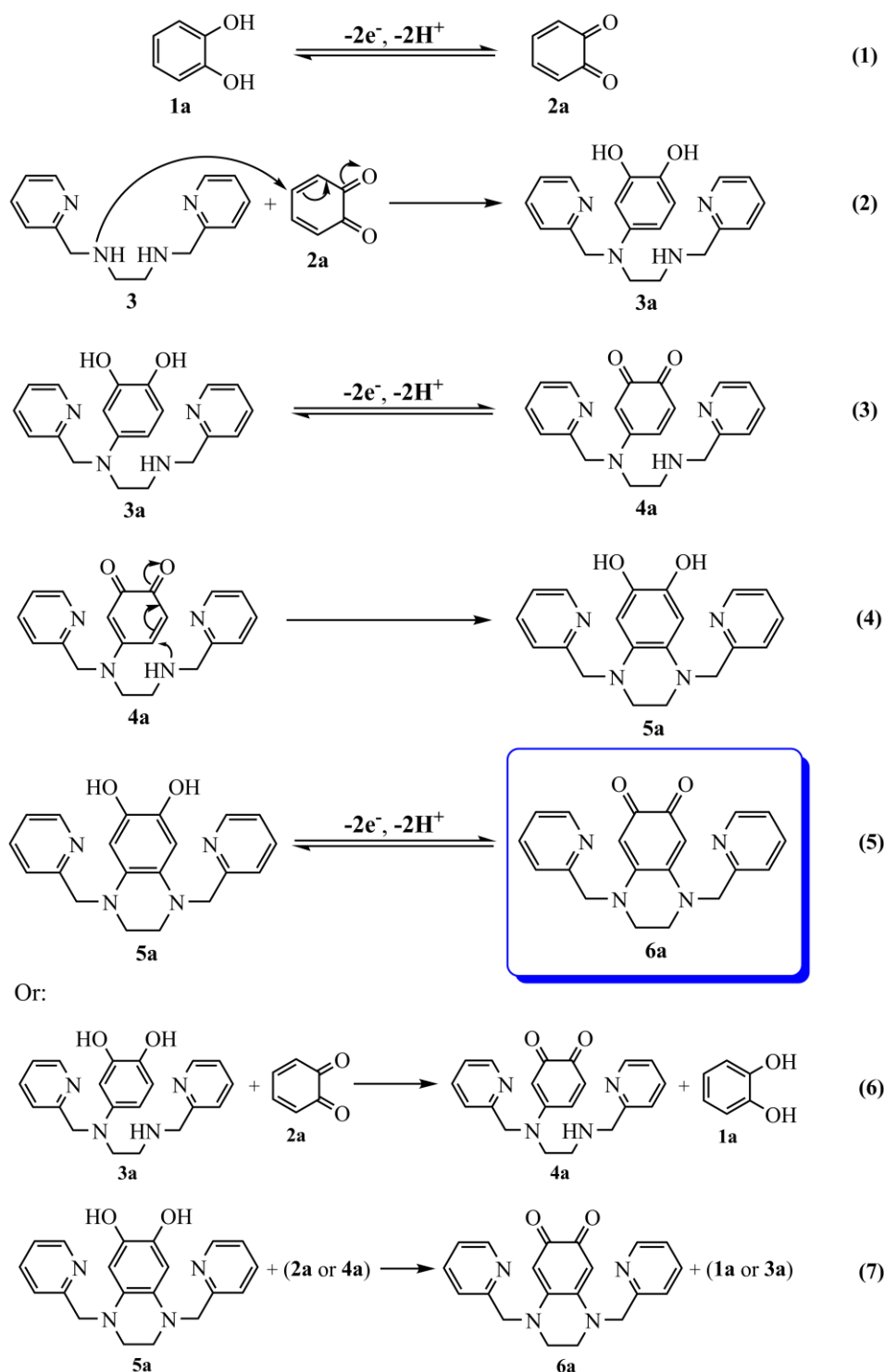


Figure 4. Cyclic voltammograms of 0.4 mmol catechol (**1a**) in the presence of 0.4 mmol N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) in 0.2 M phosphates ($\text{KH}_2\text{PO}_4/\text{K}_2\text{HPO}_4$) buffer solution during controlled-potential coulometry at 0.3 V vs. SCE: (a) before, (b) to (d) during the coulometry. Inset: variation of A_1 peak current versus consume charge. Scan rate: 50 mVs^{-1} . $T=25 \pm 1 \text{ }^\circ\text{C}$.



Scheme 1. Proposed mechanism for the electrooxidation of catechol (**1a**) in the presence of N,N' -di(2-pyridylmethyl)-ethylenediamine (**3**).

Controlled-potential coulometry was performed in an aqueous solution that contained 0.2 M phosphate buffer (pH 7), 0.4 mmol of **1a**, and 0.4 mmol of **3** at 0.3 V vs. SCE. The monitoring of the electrolysis progress was performed by cyclic voltammetry (Fig. 4). All of the anodic and cathodic peaks decreased and disappeared when the charge consumption became approximately $6e^-$ per

molecule of **1a** (Fig. 4. inset) in proportion to the advancement of coulometry [49]. These observations allow us to propose the *ECECE* pathway illustrated in Scheme 1 for the electrooxidation of **1a** in the presence of *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**). According to the obtained results, the Michael addition reaction of *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**) to *o*-quinone (**2a**) (Scheme 1, Eq. (2)) is faster than other secondary reactions and leads to the adduct **3a**. The adduct **3a** then undergoes an abstraction of a second pair of electrons, which leads to *o*-benzoquinone **4a**. The intramolecular addition of **3** to **4a** leads to the formation of the quinoxalinediol derivative **5a** and further oxidation of this compound produces the final product **6a**. The overall reaction mechanism for electrochemical oxidation of catechol in the presence of *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**) as a nucleophile is presented in Scheme 1.

In addition, the oxidation of **3a** and **5a** may take place through a solution electron transfer (SET) reaction (Scheme 1, Eqs. (6) and (7)).

3.3. Electrochemical oxidation of 4-*tert*-butylcatechol (**1b**) in the presence of **3**

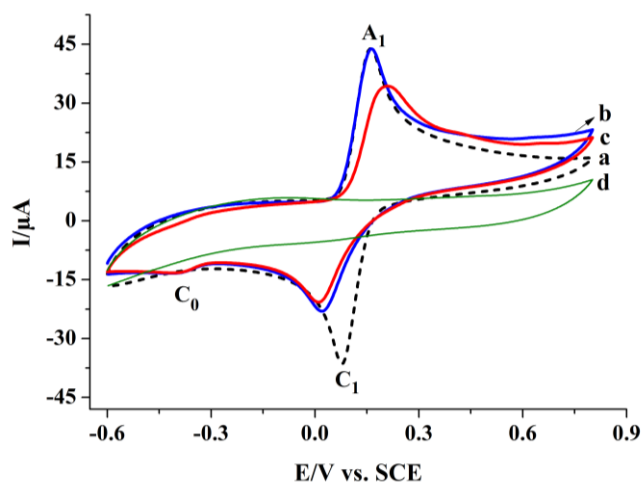


Figure 5. Cyclic voltammograms 2.0 mM 4-*tert*-butylcatechol (**1b**): (a) in the absence and (b) in the presence of 2.0 mM *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**); (c) second cycle of 2.0 mM **1b** in the presence of 2.0 mM DPMEDEA; and (d) 2.0 mM *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**), at a glassy carbon electrode in aqueous solution. Supporting electrolyte 0.2 M phosphate buffer (KH₂PO₄/K₂HPO₄); scan rate: 25 mV s⁻¹; T=25 ± 1 °C.

The electrochemical oxidation of 4-*tert*-butylcatechol (**1b**) in the presence of *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**) as a nucleophile was studied in phosphate buffer (pH 7.0, *c* = 0.2 M) to determine the effects of the presence of a group in a reactive site of the catechol ring. The reaction was investigated in a manner similar to that used for **1a** (Fig. 5). Figure 5 (curve b) shows the cyclic voltammogram of **1b** in the presence of **3**. As evident in Fig. 5, the voltammogram exhibits one anodic peak (A₁) at 0.16 V vs. SCE. During the reverse scan, two cathodic peaks (C₁ and C₀) appeared at *E*_p values of 0.08 and -0.38 V vs. SCE, respectively.

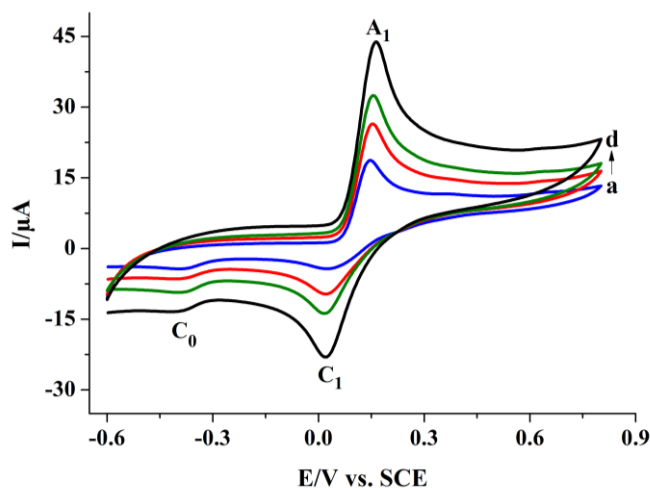
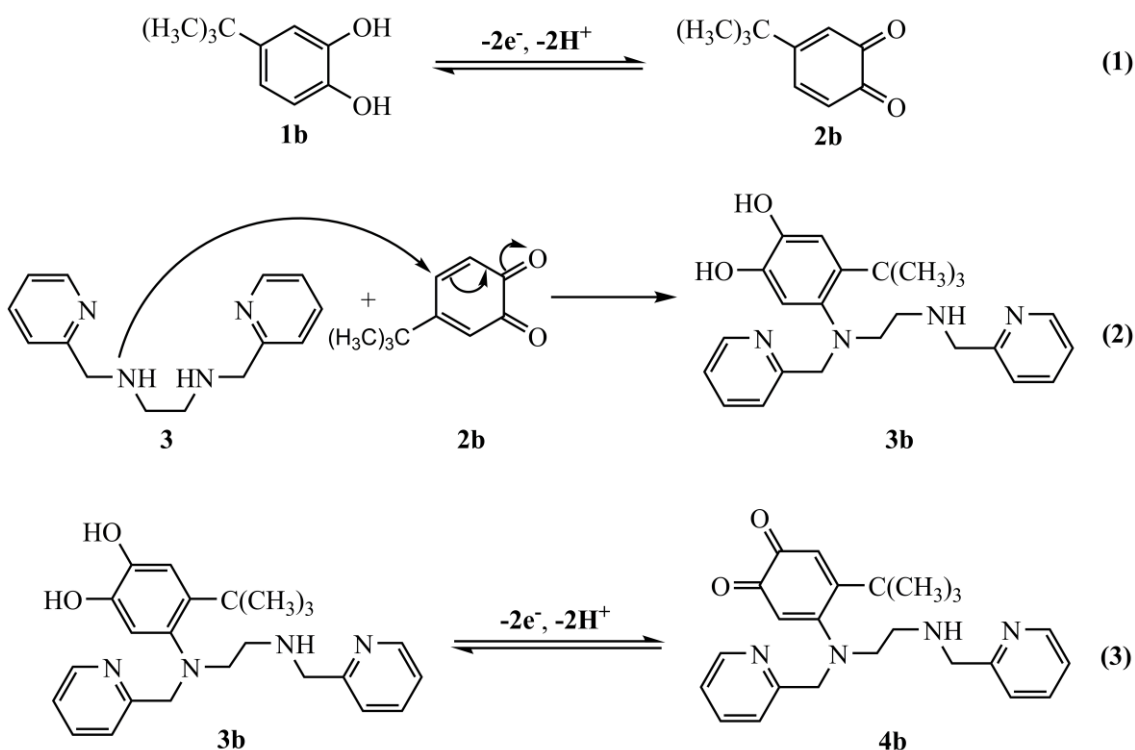


Figure 6. Cyclic voltammograms of 4-*tert*-butylcatechol (2.0 mM) (**1b**) in the presence of 2.0 mM N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**) in water containing of 0.2 M phosphates ($\text{KH}_2\text{PO}_4/\text{K}_2\text{HPO}_4$) as the buffer and supporting electrolyte (pH=7); at a glassy carbon electrode (1.8 mm diameter) and at various scan rates. Scan rate from (a) to (d) are: 5, 10, 15, and 25 mVs^{-1} , respectively. $T=25 \pm 1^\circ\text{C}$.



Scheme 2. Proposed mechanism for the electrooxidation of 4-*tert*-butylcatechol (**1b**) in the presence of N^1,N^2 -di(2-pyridylmethyl)-ethylenediamine (**3**).

The anodic peak A_1 corresponds to the oxidation of 4-*tert*-butylcatechol (**1b**) to *o*-benzoquinone **2b** and vice-versa [50,51]. Obviously, the cathodic peaks C_1 and C_0 are also related to the reduction of **2b** and 4-*tert*-butyl-5-((2-pyridylmethyl)(2-(2-pyridylmethyl-amino)ethyl)amino)cycl-

ohexa-3,5-diene-1,2-dione (C₂₄H₂₈N₄O₂) (**4b**) to 4-*tert*-butylcatechol (**1b**) and 4-*tert*-butyl-5-((2-pyridylmethyl)(2-(2-pyridylmethylamino)ethyl)amino)benzene-1,2-diol (C₂₄H₃₀N₄O₂) (**3b**), respectively. In the presence of **3**, the cathodic peak for the reduction of **2b** and **4b** increased significantly at higher scan rates (Fig. 6). According to the obtained data, we propose an *ECE* mechanism for the electro-oxidation of **1b** in the presence of *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**) (Scheme 2).

4. CONCLUSIONS

The results of this work indicate that catechols (**1a,1b**) are oxidized in aqueous media to their respective *o*-benzoquinones. The benzoquinone is then attacked by *N*¹,*N*²-di(2-pyridylmethyl)-ethylenediamine (**3**) at the uinine to produce the adducts **3a** and **3b**. The adducts **3a** and **3b** then undergo abstraction of a second pair of electrons, which leads to *o*-benzoquinones **4a** and **4b**. The intramolecular addition of **3** to **4a** leads to the formation of the adduct **5a** and then the oxidation of **5a** produces the final product **6a**. According to our results, the Michael reaction of these nucleophiles to form *o*-quinones leads to the formation of new quinoxalinediones as the final products. The reaction to prepare 1,4-di(2-pyridylmethyl)-1,2,3,4-tetrahydroquinoxalin-e-6,7-dione (**6a**) follows the *ECECE* mechanism, whereas, in the case of 4-*tert*-butylcatechol (**1b**), the reaction mechanism is *ECE* because the presence of a *tert*-butyl group in the structure of respective uinine (**2b**) prohibits the second chemical reaction. The synthesis of new nucleophiles and, consequently, new hydroquinoxalines is possible through changing the primary amines with active carbonyls.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support of this work by the Universiti Kebangsaan Malaysia (UKM). This research was supported by the Research University Grants, UKM-DIP-2012-22 (Sintesis dan Katalisis), and UKM-DLP-2012-024.

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