A Facile and Efficient One-Pot Electrochemical Synthesis of Thiazole Derivatives in Aqueous Solution

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In the present work, the electrooxidation of hydroquinones **1a** and **1b**, and catechols **1c** and **1d** was studied in the presence of rhodanine (**3**) as nucleophile in a mixture of EtOH and phosphate buffer solution as 'green' media using cyclic voltammetry and controlled-potential coulometry. The results indicated that the corresponding *p*- and *o*-quinones formed from the hydroquinones and catechols, respectively, participate in *Michael* addition reaction to yield new thiazole derivatives. The electrochemical syntheses of these new thiazole derivatives were performed successfully at three graphite rod electrodes in undivided cells in good-to-excellent yields at room temperature without any catalyst.

1. Introduction. – Thiazole and its derivatives constitute an important class of heterocyclic compounds with remarkable biological features, such as antimicrobial [1], antipyretic [2], antiparasitic [3], antihistaminic [4], and antiviral activities [5]. These compounds have found wide applications in drug design for treatment of inflammation [6], hypertension [7], bacterial [8] and HIV infections [9], and some other diseases [10]. Also, thiazoles can be used as starting compound for the synthesis of various dyes.

Thiazole derivatives are traditionally synthesized by the *Hantzsch* synthesis [11][12], which suffers from some disadvantages such as long reaction times, low yields, and harsh reaction conditions [13]. Although there are some reports on new methods to overcome these disadvantages [14][15], the development of novel and ecological methods for their synthesis is still in demand.

Over the last two years, *Nematollahi* and co-workers have shown that benzene-1,2- and -1,4-diols can be oxidized electrochemically to *o*- and *p*-quinones, respectively. These *o*- and *p*-quinones can be attacked by a variety of nucleophiles [16–19].

In the present work, the electrochemical oxidation of some catechols and hydroquinones was studied in phosphate buffer solutions mixed with EtOH in the presence of rhodanine (**3**) as nucleophile. The results indicated various mechanisms such as EC (electrochemical reaction) and ECEC (electrochemical reaction – chemical reaction – electrochemical reaction – chemical reaction).

The developed procedure is characterized by high atomic economy, safe waste (phosphate salts), mild conditions (room temperature and atmospheric pressure), and easy handling with an undivided cell using three graphite electrodes.

2. Results and Discussion. – 2.1. *Electrochemical Oxidation of Hydroquinone* (**1a**) *in Both the Absence and Presence of Rhodanine* (**3**). The cyclic voltammogram of 2 mM **1a** in phosphate buffer solution (0.15M, pH 7.0)/EtOH 4:1 shows one anodic (A_1) and the corresponding cathodic peak (C_1), which are related to the transformation of **1a** to *p*-benzoquinone (**2a**) and *vice versa* by a *quasi*-reversible two-electron process (*Fig. 1*,*a*). A peak current ratio (I_{PC1}/I_{PA1}) of nearly unity, particularly during the recycling of potential, can be considered as criteria for the stability of **2a** formed at the surface of the electrode under optimum experimental conditions. In other words, any side reactions such as hydroxylation or dimerization [19] are too slow to be observed in the time scale of cyclic voltammetry. The electrochemical oxidation of **1a** in the presence of rhodanine (**3**) as nucleophile was investigated in detail. *Fig. 1*,*b* shows the cyclic voltammogram recorded for a solution of 2 mM **1a** in the presence of 2 mM **3** as nucleophile. Under these optimum conditions, the cathodic counterpart of anodic peak A_1 decreases, and a new cathodic peak (C_0) appears at potentials more negative than cathodic peak C_1, which is related to the electroreduction of intermediate **5a** to **4a**. In addition, *Fig. 1*,*c* shows the cyclic voltammogram obtained for a solution of 2 mM **3** in the absence of **1a** under experimental conditions. The cyclic voltammograms of **1a** in the presence of **3** at different potential sweep rates

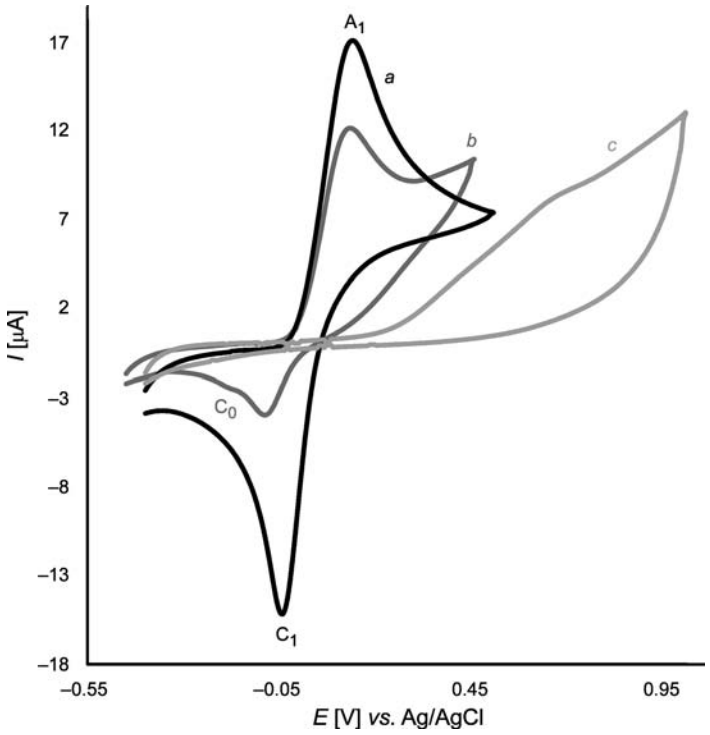


Fig. 1. *Cyclic voltammograms of 2 mM hydroquinone* (**1a**) *in* a) *the absence and* b) *the presence of 4 mM rhodanine* (**3**), *and* c) *that of 2 mM* **3** *in the absence of* **1a** *at the surface of a glassy C electrode in 0.15* M *phosphate buffer solution* (pH 7.0)/*EtOH* 4:1*; scan rate, 50 mV s*$^{-1}$

are shown in *Fig. 2*. It can be seen that, proportional to the raising of the scan rate in parallel with the decrease in the height of the C_0 peak, the height of the C_1 peak of **1a** increases. A similar situation is also observed when the **3**/**1a** concentration ratio was decreased. On the other hand, the increasing current ratio I_{PC1}/I_{PA1} with increasing scan rate is a good indication of the reactivity of $\mathbf{3}^-$ toward **2a**.

Multicyclic voltammetry of **1a** in the presence of **3** shows that, in the second and third cycles, parallel to the shift of the A_1 peak in a positive direction, a new anodic peak (A_0) appears with an E_p value of -0.1 V *vs.* Ag/AgCl electrode, and the peak current slightly increases in a third scan of potential (*Fig. 3*). This new peak is related to the electrochemical oxidation of intermediate **4a** to **5a**. The positive shift of the A_1 peak in the presence of **3** is probably due to the formation of a thin film of product at the surface of the electrode, inhibiting, to a certain extent, the performance of the electrode process [20]. According to our results, it appears that the *Michael* addition reaction of $\mathbf{3}^-$ to **2a** is much faster than other side reactions, leading to the formation of intermediate **4a**. The electrooxidation of this compound is easier than the electrooxidation of the parent starting molecule **1a** due to the presence of an electron-donating group. After transformation of intermediate **4a** to **5a**, the latter can be

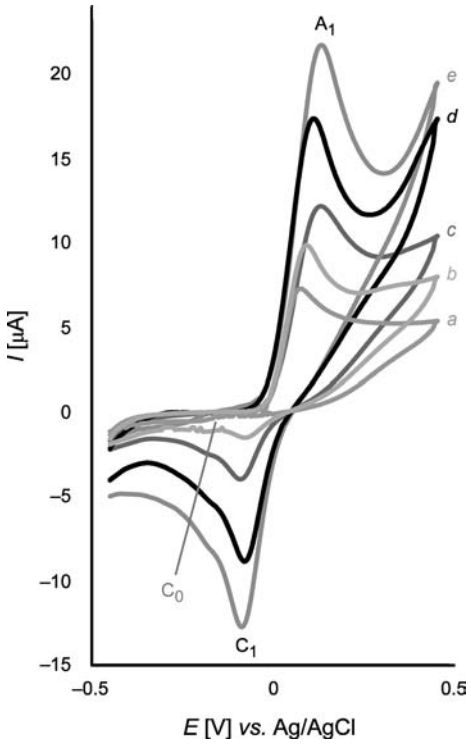


Fig. 2. *Typical voltammograms of 2 mM hydroquinone* (**1a**) *in the presence of* **3** *at a glassy C electrode, in 0.15 M phosphate buffer solution* (pH 7.0)/*EtOH* 4 :1 *at various scan rates:* a) *10,* b) *25,* c) *50,* d) *100, and* e) *150 mV s*$^{-1}$

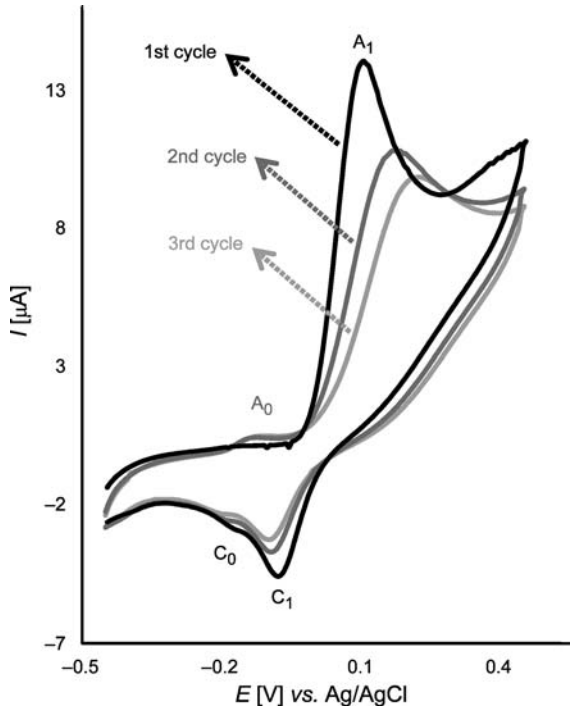


Fig. 3. *Multicyclic voltammograms of 2 mM hydroquinone* (**1a**) *in the presence of 4 mM* **3** *at a glassy C electrode in 0.15 M phosphate buffer solution* (pH 7.0)/*EtOH* 4:1*; scan rate, 50 mV s*$^{-1}$

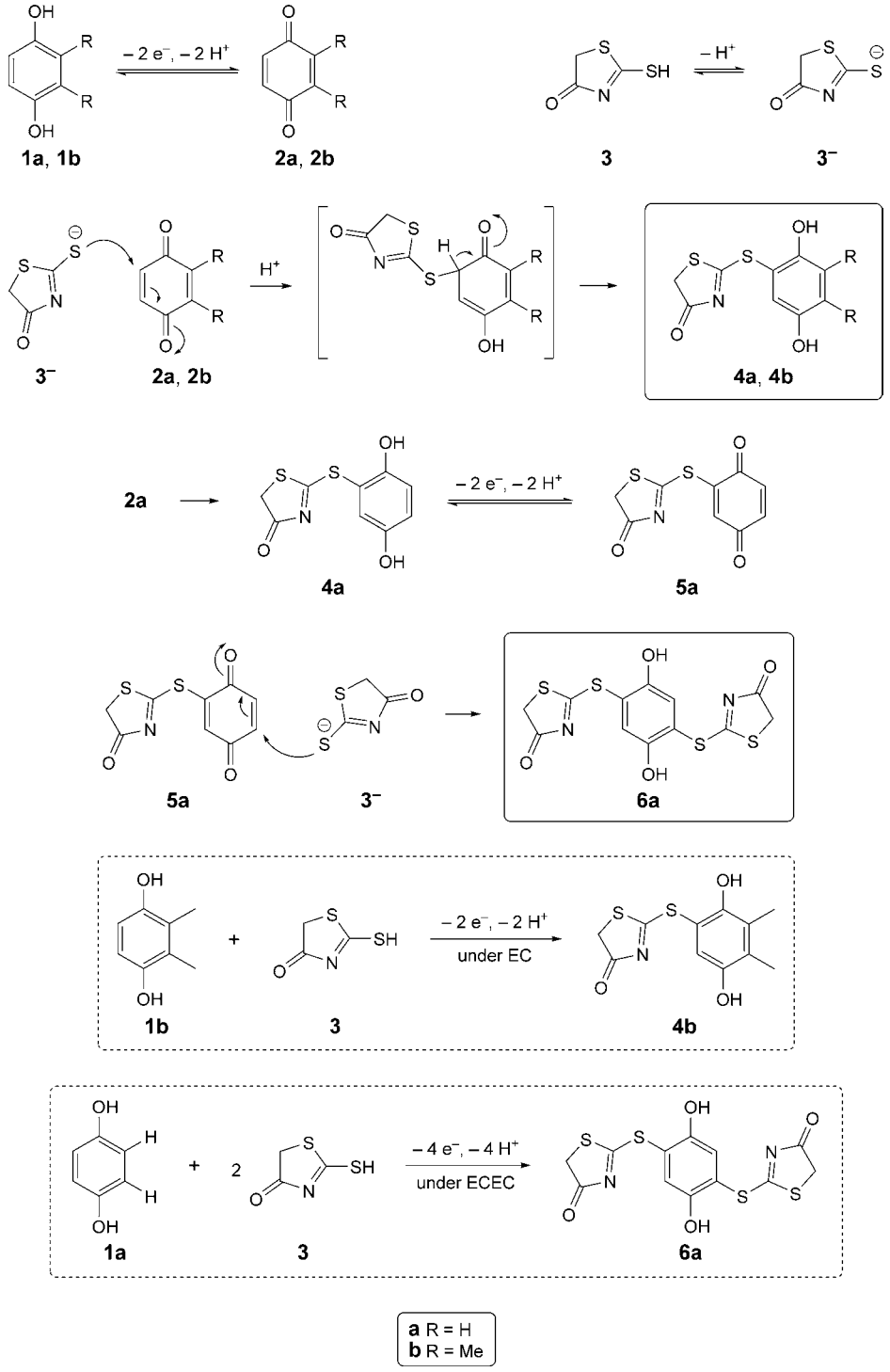
attacked with another **3**$^{-}$ (*Scheme 1*). Product **6a** is insoluble in the 4:1 mixture of buffer solution and EtOH as the supporting electrolyte (0.15M, pH 7.0).

Controlled-potential coulometry was carried out in phosphate buffer solution (0.15M, pH 7.0)/EtOH 4:1 containing 0.3 mmol of **1a** and 0.6 mmol of rhodanine (**3**) at 0.35 V *vs.* Ag/AgCl electrode. The electrolysis progress was monitored by using differential pulse voltammetry (*Fig. 4*). It is shown that, proportionally to the progress of coulometry under constant potential, the anodic peak (A_1) decreases and disappears when the charge consumption becomes about 4e^{-} per molecule of **1a**, and the new anodic peak (A_0) is related to the transformation of intermediate **4a** to **5a** (*Scheme 1*).

The ^{1}H-NMR spectrum shown in *Fig. 5* displays one *singlet* for the aromatic H-atoms. Based on this observation, the formation of **8a** through *Path C* can be excluded. Due to TLC and the appearance of one-spot product, we could easily recognize (*Scheme 2*) which product was obtained. Furthermore, on the basis of the data obtained by GAUSSIAN calculations [21], the stability of **6a** ($E_f = -1524273.4$ kcal) is higher than that of **7a** ($E_f = -1524272.8$ kcal). Thus, this observation allowed us to propose *Path A* in *Scheme 2* for the electrochemical oxidation of **1a** in the presence of **3** by an ECEC mechanism.

The influence of pH on the electrochemical oxidation of hydroquinone **1a**, both in the absence and presence of rhodanine (**3**), was studied by testing the corresponding

Scheme 1



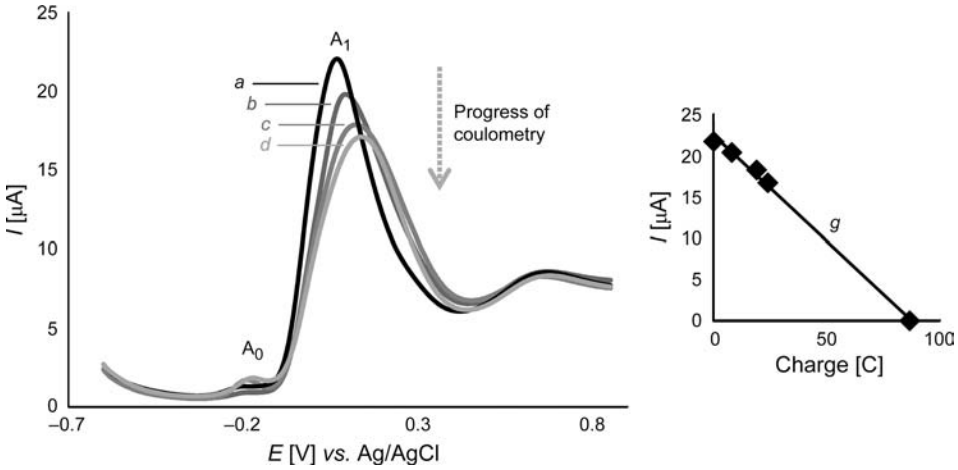


Fig. 4. *Differential pulse voltammograms of 2 mM hydroquinone* (**1a**) *in the presence of 4 mM* **3** *at a glassy C electrode in 0.15 M phosphate buffer solution* (pH 7.0)/*EtOH* (4:1) *during controlled-potential coulometry at 0.35 V* vs. *Ag*/*AgCl* (scan rate, 20 mV s^{-1}). After the consumption of: *a*) 0, *b*) 8, *c*) 19, and *d*) 24 C. Progress of coulometry is associated with decreased anodic peak (A_1) current. *g*) Variation of peak current *vs.* charge consumed.

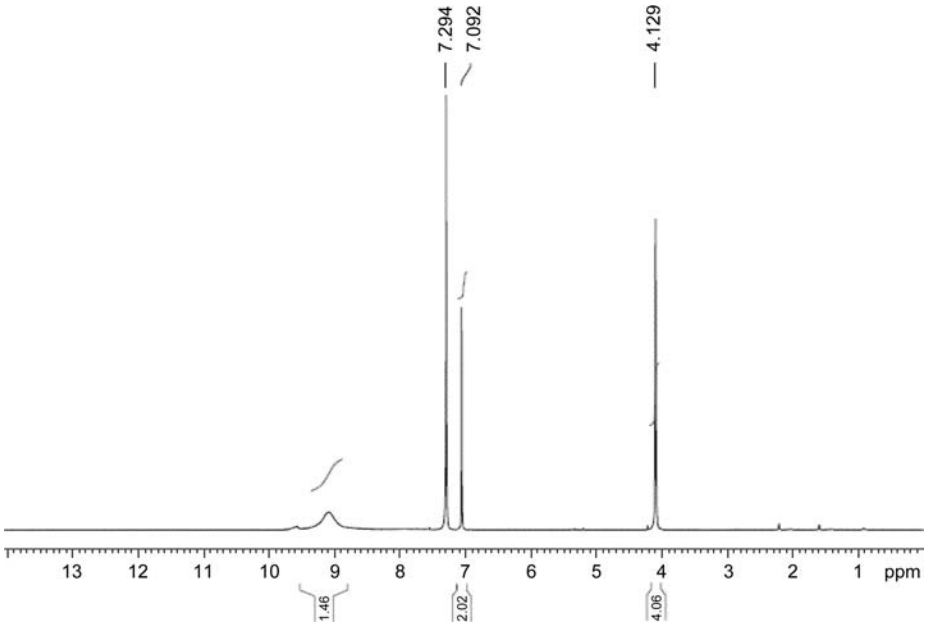
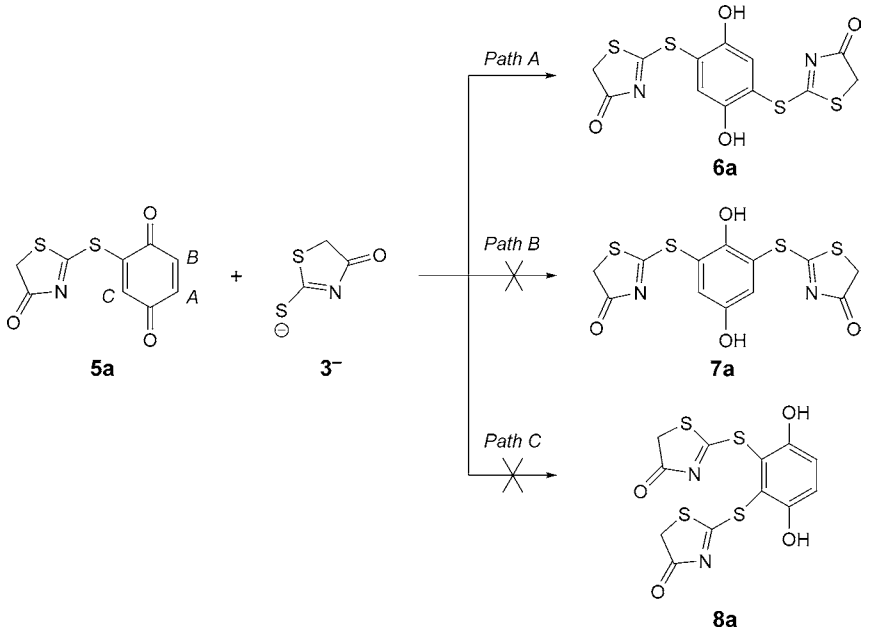


Fig. 5. *^{1}H-NMR Spectrum of* **6a** *in $CDCl_3$*

response at pH 4.0 to 7.0. The cyclic voltammograms of **1a** at pH 4.0 to 7.0 show one anodic (A_1) and a corresponding cathodic peak (C_1), which correspond to the transformation of **1a** to *p*-benzoquinone (**2a**) and *vice versa* by a *quasi*-reversible two-

Scheme 2



electron process [19]. A peak current ratio (I_{PC1}/I_{PA1}) of nearly unity, particularly during recycling of the potential, can be considered as a criterion for the stability of **2a** produced at the surface of the electrode under the experimental conditions. In other words, in this pH range, the side-reactions such as hydroxylation or dimerization are too slow to be observed on the cyclic voltammetry time scale. However, in more basic solutions, the peak current ratio (I_{PC1}/I_{PA1}) is less than unity and decreases with increasing pH (due to an increase of side-reaction rates) [19]. These changes can be related to the coupling of the anionic or dianionic forms of hydroquinone with *p*-quinones (dimerization reactions) [22]. On the other hand, under acidic and neutral pH values, the peak current ratio (I_{PC1}/I_{PA1}) is almost constant and near to unity [19]. The results of the electrochemical behavior of **1a** in the presence of **3** in phosphate buffer solution (pH 4.0–7.0)/EtOH are shown in *Fig. 6*. Electrochemical oxidation of **1a** in the presence of **3** showed that the peak current ratio (I_{PC1}/I_{PA1}) decreases with increasing pH value. This can be related to the reactivity of $\mathbf{3}^-$ for participating in the 1,4-*Michael* addition reaction. Phosphate buffer solution with pH 7.0 was selected as the most suitable solution for this reaction.

2.2. *Electrochemical Oxidation of Catechol* (**1c**) *in Both the Absence and Presence of Rhodanine* (**3**). The electrochemical oxidation of catechol (= benzene-1,2-diol; **1c**) in the presence of **3** as nucleophile was studied in some detail. *Fig. 7* shows the cyclic voltammogram recorded for a 2 mM solution of **1c** in the absence (*a*) and the presence (*b*) of 2 mM **3**. Under optimum experimental conditions, the peak current ratio (I_{PC1}/I_{PA1}) is less than unity and decreases with decreasing the scan rate (*Fig. 8*) and increasing nucleophile concentration. Also, the current function for peak A_1 (I_{PC1}/I_{PA1})

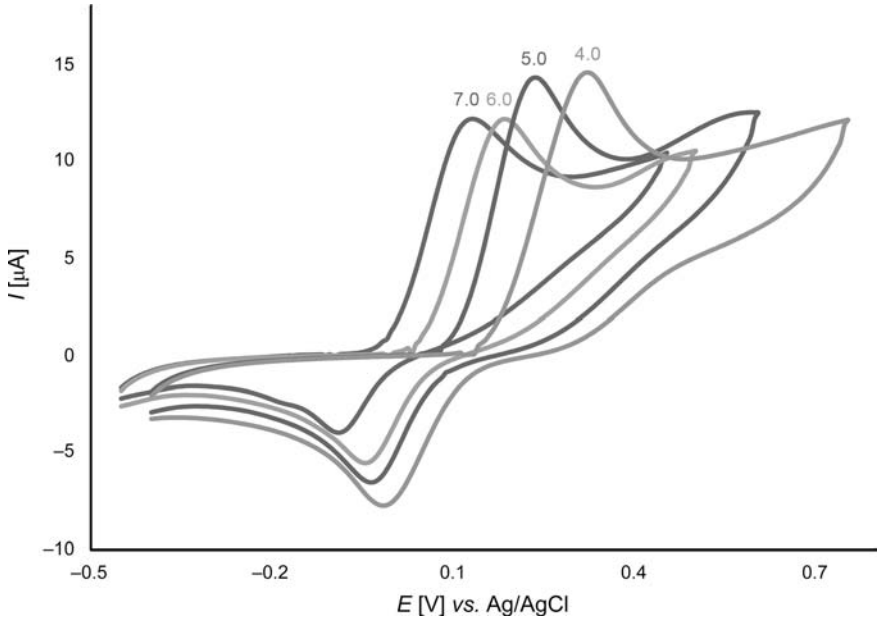


Fig. 6. *Cyclic voltammograms of 2 mM hydroquinone* (**1a**) *in the presence of* **3** *at a glassy C electrode in buffer solution/EtOH* 4 : 1 *at various pH values* (4.0, 5.0, 6.0, and 7.0)*; scan rate, 50 mV s*$^{-1}$

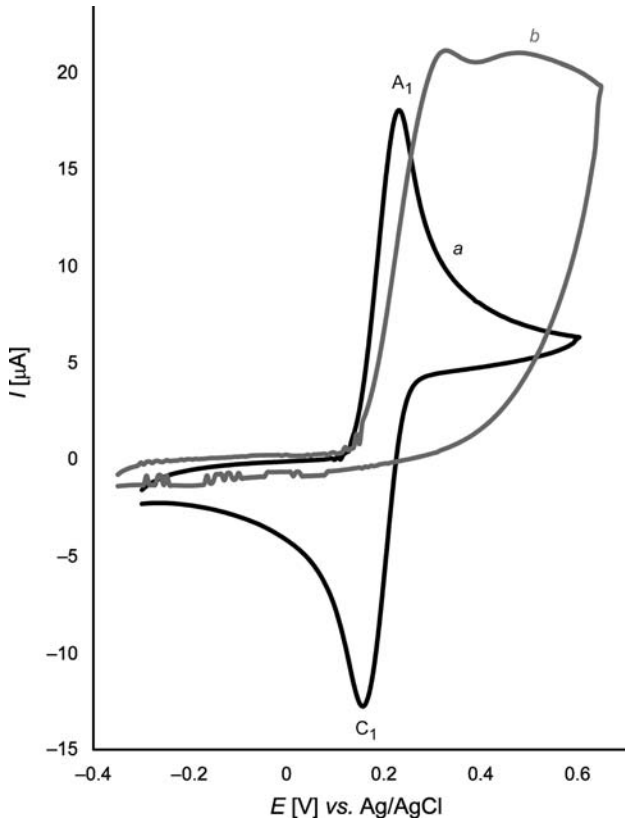


Fig. 7. *Cyclic voltammograms of 2 mM catechol* (**1c**) *in* a) *the absence and* b) *the presence of 2 mM rhodanine* (**3**) *at a glassy C electrode in 0.15M phosphate buffer* (pH 6.0)/*EtOH* 4 : 1*; scan rate, 50 mV s*$^{-1}$

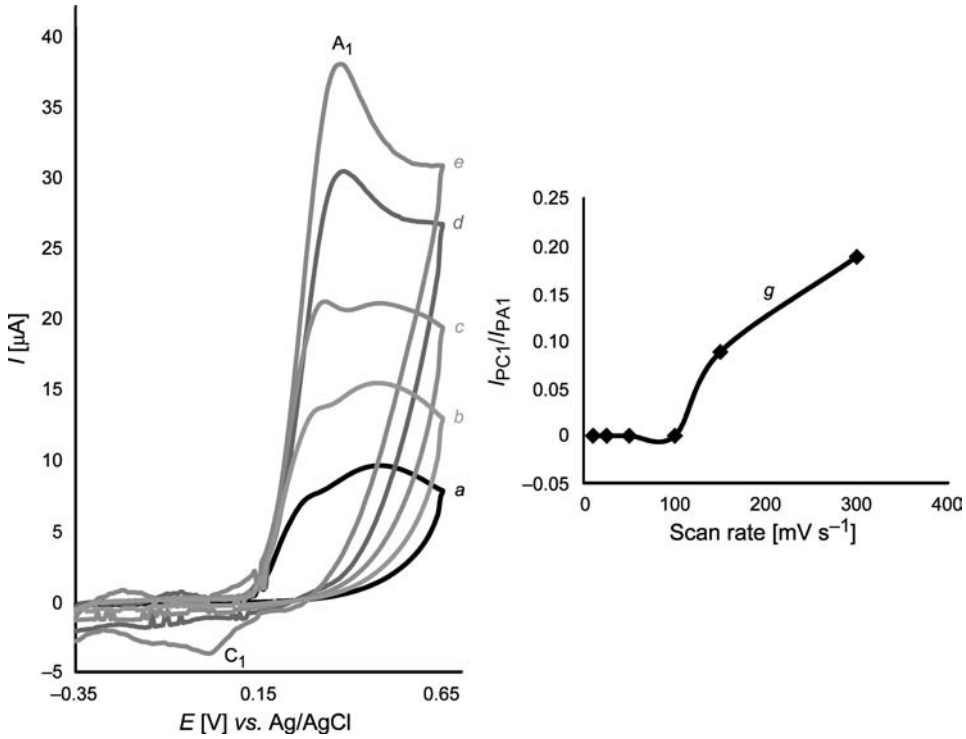


Fig. 8. *Typical voltammograms of 2 mM catechol* (**1c**) *in the presence of* **3** *at a glassy C electrode in 0.15M phosphate buffer* (pH 6.0)/*EtOH* 4 : 1 *at various scan rates:* a) *10,* b) *25,* c) *50,* d) *100, and* e) *150 mV s*$^{-1}$. *g*) Variation of peak current ratio I_{PC1}/I_{PA1} *vs.* scan rate for 2 mM **1c** in the presence of 2 mM **3**.

increases (slightly) with increasing the potential sweep rate (*Fig. 8*,*g*). These results confirmed the reactivity of *o*-quinone (**2c**) towards **3**$^-$. These data correspond to the EC mechanism [23].

The multicyclic voltammograms of **1c** in the presence of **3** are shown in *Fig. 9*. These voltammograms exhibit a decrease relatively in the anodic peak (A_1) and some potential shift to positive potential. The positive shift of peak A_1 in the presence of **3** is due to the formation of a thin film of product at the electrode surface [20].

Controlled-potential coulometry was performed in phosphate buffer solution (0.15M, pH 6.0)/EtOH 4 : 1 containing 0.2 mM of **1c** and 0.2 mmol of **3** at 0.4 V *vs.* Ag/AgCl. The monitoring of the coulometry progress was carried out by cyclic voltammetry (*Fig. 10*). As can be seen, proportional to the progression of coulometry, the anodic peak (A_1) decreases and disappears, when the charge consumption becomes about 2e$^-$ per molecule of **1c**. Also the *Coulomb* consumption of this electrochemical reaction of *ca.* 32 C agrees with the proposed mechanism in *Scheme 3* (EC mechanism), which also holds for the electrochemical oxidation of **1d** in the presence of **3**.

Due to our results, the *Michael* reaction of rhodanine (**3**) with benzoquinones **2c** and **2d** leads to the formation of the new thiazole derivatives **4c** and **4d**, respectively, in good yields and high purities.

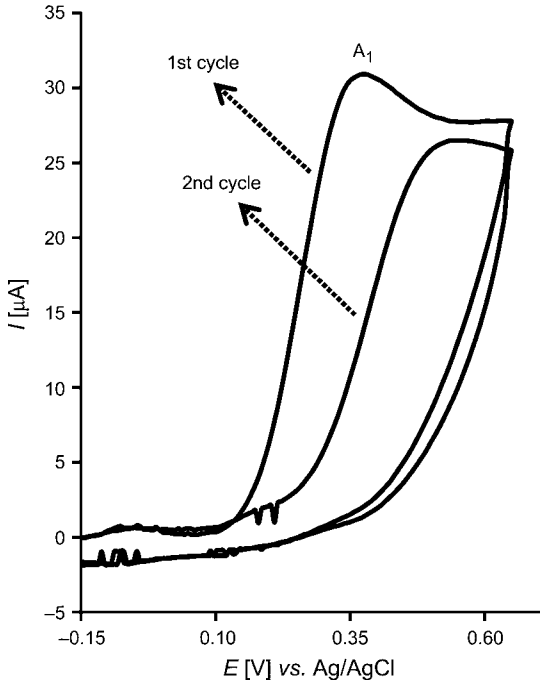


Fig. 9. *Multicyclic voltammograms of 2 mM catechol* (**1c**) *in the presence of 2 mM* **3** *at a glassy C electrode in 0.15M phosphate buffer solution* (pH 6.0)/*EtOH* 4 : 1*; scan rate, 100 mV s*$^{-1}$

The effect of pH on the electrooxidation of catechol (**1c**) in the absence as well as in the presence of **3** was investigated *via* receiving the electrode response at various pH values from 4.0 to 7.0. The position of the redox couple was found to be dependent on pH value. In acidic and neutral media, the cyclic voltammograms of **1c** show one anodic (A_1) and a corresponding cathodic peak (C_1), with a peak current ratio cathode to anode (I_{PC1}/I_{PA1}) near unity. However, at higher pH values (> 7.0), I_{PC1}/I_{PA1} is less than unity, and decreases with increasing pH and decreasing scan rate. This is related to the pairing of anionic or dianionic forms of catechols with their *o*-benzoquinone forms (side-reactions such as dimerization reaction) [22]. The electrooxidation of **1c** in the presence of **3** was investigated at different pH values. The obtained results show that I_{PC1}/I_{PA1} increases with decreasing pH (*Fig. 11*). This can be related to the protonation of S^- in $\mathbf{3}^-$ and their subsequent inactivation toward 1,4-*Michael* addition reaction. Essentially, for reducing the rate of the secondary reaction of catechol (dimerization), and increasing the rate of the pairing reaction between *o*-benzoquinone (**2c**) and **3**, a phosphate buffer solution (0.15M, pH 6.0, 80 ml)/EtOH (20 ml) mixture was selected as the most suitable supporting electrolyte for this electrosynthesis.

3. Conclusions. – The aim of the present study was the electroorganic synthesis of new thiazole derivatives *via* a facile, one-pot, and clean path based on the electrochemical oxidation of hydroquinones, **1a** and **1b**, and catechols, **1c** and **1d**, in the

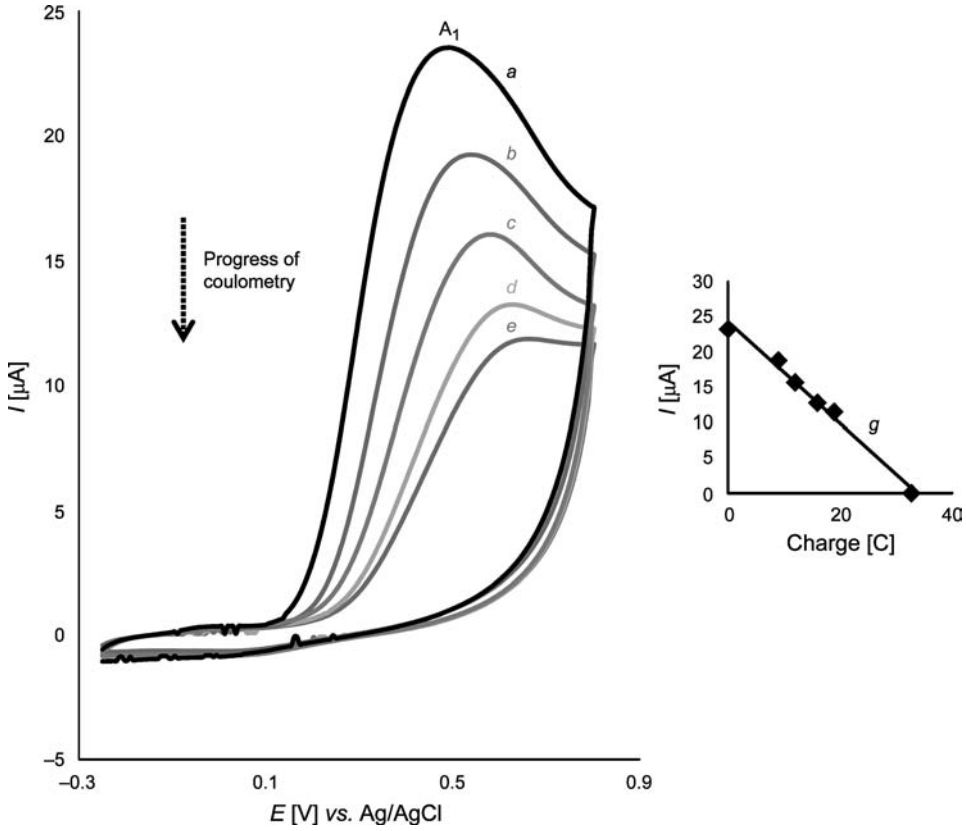


Fig. 10. *Cyclic voltammograms of 2 mM catechol* (**1c**) *in the presence of 2 mM* ***3*** *at a glassy C electrode in 0.15M phosphate buffer solution* (pH 6.0)/*EtOH* 4 : 1 *during controlled-potential coulometry at 0.4 V* vs. *Ag*/*AgCl* (scan rate, 50 mV s^{-1}). After the consumption of *a*) 0, *b*) 9, *c*) 12, *d*) 16, and *e*) 19 C. Progress of coulometry is associated with decreased anodic peak (A_1) current. *g*) Variation of peak current (A_1) *vs.* charge consumed.

presence of rhodanine (**3**). Cyclic voltammetry, differential pulse voltammetry, and coulometry under constant potential indicated that the electrochemical oxidation of **1a**–**1d** in the presence of **3** follows ECEC and EC mechanisms, which are depicted in *Schemes 1* and *3*, respectively. Four- and two-electron process of the mentioned reaction mechanisms (ECEC and EC) was confirmed by controlled-potential coulometry. Clean and ecological synthesis, the use of electricity instead of chemical reagents, no need of high temperature (reflux), as well as a one-step process conducted under mild conditions are advantages of this method. Furthermore, this work introduces electrochemistry as a 'powerful tool' for the synthesis of new important organic compounds such as thiazoles.

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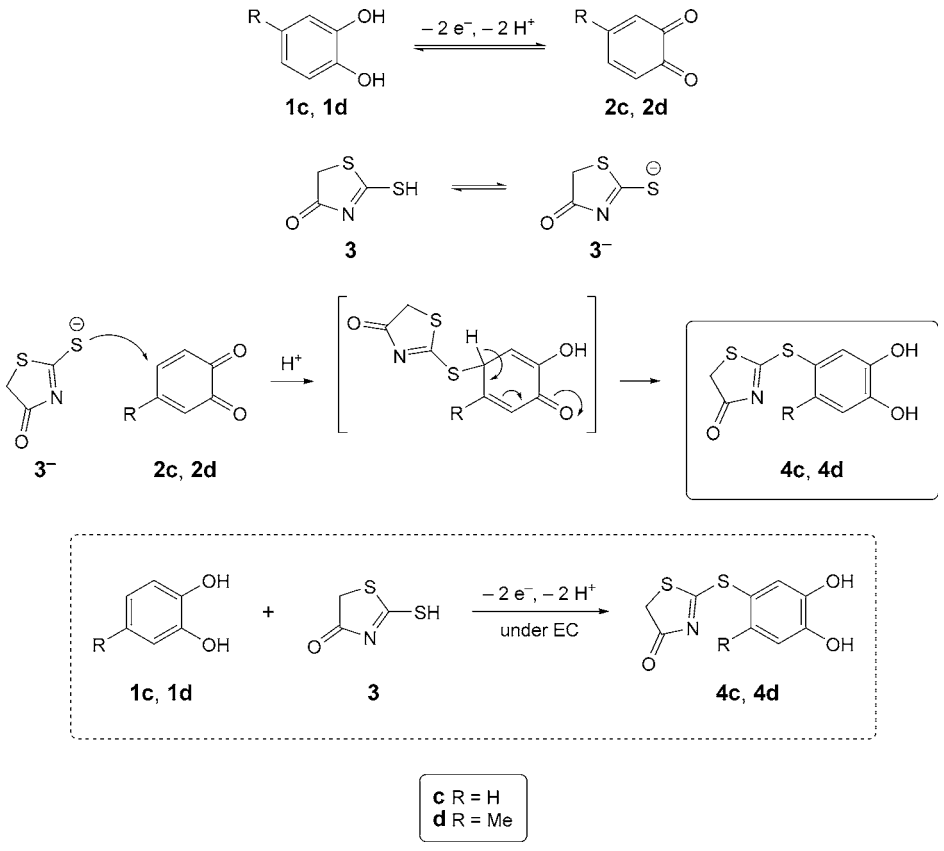
Scheme 3



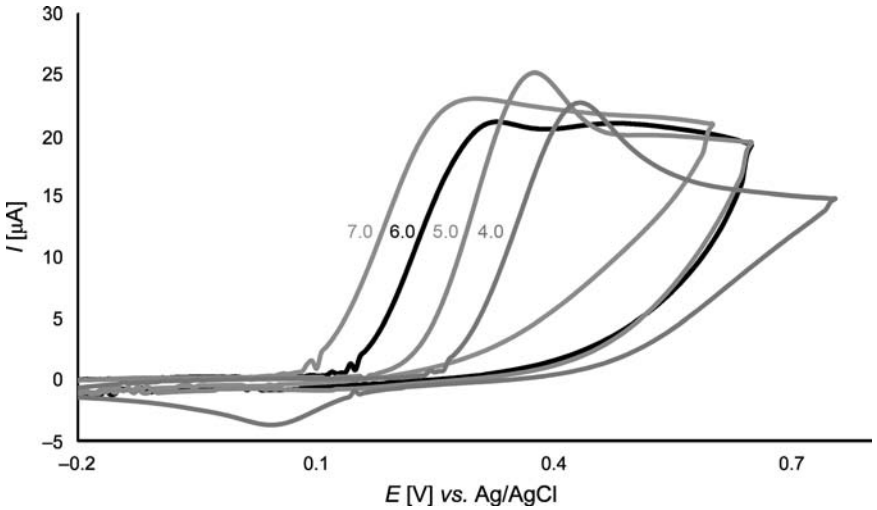


Fig. 11. *Cyclic voltammograms of 2 mM catechol* (**1c**) *in the presence of* **3** *at a glassy C electrode in buffer solution/EtOH 4 : 1 at various pH values* (4.0, 5.0, 6.0, and 7.0)*; scan rate, 50 mV s*$^{-1}$

Experimental Part

General. All chemicals were purchased from *Merck* (DE-Darmstadt). They were used without further purification. For electrochemical instruments, see [18]. IR Spectra: *Shimadzu 8400S* FT-IR spectrophotometer (Tokyo, Japan); $\tilde{\nu}$ in cm^{-1}. ^1H- and ^{13}C-NMR spectra: *Bruker DRX-400 Avance* instrument (Germany); in $CDCl_3$; δ in ppm rel. to Me_4Si as internal standard, J in Hz.

Representative Procedure for the Synthesis of New Thiazole Derivatives **4b** *and* **6a**. In this procedure, 100 ml of H_2O/EtOH 4:1 containing 0.15 M phosphate buffer (pH 7.0) as supporting electrolyte were submitted to pre-electrolysis at 0.35 V *vs.* Ag/AgCl (3 M) electrode in an undivided cell. Subsequently, 1 mmol of *2,3-dimethylhydroquinone* (**1b**) and 1 mmol of *rhodanine* (**3**) were added to the cell (for the synthesis of **6a**, 100 ml of H_2O/EtOH 4:1 containing 0.15 M phosphate buffer with pH 7.0 as supporting electrolyte were selected; subsequently, 0.5 mmol of **1a** and 1 mmol of **3** were added to the cell). Finally, the electrolysis was performed at 0.35 V *vs.* Ag/AgCl (3 M). The electrolysis was terminated, when the current decreased by more than 95%. The process was interrupted several times during electrolysis, and the C anode was washed with THF to reactivate. At the end of electrolysis, several droplets of H_3PO_4 were added to the cell. The precipitated solid was collected by centrifugation and washed several times with H_2O/EtOH 1:1 to remove remaining rhodanine. After purification, products **6a** and **4b** were characterized using FT-IR, and ^1H- and ^{13}C-NMR spectrocospy, and elemental analysis (CHN).

Representative Procedure for the Synthesis of Thiazole Derivatives **4c** *and* **4d**. In a typical procedure, 100 ml of H_2O/EtOH 4:1 containing phosphate buffer (0.15 M, pH 6.0) as supporting electrolyte were submitted to pre-electrolysis at 0.4 V *vs.* Ag/AgCl (3 M) in an undivided cell. Then, 1 mmol of **1c** or **1d** and 1 mmol of **3** were added to the cell. Finally, the electrolysis was carried out at 0.4 V *vs.* Ag/AgCl (3 M). The electrolysis was terminated, when the current decreased by more than 95%. The process was interrupted several times during the electrolysis, and the C anode was washed with THF to reactivate. At the end of electrolysis, several droplets of H_3PO_4 were added to the cell. The precipitated solid was collected by centrifugation and washed with H_2O/EtOH 1:1 to remove remaining **3**. After purification, products **4c** and **4d** were characterized using FT-IR, and ^1H- and ^{13}C-NMR spectroscopy, and elemental analysis.

*2-[(3,4-Dihydroxyphenyl)sulfanyl]-1,3-thiazol-4(5*H*)-one* (**4c**). Yield: 86%. M.p. 260–263°. IR (KBr): 3406 (OH), 2970, 1685 (C=O), 1600 (arom.), 1510 (arom.). ^1H-NMR: 3.92 (*s*, CH_2); 6.95 (*d*, J = 8.4, 1 arom. H); 7.02 (*d*, J = 7.6, 1 arom. H); 7.16 (*s*, 1 arom. H); 8.80 (br., 2 H, OH). ^{13}C-NMR: 40.7; 117.3; 119.7; 122.1; 127.4; 144.7; 146.6; 162.6; 177.3. Anal. calc. for $C_9H_7NO_3S_2$ (240.99): C 44.80, H 2.92, N 5.81; found: C 44.73, H 2.88, N 5.72.

*2-[(4,5-Dihydroxy-2-methylphenyl)sulfanyl]-1,3-thiazol-4(5*H*)-one* (**4d**). Yield: 89%. M.p. 190–192°. IR (KBr): 3432 (OH), 2980, 1683 (C=O), 1612 (Ph), 1503 (Ph). ^1H-NMR: 2.33 (*s*, Me); 3.97 (*s*, CH_2); 6.65 (*s*, 1 arom. H); 6.67 (*s*, 1 arom. H); 9.1 (br., 2 H, OH). ^{13}C-NMR: 20.7; 40.5; 117.3; 121.3; 123.1; 127; 143.3; 144.7; 163; 177.8. Anal. calc. for $C_{10}H_9NO_3S_2$ (255.00): C 47.04, H 3.55, N 5.49; found: C 47.15, H 3.48, N 5.44.

*2-[(2,5-Dihydroxy-3,4-dimethylphenyl)sulfanyl]-1,3-thiazol-4(5*H*)-one* (**4b**). Yield: 90%. M.p. > 300 (dec.). IR (KBr): 3413 (OH), 2972, 1685 (C=O), 1624 (Ph), 1502 (Ph). ^1H-NMR: 2.07 (*s*, Me); 2.15 (*s*, Me); 4.04 (*s*, CH_2); 6.95 (*s*, 1 arom. H); 9.20 (br., 1 H, OH); 9.70 (br., 1 H, OH). ^{13}C-NMR: 11.1; 12.0; 37.1; 118.1; 125.5; 126.4; 131.1; 145.6; 146.5; 163.3; 177.1. Anal. calc. for $C_{11}H_{11}NO_3S_2$ (269.02): C 49.05, H 4.12, N 5.20; found: C 49.11, H 4.14, N 5.13.

*2,2′-[(2,5-Dihydroxybenzene-1,4-diyl)disulfanediyl]bis(1,3-thiazol-4(5*H*)-one)* (**6a**). Yield: 80%. M.p. 270–272°. IR (KBr): 3420 (br., OH), 2977, 1672 (C=O), 1624 (Ph), 1516 (Ph). ^1H-NMR: 4.12 (*s*, 4 H, CH_2); 7.09 (*s*, 1 arom. H); 9.05–9.60 (br., 2 H, OH). Anal. calc. for $C_{12}H_8N_2O_4S_4$ (371.94): C 38.70, H 2.16, N 7.52; found: C 38.80, H 2.11, N 7.61.

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