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PAPER

Electrochemical and Chemical Synthesis of Different Types of Sulfonamide Derivatives of *N,N*-Dimethyl-1,4-benzenediamine Using 4-Nitroso-*N,N*-dimethylaniline†

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Syntheses of two different types of disulfonamide and sulfonamide derivatives of *N,N*-dimethyl-1,4-benzenediamine were carried out by the electrochemical oxidation of 4-nitroso-*N,N*-dimethylaniline in the presence of arylsulfonic acids as nucleophiles and by the chemical reaction of 4-nitroso-*N,N*-dimethylaniline with arylsulfonic acids. The electrochemical synthesis was carried out in aqueous ethanol at pH 7.0 and gave the pure *N,N*-diarylsulfonyl derivatives in 55-76% yield. The chemical synthesis, carried out in water at pH 2.0, provided the pure *N*-arylsulfonyl-3-arylsulfonyl derivatives in 75-85% yield.

Introduction

Electrochemistry is a powerful tool for the synthesis of complex organic compounds.¹ Since electrons are essentially clean reagents and the electrodes can simply be removed at the end of the electrolysis, electrosynthesis is in accordance with all the principles of “green chemistry”.² On the other hand, when the solvents such as water, ethanol and ionic liquids are used in electrosynthesis, it becomes more ecologically friendly.² There is also no need for oxidizing and reducing reagents.

On the other hand, sulfonamides are a large class of antibiotics. They are bacteriostatic that interfere with the bacterial synthesis of folic acid from *p*-aminobenzoic acid.³ They are active against gram-positive and gram-negative aerobes. They are also effective on some protozoans, specially coccidian and *Toxoplasma sp.*³ The importance of sulfonamides has promoted us and other workers to synthesize a number of these compounds.⁴ Following our experience in electrochemical synthesis of organic compounds based on oxidation and in-situ generation of Michael acceptor,⁵ we thought that synthesis of new sulfonamide derivatives based on the oxidation of 4-nitroso-*N,N*-dimethylaniline in the presence of arylsulfonic acids (**5a-5c**) would be useful from the point of view of pharmaceutical properties and electrochemical aspect.

This idea prompted us to investigate electrochemical oxidation of 4-nitroso-*N,N*-dimethylaniline (**1**) (Fig. 1A) in the presence of arylsulfonic acids (**5a-5c**) as nucleophiles (Fig. 1B). This method represents a facile and one-pot electrochemical process for the synthesis of some new disulfonamide derivatives (**8a-8c**) (Fig. 1C) in high yield and purity via electrochemical oxidation of 4-nitroso-*N,N*-dimethylaniline in the presence of arylsulfonic acids under sustainable conditions, without toxic reagents and solvents at a carbon electrode in a divided cell, using an environmentally friendly method. In addition, in this paper the chemical synthesis of some new different sulfonamide derivatives (**12a** and **12c**) (Fig. 1D) in high yields and purities without any reagent and catalyst under green conditions has been discussed.

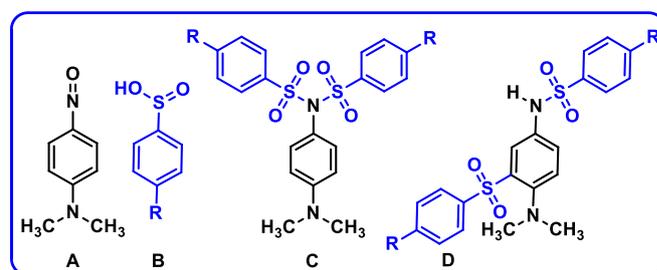


Fig. 1. The structures of 4-nitroso-*N,N*-dimethylaniline (**A**), arylsulfonic acids (**B**), electrochemically synthesized compounds (**C**) and chemically synthesized compounds (**D**).

Results and discussion

The effect of pH

Electrochemical generation of *p*-quinonediimines (**4**) for the synthesis of new sulfonamide derivatives on the one hand, and minimizing its participation in other possible reactions, on the other hand, are the main goal of this work. Therefore, the electrochemical oxidation of 4-nitroso-*N,N*-dimethylaniline (**1**)

has been studied in various pHs. Cyclic voltammogram (CV) of **1** (1.0 mM) in aqueous solution at pH = 2.0 is shown in Fig. 2. At this pH value, the CV exhibits two anodic (A₂ and A₃) and three cathodic peaks (C₁, C₂ and C₃). Peak C₁ is related to the reduction of **1** to *N,N*-dimethyl-*p*-phenylenediamine (**2**) within an irreversible four-electron process (Scheme 1, Eq. 1).⁶ Peaks A₂ and C₂ are related to the oxidation of **2** to the *p*-quinonediimine cation **3** and reduction of the latter in a reversible two-electron process (Scheme 1, Eq. 2)⁷ and peaks A₃ and C₃ are related to the oxidation of **1** to *p*-quinonediimine (**4**) and vice versa within a reversible two-electron process (Scheme 1, Eq. 3).

Cyclic voltammograms of **1** (1.0 mM) in aqueous solution with various pH values are shown in Fig. 3. By increasing pH, the peaks potential for C₁, A₂, C₂, A₃ and C₃ shift to negative values. This confirms the participation of proton(s) in the reduction of **1** to *N,N*-dimethyl-*p*-phenylenediamine (**2**), oxidation of **1** to *p*-quinonediimine (**4**) and oxidation of **2** to *p*-quinonediimines (**3**). The half-wave potential ($E_{1/2}$) for reversible peaks, A₃/C₃, is given by:

$$E_{1/2} = E_{1/2(\text{pH}=0)} - (2.303 mRT/2F) \text{pH}$$

where m is the number of protons involved in the reaction, $E_{1/2(\text{pH}0)}$ is the half-wave potential at pH = 0.0 and R , T , and F have their usual meanings. The $E_{1/2}$ values were calculated for A₃ and C₃ as the average of the anodic and cathodic peak potentials

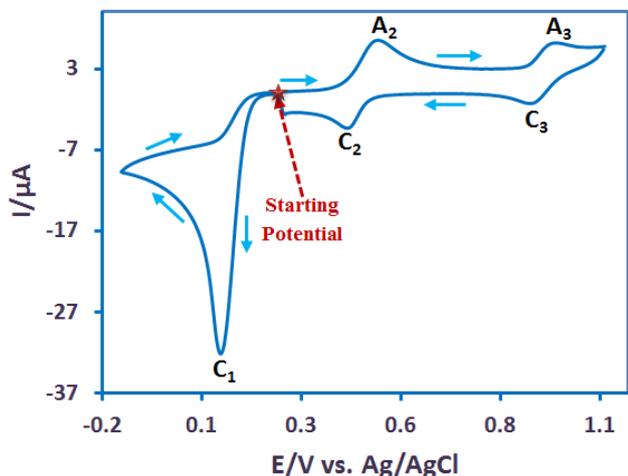
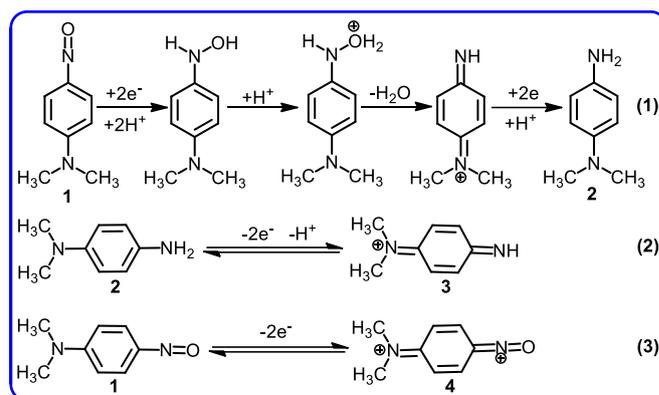


Fig. 2. CVs of 4-nitroso-*N,N*-dimethylaniline (**1**) (1.0 mM) in buffered solution with pH = 2.0, at glassy carbon electrode. Sweeping direction: reduction of **1** at the first stage and oxidation at the second stage. Scan rate: 100 mV s⁻¹. $t = 25 \pm 1$ °C. Vectors show the sweeping direction.



Scheme 1. Proposed mechanism for the oxidation and reduction of 4-nitroso-*N,N*-dimethylaniline (**1**).

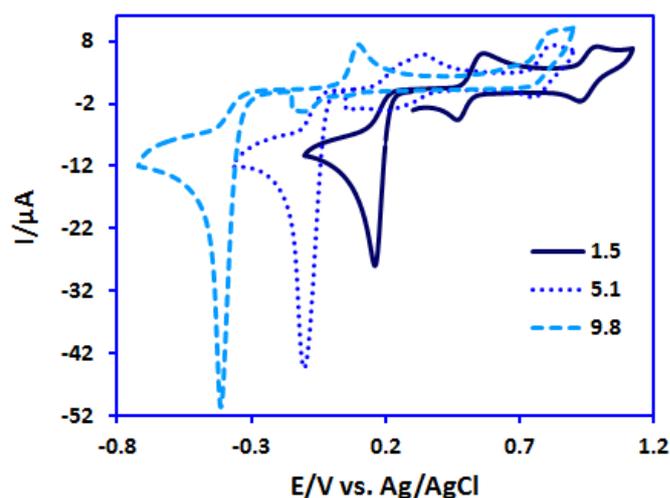


Fig. 3. CVs of 4-nitroso-*N,N*-dimethylaniline (**1**) (1.0 mM) at a glassy carbon electrode, in buffered solutions at various pHs and same ionic strength. Scan rate: 100 mV s⁻¹. $t = 25 \pm 1$ °C.

of the CVs ($(E_{\text{pa}} + E_{\text{pc}})/2$). A potential-pH diagram for A₃/C₃ peaks includes three lines with different equations and slopes around pH values 3.2 and 5.6 (Fig. 4). This diagram indicates that **1** in the aqueous solution can exist in four forms. Three of them are in reduced forms and one other in oxidized form that their relative amounts are determined by pH and electrode potential. At the pHs lower than 3.2, the $E_{1/2}$ value shifts by -63 mV/pH indicating that the redox reaction is two-electron/two-proton process involving the oxidation of “fully protonated” **1** (**FP1**) to **4** in the forward scan and reduction of **4** to **FP1** in the reverse scan (Scheme 2, Eq. 1). On the other hand, in the pH range 3.2-5.6, the $E_{1/2}$ value shifts by -31 mV/pH, indicating that the electrode reaction is two-electron/one-proton process involving the oxidation of “protonated” **1** (**P1**) to **4** and vice versa (Scheme 2, Eq. 2) and finally at the pHs > 5.6, the $E_{1/2}$ value is independent to pH, showing that the redox reaction involves a two-electron process without participation of proton (Scheme 2, Eq. 3). Also, the calculated p*K*_a for acid/base couples **FP1/P1** and **P1/1** are shown in Scheme 3.

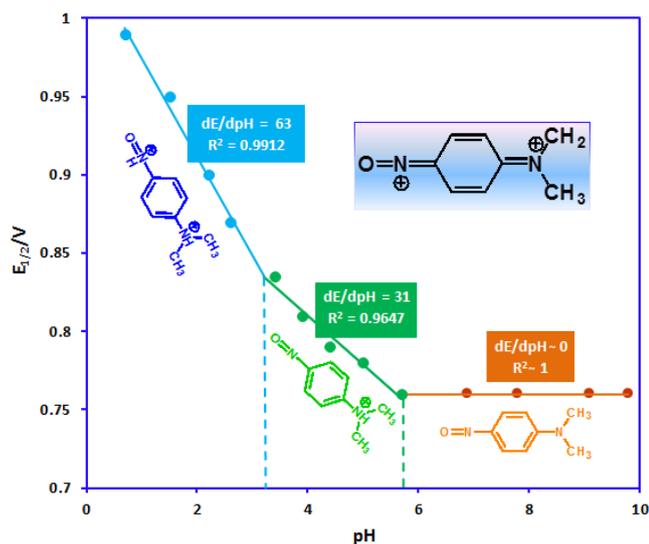
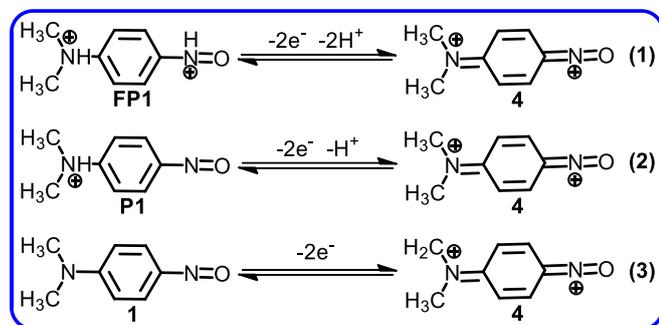


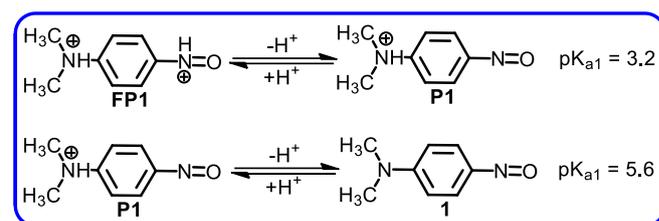
Fig. 4. The potential-pH diagram for oxidation of 4-nitroso-*N,N*-dimethylaniline (**1**).



Scheme 2. Proposed mechanism for the oxidation of 4-nitroso-*N,N*-dimethylaniline (**1**) at various pH values.

CPC study of 4-nitroso-*N,N*-dimethylaniline (**1**)

Controlled-potential coulometry (CPC) was performed in an aqueous phosphate buffer solution ($c = 0.2$ M, $\text{pH} = 2.0$), containing of **1** (0.5 mmol) at 0.90 V versus SCE. The monitoring of the electrolysis process was carried out by cyclic voltammetry (Fig. 5). It shows that proportional to the progress of coulometry, the anodic and cathodic peaks A_3/C_3 decreased and disappears when the charge consumption becomes about $4e^-$ per molecule of **1**. Absorption spectra of **1** were also collected during a controlled-potential coulometry experiment (Fig. 6). These spectra show as the coulometry experiment is carried out, an absorption peak with λ_{max} at 550 nm appears, its height increases and the height of the absorption peak with λ_{max} at 350 nm decreases.



Scheme 3. Acid/base equilibrium for FP1/P1 and P1/1 couples.

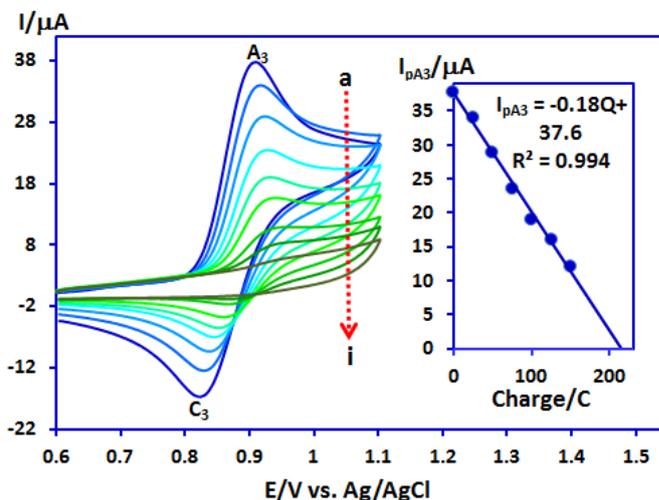


Fig. 5. CVs of **1** (0.5 mmol) at a glassy carbon electrode in phosphate buffer solution ($c = 0.2$ M, $\text{pH} = 2.0$); during controlled-potential coulometry at 0.90 V vs. SCE after consumption of (a) 0, (b) 25, (c) 50, (d) 75, (e) 100, (f) 125, (g) 150, (h) 175 and (i) 205 C. Scan rate: 100 mV s^{-1} . $T = 25 \pm 1$ °C. Inset: variation of $I_{\text{pA}3}$ versus charge consumed.

Mechanistic studies

Cyclic voltammogram of **1** (1.0 mM) in water (phosphate buffer, $c = 0.2$ M, $\text{pH} = 7.0$)/ethanol mixture (70/30, v/v) is shown in Fig. 7 curve a. The oxidation of **1** in the presence of 4-toluenesulfonic acid (**5a**) as a nucleophile was studied in some detail. Figure 7 curve b, shows the CV obtained for **1** (1.0 mM) in the presence of **5a** (1.0 mM).

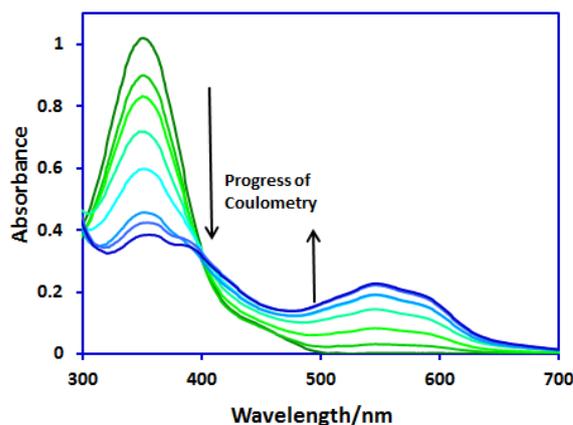


Fig. 6. Absorption spectra of **1** (0.25 mmol) in phosphate buffer solution ($c = 0.2$ M, $\text{pH} = 2.0$), during controlled-potential coulometry at 0.90 V versus Ag/AgCl.

Comparison of this voltammogram with that of **1** in the absence of **5a** (curve a) shows that: (a) a new anodic peak A_4 appears at 0.61 V versus Ag/AgCl (this peak is related to the oxidation of **5a**), (b) in the reverse scan, the cathodic peak C_3 disappears. More studies were performed by varying the potential scan rate in a solution of **1** in the presence of **5a**. The results indicate that the peak current ratio (I_{PC3}/I_{PA3}) is dependent on the potential scan rate and increases with increasing it. The same result was obtained by decreasing the concentration of 4-toluenesulfinic acid (**5a**).

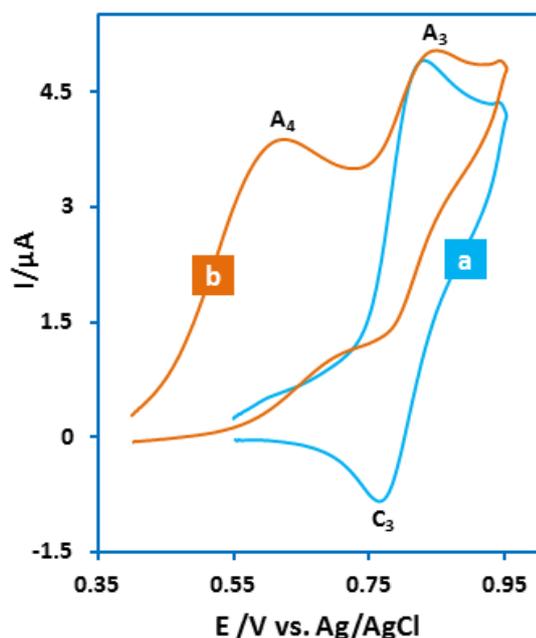


Fig. 7. (a) CV of **1** (1.0 mM) in the absence. (b) CV of **1** (1.0 mM) in the presence of 4-toluenesulfinic acid (**5a**) (1 mM) at a glassy carbon electrode, in water (phosphate buffer, $c = 0.2$ M, $\text{pH} = 7.0$)/ethanol mixture (70/30, v/v). Scan rate: 50 mV s^{-1} , $T = 25 \pm 1$ °C.

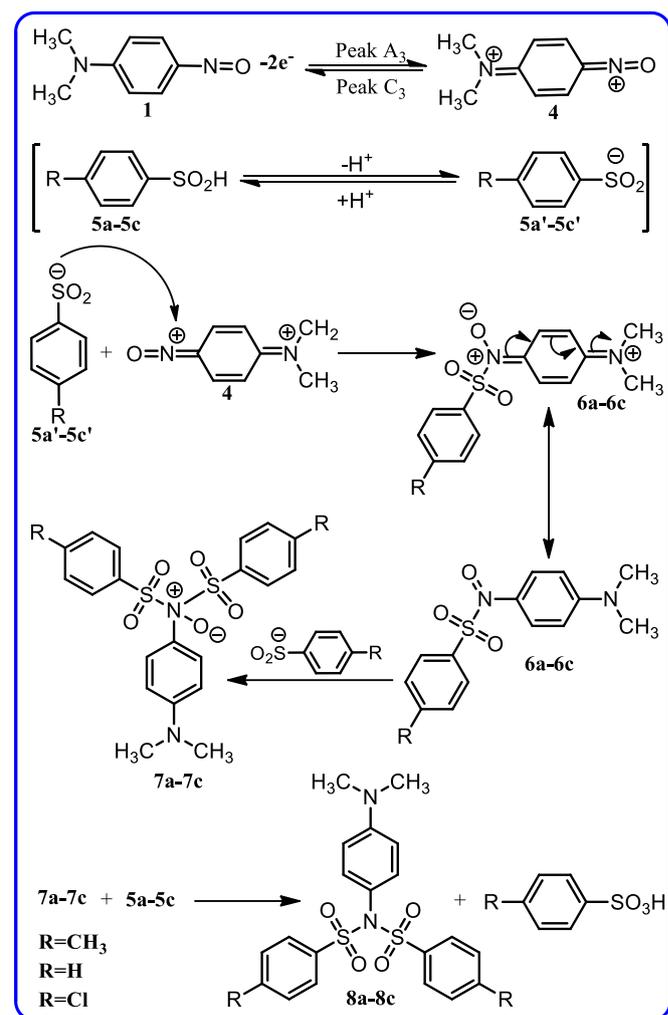
The voltammetry results accompanied by the IR, NMR (^1H and ^{13}C) data and molecular mass of 444 of the final product allow us to propose the following mechanism for the electrochemical oxidation of **1** in the presence of **5a** (Scheme 4).

As shown in Scheme 4, the generation of the *p*-quinonediimine dication **4** is followed by the addition of **5a'-5c'**, which gives the *N*-oxides **6a-6c**. Addition of a second molecule of **5a'-5c'** to the intermediates **6a-6c** generates the *N*-oxides **7a-7c**. Their reduction by **5a'-5c'** gives the diphenylsulfones **8a-8c** as the final product. The oxidation of **8a-8c** should be more difficult than the oxidation of the starting molecule **1** since no products of oxidation of **8a-8c** have been detected. This suggests that the *N,N*-diarylsulfonyl substituent at the *para* position is more electron-withdrawing than the nitroso group. The formation of the disulfonamides **8a-8c** from the *N*-oxides **7a-7c** and the sulfinates **5a'-5c'** can be rationalized as in Scheme 5: addition of **7a-7c** to **5a'-5c'** to give the intermediates **7a'-7c'** which afford **8a-8c** and the arylsulfinic acid by cleavage of the N-O bond. The

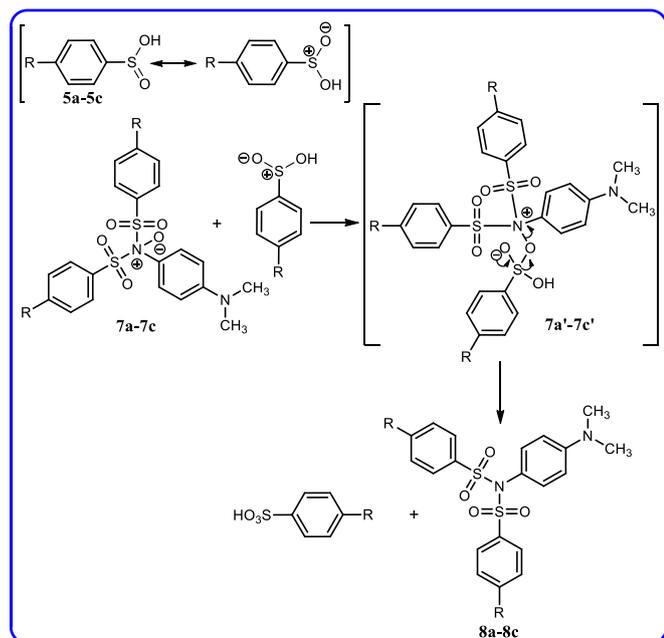
disulfonamides **8a**, **8b**, and **8c** were isolated in 76%, 66%, and 55% yield respectively.

Chemical reaction of **1** with **5a**

The solution of **1** (0.25 mmol) and **5a** (0.5 mmol) in phosphate buffer solution ($c = 0.2$ M, $\text{pH} = 2.0$) was stirred for 12 hour. The monitoring of the reaction was carried out by cyclic voltammetry (Fig. 8). It is shown that, with the passage of time, the height of peak C_1 decrease and finally disappears after 12 hour. After separation and purification of the reaction product, it was characterized by IR, ^1H NMR, ^{13}C NMR, MS and SCXRD. The new compounds **12a** and **12c** (Scheme 6) were isolated in 85% and 75% yield, respectively. The structure of **12a** was further confirmed by single-crystal X-ray diffraction analysis, as shown in Figure 9.



Scheme 4. Proposed mechanism for the **8a-8c**.



Scheme 5. Proposed mechanism for the reduction of **7a-7c**.

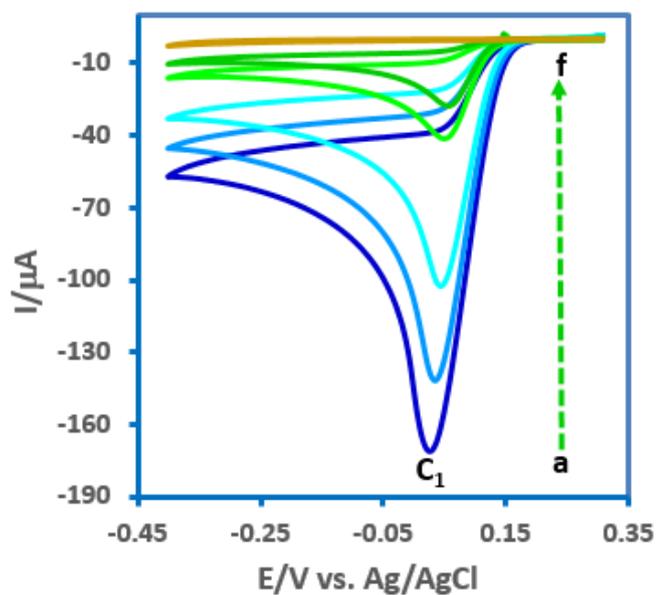
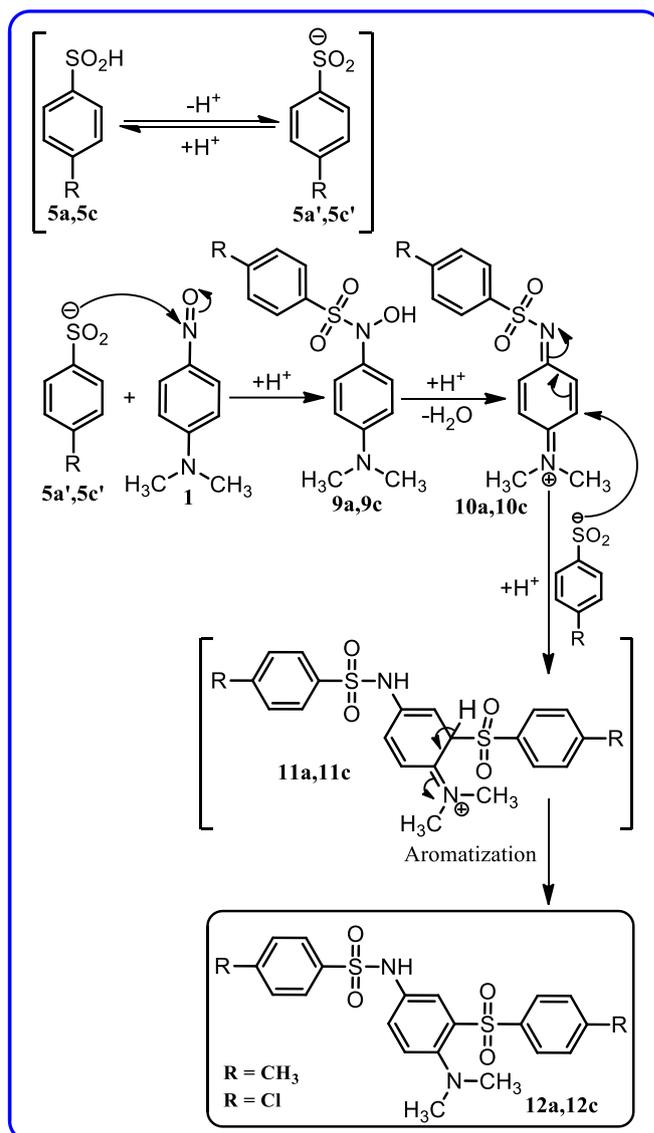


Fig. 8. CVs of **1** (0.5 mmol) in the presence of **5a** (1.0 mmol) at a glassy carbon electrode in aqueous solution containing phosphate buffer ($c = 0.2$ M, $\text{pH} = 2.0$) at different reaction time. Times are: (a) 0, (b) 42, (c) 150, (d) 300, (e) 415 and (f) 720 minute. Scan rate: 100 mV s^{-1} . $T = 25 \pm 1$ °C.



Scheme 6. Proposed mechanism for the synthesis of **12a** and **12c**.

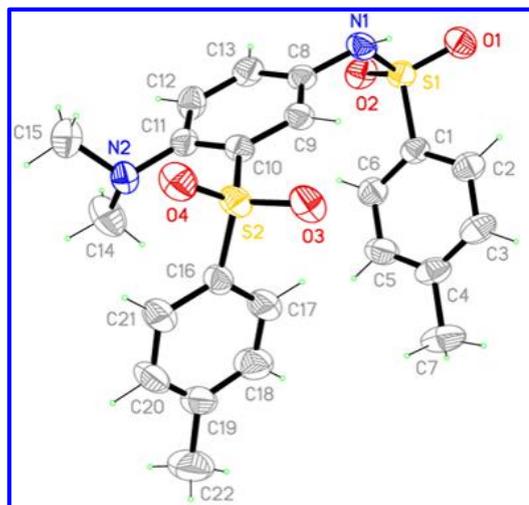


Fig. 9. ORTEP view of the X-ray crystal structure of **12a**.

A mechanism for their formation is proposed in Scheme 6. The first step would be the reaction of **1** with the conjugate base of the arylsulfonic acid (**5a'**, **5c'**) to form an *N*-hydroxysulfonamide (**9a**, **9c**)⁸ and would be followed by protonation and loss of water to produce a *p*-quinone-diimine cation (**10a**, **10c**).⁶ In the third step, the *p*-quinone-diimine cation (**10a**, **10c**) would serve as a Michael acceptor in a reaction with the arylsulfonic acid (**5a'**, **5c'**) to form the final sulfonamide (**12a**, **12c**).

Conclusions

In this work, both electrochemical oxidation of 4-nitroso-*N,N*-dimethylaniline (**1**) in the presence of arylsulfonic acids (**5a-5c**) and chemical reaction of **1** with **5a** and **5c** have been studied. The results of the first part of this work show that 4-nitroso-*N,N*-dimethylaniline (**1**) is oxidized to its corresponding *p*-quinonedimine dication **4** in a two-electron process. Two successive additions of the arylsulfonic acid gives the *N*-oxides **7a-7c**. The final products (disulfonamides **8a-8c**) are obtained in good yield by reaction with **5a-5c** involving an oxidation-reduction process. Finally, we could not obtain the desired products (**8a-8c**) in acceptable yields and purities in acidic and basic media. In the second part of this work, the chemical reaction of **1** with **5a-5b** was studied and it is shown that the direct reaction of **1** with **5a-5b**, leads to the synthesis of sulfonamides **12a** and **12c** in high yield and purity. The comparison of the two methods shows that the electrochemical method allows to synthesize compounds which are different from those obtained by the chemical method.

The present methods for the synthesis of disulfonamides **8a-8c** and sulfonamide **12a**, **12c** have several advantages. Both processes are practically convenient to carry out and can be performed at atmospheric pressure and room temperature. Neither catalyst nor organic/inorganic oxidizing agents are necessary and the reaction can be performed under sustainable (**8a-8c**) and green (**12a**, **12c**) conditions.

Experimental section

General remarks

The working electrode used in macro-scale electrolysis and controlled-potential coulometry was an assembly of four ordinary soft carbon rods (6 mm diameter and 4 cm length), placed as single rods in the edges of a square with a distance of 3 cm, and a large stainless steel cylinder (25 cm² area) constituted the counter electrode. The working electrode used in the cyclic voltammetry experiments was a glassy carbon disc (1.8 mm diameter) and a glass carbon rod was used as the counter electrode. The electrochemistry was performed under controlled-potential condition in a two compartments cell, separated by an ordinary porous fritted-glass diaphragm (a tube with 1.5 cm diameter) and equipped with a magnetic stirrer. 4-nitroso-*N,N*-dimethylaniline (**1**), arylsulfonic acids, phosphate salts and ethanol were obtained from commercial sources.

These chemicals were used without further purification. The glassy carbon electrode was polished using alumina slurry (from Iran Alumina Co.). More details are described in the previous paper.⁹

Electroorganic synthesis of **8a-8c**.

An ethanol/phosphate buffer (*c* = 0.2 M, pH 7.0) mixture (30/70, v/v) solution containing **1** (0.5 mmol) and arylsulfonic acid (**5a-5c**) (1.5 mmol) was electrolyzed in a divided cell at 0.90 V vs. Ag/AgCl. The electrolysis was terminated when the current decreased by more than 95% (current yield 62%). At the end of electrolysis, the precipitated solid was collected by filtration and washed several times with cold water and recrystallized from a mixture of methanol/acetone. After recrystallization, products were characterized by IR, ¹H NMR, ¹³C NMR and MS.

Synthesis of compounds **12a** and **12c**.

An aqueous phosphate buffer solution (*c* = 0.2 M, pH= 2.0), containing **1** (0.5 mmol) and arylsulfonic acid (**5a** and **5c**) (1.0 mmol) was stirred for 12 hour. At the end of this time, the precipitated solid was collected by filtration and washed several times with cold water and recrystallized from a mixture of methanol/acetone. After recrystallization, products were characterized by IR, ¹H NMR, ¹³C NMR, MS and SCXRD.

N-(4-(dimethylamino)phenyl)-4-methyl-*N*-tosylbenzene sulfonamide (C₂₂H₂₄N₂O₄S₂) (**8a**)

Isolated yield: 76%. Mp: 179-181 °C. ¹H NMR (600 MHz, CDCl₃): δ 2.46 (s, 6H, aliphatic), 2.98 (s, 6H, aliphatic), 6.58 (d, *J* = 9.0 Hz, 2H, aromatic), 6.84 (d, *J* = 9.0 Hz, 2H, aromatic), 7.33 (d, *J* = 8.4 Hz, 4H, aromatic), 7.84 (d, *J* = 8.4 Hz, 4H, aromatic). ¹³C NMR (150 MHz, CDCl₃): δ 21.74 (C-10), 40.26 (C-1), 111.8 (C-3), 121.7 (C-5), 128.6 (C-7), 129.5 (C-8), 132.1 (C-4), 137.0 (C-6), 144.6 (C-9), 151.1 (C-2). IR (KBr): 3062 (weak, C-H), 2837, 2794 (weak, C-H), 1522 (strong C=C), 1488 (strong, C=C), 1370, 1170 (strong, S=O), 1089, 884, 722, 579 cm⁻¹. MS (EI, 70 eV): *m/z* (relative intensity) 444 (M⁺, 46), 289 (100), 133 (46), 119 (93), 91 (66).

N-(4-(dimethylamino)phenyl)-*N*-(phenylsulfonyl)benzenesulfonamide (C₂₀H₂₀N₂O₄S₂) (**8b**)

Isolated yield: 66%. Mp: 168-169 °C. ¹H NMR (400 MHz, CDCl₃): δ 2.92 (s, 6H, aliphatic), 6.50 (d, *J* = 9.2 Hz, 2H, aromatic), 6.75 (d, *J* = 9.2 Hz, 2H, aromatic), 7.47 (t, *J* = 8.0 Hz, 4H, aromatic), 7.58 (t, *J* = 8.0 Hz, 2H, aromatic), 7.88 (d, *J* = 7.2 Hz, ~4H, aromatic). ¹³C NMR (100 MHz, CDCl₃): δ 40.22 (C-1), 111.8 (C-3), 123.1 (C-5), 128.5 (C-7), 128.9 (C-8), 132.1 (C-4), 133.7 (C-6), 139.8 (C-9), 151.2 (C-2). IR (KBr): 3061 (weak, C-H), 2923 (weak, C-H), 1602 (strong C=C), 1521 (strong, C=C), 1383, 1158 (strong, S=O), 1084, 954, 895, 754, 721, 579, 551 cm⁻¹. MS (EI, 70 eV): *m/z* (relative intensity) 416 (M⁺, 20), 275 (100), 134 (90), 119 (41), 77 (59).

4-Chloro-*N*-((4-chlorophenyl)sulfonyl)-*N*-(4-(dimethylamino)phenyl)benzenesulfonamide (C₂₀H₁₇Cl₂N₂O₄S₂) (**8c**)

Isolated yield: 55%. Mp: 198–200 °C. ^1H NMR (400 MHz, DMSO- d_6): δ 2.95 (s, 6H, aliphatic), 6.66 (d, J = 8.8 Hz, 2H, aromatic), 6.78 (d, J = 8.8 Hz, 2H, aromatic), 7.79 (d, J = 8.8 Hz, 4H, aromatic), 7.84 (d, J = 8.8 Hz, 4H, aromatic). ^{13}C NMR (100 MHz, DMSO- d_6): δ 39.72 (C-1), 111.9 (C-3), 119.8 (C-5), 129.7 (C-7), 129.8 (C-8), 131.7 (C-4), 137.5 (C-6), 139.5 (C-9), 151.1 (C-2). IR (KBr): 3095 (weak, C-H), 2925, 2854 (weak, C-H), 1603 (strong C=C), 1520 (strong, C=C), 1382, 1168 (strong, S=O), 1092, 896, 754, 613, 553 cm^{-1} . MS (EI, 70 eV): m/z (relative intensity) 484 (M^+ , 10), 309 (100), 134 (55), 119 (17).

***N*-(4-(dimethylamino)-3-tosylphenyl)-4-methylbenzene sulfonamide (C₂₂H₂₄N₂O₄S₂) (12a)**

Isolated yield: 85%. Mp: 182–183 °C. ^1H NMR (600 MHz, DMSO- d_6): δ 2.24 (s, 6H, aliphatic), 2.35 (s, 3H, aliphatic), 2.36 (s, 3H, aliphatic), 7.34 (d, J = 8.4 Hz, 4H, aromatic), 7.39 (d, J = 8.4 Hz, 2H, aromatic), 7.55 (d, J = 8.4 Hz, 2H, aromatic), 7.66 (d, J = 8.4 Hz, 2H, aromatic), 7.81 (d, J = 1.8 Hz, 1H, aromatic), 10.54 (1H, NH). ^{13}C NMR (150 MHz, DMSO- d_6): δ 21.4 (C-1), 21.5 (C-16), 45.5 (C-17), 120.5 (C-10), 126.5 (C-7), 126.9 (C-8), 127.3 (C-4), 128.2 (C-13), 129.4 (C-3), 130.3 (C-14), 135.3 (C-11), 136.7 (C-6), 138.5 (C-5), 138.8 (C-12), 144.0 (C-9), 144.1 (C-2), 149.5 (C-15). IR (KBr): 3234 (medium, N-H), 2946 and 2871 (weak, C-H, aliphatic), 1599 and 1490 (medium C=C), 1371 and 1288 (strong, C=C), 1332 and 1166 (strong, S=O), 1092 (medium C-O), 894, 811, 700, 660, 596, 554 cm^{-1} . MS (EI, 70 eV): m/z (relative intensity) 444 (M^+ , 46), 289 (100), 133 (46), 119 (93), 91 (66).

***4*-Chloro-*N*-(3-((4-chlorophenyl)sulfonyl)-4-(dimethylamino)phenyl)benzenesulfonamide (C₂₀H₁₇Cl₂N₂O₄S₂) (12c)**

Isolated yield: 75%. Mp: 238–240 °C. ^1H NMR (600 MHz, DMSO- d_6): δ 2.25 (s, 6H, aliphatic), 7.38 (d, J = 2.4 Hz, 2H, aromatic), 7.61 (s, 1H, aromatic), 7.62 (d, J = 1.8 Hz, 1H, aromatic), 7.67 (d, J = 1.8 Hz, 2H, aromatic), 7.68 (d, J = 1.8 Hz, 2H, aromatic), 7.70 (d, J = 4.8 Hz, 2H, aromatic), 7.78 (dd, J = 4.8 & 1.8 Hz, 1H, aromatic), 10.72 (1H, NH). ^{13}C NMR (150 MHz, DMSO- d_6): δ 45.3, 120.9, 126.8, 127.7, 129.2, 130.1, 130.1, 135.0, 137.9, 138.3, 138.6, 138.6, 140.5, 149.8. IR (KBr): 3250 (medium, N-H), 3092 (weak, C-H), 2985 and 2890 (weak, C-H, aliphatic), 1604 and 1522 (medium, C=C), 1381 and 1170 (strong, S=O), 1091 (medium C-O), 1013, 941, 891, 758, 682, 614 cm^{-1} . MS (EI, 70 eV): m/z (relative intensity) 484 (M^+ , 10), 309 (100), 134 (55), 119.

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Notes and references

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† Electronic Supplementary Information (ESI) available: FT-IR, ^1H NMR and ^{13}C NMR spectra of **8a-8c** and **12a** and **12c** and crystallographic Information data of **12a** (CIF). See DOI: 10.1039/c000000x/

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Electrochemical and Chemical Synthesis of Different Types of Sulfonamide Derivatives of *N,N*-Dimethyl-1,4-benzenediamine Using 4-Nitroso-*N,N*-dimethylaniline

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Graphical Abstract

Syntheses of disulfonamides and sulfonamides were carried out via the electrooxidation of 4-nitroso-*N,N*-dimethylaniline in the presence of arylsulfonic acids and direct reaction of 4-nitroso-*N,N*-dimethylaniline with arylsulfonic acids, respectively.

