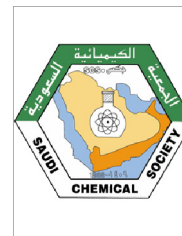




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ORIGINAL ARTICLE

Synthesis of Schiff bases by aromatic amine condensation with 3,3'-bithiophenes-2,2' and 4,4'-dicarbaldehydes

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Abstract Reactions of aromatic amines with 3,3'-bithiophene-2,2'-dicarbaldehyde **1** and 3,3'-bithiophene-4,4'-dicarbaldehyde **2** gave the 2,2'-(*N*-(aryl)diimino)-3,3'-bithiophene **3** and 4,4'-(*N*-(aryl)diimino)-3,3'-bithiophene **4** in good yields. Orthophenylenediamine reacted with **1** and **2** to give dithieno[3,4-*c*;4',3'-*e*]azepino[1,2-*a*]benzimidazole **5** and dithieno[2,3-*c*;3',2'-*e*]azepino[1,2-*a*]benzimidazole **6**. All these original products have been characterized by spectroscopic techniques and elemental analysis.

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1. Introduction

Considerable amount of work has been focused on the preparation of Schiff bases in the last decade (Bindu et al., 1999). Indeed, Schiff bases are often used as starting materials in the synthesis of industrial (Halve and Goyal, 2001; Choi et al., 2000; Hansongnorn et al., 2003; Taggi et al., 2002) and biolog-

ical compounds (Bindu et al., 1999; Halve and Goyal, 2001; Choi et al., 2000; Hansongnorn et al., 2003; Taggi et al., 2002; Aydogan et al., 2001). Moreover, it appears from the literature that Schiff bases with a sulfur atom are a class of important compounds in medicinal and pharmaceutical fields. They show biological activities including antifungal (Aydogan et al., 2001; Samadhiya and Halve, 2001; Pathak et al., 2000; Desai et al., 2001) anticancer (Choi et al., 2000; Hansongnorn et al., 2003; Taggi et al., 2002; Aydogan et al., 2001; Samadhiya and Halve, 2001; Pathak et al., 2000; Desai et al., 2001), antibacterial (Desai et al., 2001; Singh and Dash, 1988; Pandeya et al., 1999; Baseer et al., 2000; More et al., 2001) and herbicidal (Pathak et al., 2000) activities. This is due to their remarkable biological activity, which has been shown to be related to their metal complexing ability (Agarwal et al., 1994, 2004; Labisbal et al., 2003). Recently, we have reported that the 4,8-dihydro-benzo[1,2-*b*;5,4-*b'*]dithiophene-4,8-dione-2-carboxylic acid and 2-hydroxy methyl-4,8-dihydrobenzo[1,2-*b*;5,4-*b'*]dithiophene-4,8-dione can efficiently induce differentiation and apoptosis in leukemia cells (Xu

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et al., 2005; Jing et al., 2005). Induction of apoptosis and cell differentiation is considered as important mechanisms for anti-leukemic therapy. Therefore, it seemed interesting to consider the synthesis of new molecules by condensing aromatic amines and orthophenylenediamine on 3,3'-bithiophene-2,2' and -4,4'-dicarbaldehyde.

In this paper we report the synthesis and characterization of 2,2'-(*N*-(aryl)diimino)-3,3'-bithiophene **3**, 4,4'-(*N*-(aryl)diimino)-3,3'-bithiophene **4**, dithieno[3,4-*c*;4',3'-*e*]azepino [1,2-*a*]benzimidazole **5** and dithieno[2,3-*c*;3',2'-*e*]azepino[1,2-*a*]benzimidazole **6**.

2. Experimental

2.1. Materials and equipments

All melting points are uncorrected and were determined with a Kofler hot-stage apparatus. UV-vis spectra were measured on a Perkin-Elmer 559 spectrometer.

The IR spectra were obtained on a Perkin-Elmer 580B instrument on 1% potassium bromide disks. Mass spectra were recorded at 70 eV and 150 °C on an MS 3300 Finnigan mass spectrometer. The ¹H NMR spectra (δ scale) were recorded at Burgundy's University ¹H NMR facility (CER-EMA) on a Bruker WM400 spectrometer. Analyses were performed by the "Service Central du C.N.R.S."

2.2. General procedure for preparation of 2,2'-(*N*-(aryl)diimino)-3,3'-bithiophene **3** and 4,4'-(*N*-(aryl)diimino)-3,3'-bithiophene **4**

A solution of 3,3'-bithiophene-2,2'-dicarbaldehyde **1** (0.444 g, 2 mmol) or 3,3'-bithiophene-4,4'-dicarbaldehyde **2** (0.444 g, 2 mmol), the appropriate aromatic amine (4.4 mmol) and *para*-toluenesulfonic acid (a small amount), in anhydrous benzene (60 ml) was refluxed for 2 h while water was removed in a Dean-Stark trap. After removal of the solvent, the residue was chromatographed (ethyl acetate/petroleum ether 2:8) giving desired compounds **3** and **4**.

2.2.1. 2,2'-(*N*-(Phenyl)diimino)-3,3'-bithiophene (**3a**)

Yield: 60%. Mp 176 °C (recrystallized from petroleum ether/ethyl acetate or methylene chloride/heptane); ¹H NMR (400 MHz, CDCl₃) δ 7.10–7.34 (m, 10H, ArH), 7.36 (d, 1H, *J* = 5.13 Hz, H(4')), 7.36 (d, 1H, *J* = 5.13 Hz, H(4)), 7.83 (dd, 2H, *J*₁ = 0.93 Hz, *J*₂ = 5.13 Hz, H(5) and H(5')), 8.50 (d, 2H, *J* = 0.93 Hz, H(6) and H(6')); UV (EtOH) 336 (log ϵ 3.75), 262 (4.27); IR (KBr) 1611 (CH=N); MS (70 eV) *m/z* 372 (M⁺, 5.6), 327 (163), 282 (11.6), 281 (25.5), 280 (100), 279 (34.1), 268 (34.4), 149 (24.3), 77 (39), 51 (18.4). Anal. Calcd for C₂₂H₁₆N₂S₂: C, 70.96; H, 4.30; N, 7.52; S, 17.20. Found: C, 70.55; H, 4.30; N, 7.58; S, 17.23.

2.2.2. 2,2'-(*N*-(Benzyl)diimino)-3,3'-bithiophene (**3b**)

Yield: 50%. Mp 134 °C (recrystallized from petroleum ether/ethyl acetate or methylene chloride/heptane); ¹H NMR (400 MHz, acetone-*d*₆) δ 4.68 (d, 4H, *J* = 1.40 Hz, CH₂ and CH₂), 7.18 (d, 2H, *J* = 5.1 Hz, H(4) and H(4')), 7.20–7.30 (m, 10H, ArH), 7.68 (dd, 2H, *J*₁ = 0.93 Hz, *J*₂ = 5.1 Hz, H(5) and H(5')), 8.37 (dt, 2H, *J*₁ = 0.93 Hz, *J*₂ = 1.40 Hz, H(6) and H(6')); UV (EtOH) 280.7 (log ϵ 4.33); IR (KBr) 1595

(CH=N); MS (70 eV) *m/z* 401 [(M+H)⁺, 400 (M⁺, 1.2), 399 (2.2), 397 (2.4), 356 (2), 355 (7.3), 311 (2.7), 310 (9), 309 (3.5), 298 (2.8), 296 (7.4), 294 (2.8), 282 (9.5), 206 (4.5), 204 (5.3), 125 (2.6), 192 (2.4), 194 (0.5), 108 (49.2), 106 (44.4), 91 (12). Anal. Calcd for C₂₄H₂₀N₂S₂: C, 72.00; H, 5.00; N, 7.00; S, 16.00. Found: C, 71.59; H, 5.01; N, 6.89; S, 15.79.

2.2.3. 2,2'-(*N*-(*para*-Methoxy)phenyl)diimino)-3,3'-bithiophene (**3c**)

Yield: 83%. Mp 149 °C (recrystallized from benzene or methylene chloride/heptane); ¹H NMR (400 MHz, CDCl₃) δ 3.76 (s, 6H, CH₃ and CH₃), 6.86–7.75 (m, 8H, ArH), 7.32 (d, 2H, *J* = 5.12 Hz, H(4) and H(4')), 7.78 (dd, 2H, *J*₁ = 0.84 Hz, *J*₂ = 5.12 Hz, H(5) and H(5')), 8.51 (d, 2H, *J* = 0.84 Hz, H(6) and H(6')); UV (EtOH) 355 (log ϵ 4.24), 292 (4.30); IR (KBr) 1611 (CH=N); MS (70 eV) *m/z* 432 (M⁺, 1.8), 312 (11.7), 311 (23.7), 310 (100), 298.1 (17), 295 (23.2), 279 (35.9), 267 (10.9), 44 (37.9), 28 (24.1). Anal. Calcd for C₂₄H₂₀N₂O₂S₂: C, 66.66; H, 4.62; N, 6.48; O, 7.40; S, 14.81. Found: C, 64.22; H, 4.72; N, 6.74; O, 7.32; S, 14.86.

2.2.4. 4,4'-(*N*-(Phenyl)diimino)-3,3'-bithiophene (**4a**)

Yield: 5%. Mp dec > 115 °C (recrystallized from petroleum ether/ethyl acetate); ¹H NMR (400 MHz, CDCl₃) δ 6.06 (s, 1H, H(6')), 6.60 (s, 1H, H(6)), 6.75–7.25 (m, 10H, ArH), 7.31 (d, 1H, *J* = 3.53 Hz, H(2)), 7.35 (d, 1H, *J* = 3.53 Hz, H(2')), 7.38 (d, 1H, *J* = 3.53 Hz, H(5)), 7.75 (d, 1H, *J* = 3.53 Hz, H(5')).

2.2.5. 2-(Anilino)-1-(*N*-(phenyl)imino)-1-*H*-benzo[3,4-*c*;4',3'-*e*]3,3'-bithiophene (**4a'**)

Mp dec > 130 °C (recrystallized from methylene chloride/heptane); ¹H NMR (400 MHz, CDCl₃) δ 6.03 (s, 1H, H(6')), 6.27 (s, 1H, H(6)), 6.55–7.12 (m, 10H, ArH), 7.65 (d, 2H, *J* = 3.36 Hz, H(2) and H(2')), 7.70 (d, 1H, *J* = 3.36 Hz, H(5)), 7.72 (d, 1H, *J* = 3.36 Hz, H(5')); IR (KBr) 1595 (C=N), 3372 (N-H); MS (70 eV) *m/z* 372 (M⁺, 62.1), 371 (12.8), 339 (20.5), 282 (12.5), 281 (29.5), 280 (100), 279 (84), 277.9 (20.4), 253 (18.5), 236 (15), 186 (11.4), 167 (28.6), 148.9 (84.7), 150 (10), 113 (10.9), 77 (22.2), 57 (31.8), 55 (12), 51 (10), 43 (18.9), 41 (15.1), 28 (16). Anal. Calcd for C₂₂H₁₆N₂S₂: C, 70.96; H, 4.30; N, 7.52; S, 17.20. Found: C, 70.64; H, 4.72; N, 7.84; S, 16.96.

2.2.6. 4,4'-(*N*-(Benzyl)diimino)-3,3'-bithiophene (**4b**)

Yield: 45%. Mp 93–94 °C (recrystallized from petroleum ether/ethyl acetate); ¹H NMR (400 MHz, CDCl₃) δ 4.56 (d, 4H, *J* = 1.46 Hz, CH₂ and CH₂), 7.16–7.27 (m, 10H, ArH), 7.45 (d, 2H, *J* = 3.53 Hz, H(2) and H(2')), 8.08 (d, 1H, *J* = 3.53 Hz, H(5)), 8.08 (d, 1H, *J* = 3.53 Hz, H(5')), 8.17 (t, 2H, *J* = 1.46 Hz, H(6) and H(6')); UV (EtOH) 264 (log ϵ 4.07); IR (KBr) 1635 (CH=N); MS (70 eV) *m/z* 400 (M⁺, 1.0), 310 (37.9), 311 (18.8), 309 (100), 295 (22.5), 293 (12.8), 282 (3.9), 250 (15), 204 (26.9), 190 (9.6), 177 (10.5), 149 (24.1), 91 (100), 92 (36.8), 65 (41), 57 (103), 28 (12.5). Anal. Calcd for C₂₄H₂₀N₂S₂: C, 72.00; H, 5.00; N, 7.00; S, 16.00. Found: C, 71.80; H, 5.15; N, 7.21; S, 16.10.

2.2.7. 4,4'-(*N*-(*para*-Methoxy)phenyl)diimino)-3,3'-bithiophene (**4c**)

Yield: 83.7%. Mp 200 °C (recrystallized from benzene or methylene chloride/heptane); ¹H NMR (400 MHz, CDCl₃)

δ 3.74 (s, 6H, CH₃ and CH₃), 6.83–7.04 (m, 8H, ArH), 7.57 (d, 2H, $J = 3.32$ Hz, H(2) and H(2')), 8.23 (d, 2H, $J = 3.32$ Hz, H(5) and H(5')), 8.30 (s, 2H, H(6) and H(6')); UV (EtOH) 337.7 (log ϵ 3.94), 272 (4.06); IR (KBr) 1618 (CH=N); MS (70 eV) m/z 432 (M^+ , 8.2), 312 (24), 311 (23), 310 (100), 295 (23.6), 279 (27.7), 267 (10.1), 266 (12.4), 167 (12.3), 149 (38), 122 (10.4), 71 (10.2), 57.1 (15.6), 43.1 (9.5), 28.1 (12.2). Anal. Calcd for C₂₄H₂₀N₂O₂S₂: C, 66.66; H, 4.62; N, 6.48; O, 7.40; S, 14.81. Found: C, 66.66; H, 4.61; N, 6.26; O, 7.13; S, 14.56.

2.3. General procedure for preparation of dithieno[3,4-*c*;4',3'-*e*]azepino[1,2-*a*]benzimidazole **5 and dithieno[2,3-*c*;3',2'-*e*]azepino[1,2-*a*]benzimidazole **6****

A solution of orthophenylenediamine (0.108 g, 1 mmol) in 20 ml of absolute ethanol was added dropwise under reflux to a stirred solution of **1** or **2** (0.222 g, 1 mmol) in 30 ml of absolute ethanol. After the mixture was further stirred and refluxed for 4 h, the solvent was distilled off under reduced pressure. The residue was recrystallized from methylene chloride/heptane.

2.3.1. Dithieno [3,4-*c*;4',3'-*e*] azepino [1,2-*a*] benzimidazole (5**)**

Yield: 85%. Mp 198–200 °C (recrystallized from methylene chloride/heptane); ¹H NMR (400 MHz, CDCl₃) δ 5.21 (s, 2H, CH₂), 7.28–7.37 (m, 2H, ArH), 7.39 (d, 1H, $J = 3.25$ Hz, H(5')), 7.48 (d, 1H, $J = 3.13$ Hz, H(5)), 7.51 (d, 1H, o(C₆H₄)), 7.59 (d, 1H, $J = 3.25$ Hz, H(2')), 7.82 (d, 1H, o(C₆H₄)), 8.37 (d, 1H, $J = 3.13$ Hz, H(2)); UV (EtOH) 296 (log ϵ 4.05), 260 (4.04); IR (KBr) 1611 (C=N). Anal. Calcd for C₁₆H₁₀N₂S₂: C, 65.31; H, 3.40; N, 9.52; S, 21.77. Found: C, 65.07; H, 3.36; N, 9.23; S, 21.84.

2.3.2. Dithieno [2,3-*c*;3',2'-*e*] azepino [1,2-*a*] benzimidazole (6**)**

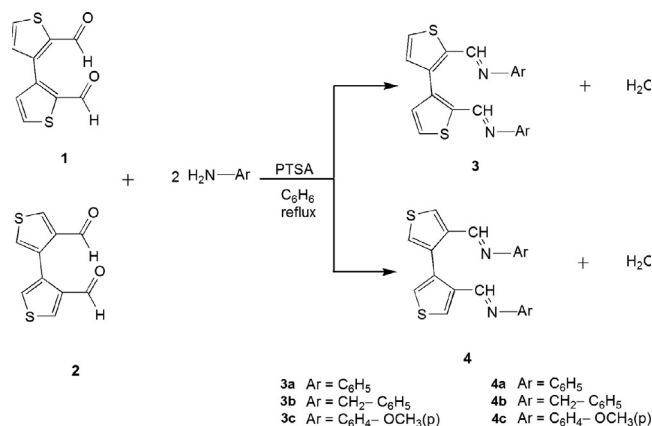
Yield: 80%. Mp 218 °C (recrystallized from methylene chloride/heptane); ¹H NMR (400 MHz, CDCl₃) δ 5.29 (s, 2H, CH₂), 7.28–7.35 (m, 2H, ArH), 7.26 (d, 1H, $J = 5.4$ Hz, H(4')), 7.26 (d, 1H, $J = 5.35$ Hz, H(4)), 7.37 (d, 1H, $J = 5.4$ Hz, H(5')), 7.49 (d, 1H, o(C₆H₄)), 7.56 (d, 1H, $J = 5.35$ Hz, H(5)) 7.82 (d, 1H, o(C₆H₄)); UV (EtOH) 330 (log ϵ 3.72), 303.5 (3.69), 272 (3.67), 246 (3.98); IR (KBr) 1608 (C=N); MS (70 eV) m/z 295 (25.6), 294 (M^+ , 100), 293 (81), 261 (11), 147 (10), 132 (16.9), 85 (47.9), 83 (55.6), 47 (15.2), 45 (11.6). Anal. Calcd for C₁₆H₁₀N₂S₂: C, 65.31; H, 3.40; N, 9.52; S, 21.77. Found: C, 64.97; H, 3.41; N, 9.32; S, 21.31.

3. Results and discussion

3.1. Preparation of compounds **3, **4**, **5** and **6****

All these syntheses involve the preliminary preparation of 3,3'-bithiophene-2,2' and 4,4'-dicarbaldehyde. Access to these products requires multiple synthesis steps which give relatively low overall yields (Mc Dowell and Maxwell, 1970; Gronowitz, 1961; Wynberg and Sinnige, 1969; Kellogg et al., 1969; Gronowitz and Karlson, 1961) The derivatives **3** and **4** were prepared by refluxing the aromatic amines with 3,3'-bithiophene-2,2' and 4,4'-dicarbaldehyde, respectively, in anhydrous benzene, and the presence of *para* toluene sulfonic acid (Scheme 1).

The 2,2'-(*N*-(aryl)diimino)-3,3'-bithiophene, **3** and 4,4'-(*N*-(aryl)diimino)-3,3'-bithiophene **4**, have been obtained in good yields from 69% to 79% with various aromatic amines.



Scheme 1

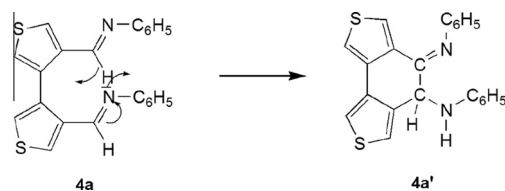
However in the case of the condensation of **2** with aniline, 4,4'-(*N*-(phenyl)diimino)-3,3'-bithiophene **4a** was isolated with a very low yield (5%).

This which is a very unstable derivative during the purification process is transformed by intramolecular migration of a hydrogen atom to deliver the tricyclic compound **4a'** (Scheme 2).

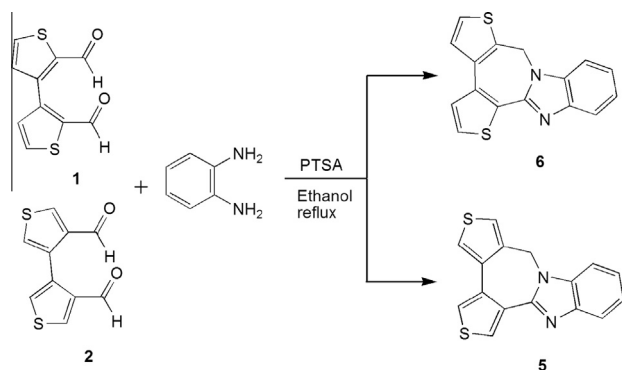
Reaction of orthophenylenediamine with 3,3'-bithiophene-4,4' and 2,2'-dicarbaldehyde, in refluxing ethanol, and the presence of *para* toluene sulfonic acid furnished dithieno[3,4-*c*,4',3'-*e*]azepino[1,2-*a*]benzimidazole **5** and dithieno[2,3-*c*,3',2'-*e*]azepino[1,2-*a*]benzimidazole **6** in 80–85% yields (Scheme 3).

3.2. Identification of products

The structure elucidation of compounds **3**, **4**, **5** and **6** was easily achieved on the basis of their IR, UV, mass, ¹H NMR and micro analytical data. The IR spectra of compounds **3**, **4**, **5** and **6** show absorption bands in the 1595–1635 cm⁻¹ range (N=C stretching) due to conversion of the primary amines into imines. A medium-intensity new band at 3372 cm⁻¹ is observed in the spectrum of derivatives **4a'**, assigned to ν_{N-H} . The electronic spectra of derivatives **3a**, **3c** and **4c** show two bands in the region of 280–355 nm; for compounds **3b** and **4b**, only one band is present in this wavelength domain. The absorption observed at about 290 nm is due to a locally excited state of the bithienal moiety of the molecule. The band appearing near 355 nm is assigned to the aromatic residue and corresponds to an excited state involving the whole molecule (Belletete et al., 1977). In the MS spectra of products **3** and **4**, the molecular ion peak appears with a relatively low intensity between 1% and 9%, enabling, however, the confirmation of the proposed chemical structure. The base fragment peaks for **3b** and **4b** are observed at 401 and 309 m/e and correspond to the $[M + H]^+$ and $[M - CH_2C_6H_5]^+$ ions, respectively. For compounds **3a**, **4a'**, **3c** and **4c**, the base peaks appear at 280, 310 m/e and correspond to the $[M - NHC_6H_5]^+$ and $[M - NHC_6H_4 - OCH_3(p)]^+$ ions, respectively. The molecular peaks are never base peaks (see Section 2). This result translates logically the unstable nature of these products. However, the fragmentation of these derivatives is characteristic of the proposed structures (see Section 2). The ¹H NMR spectra of 2,2'-(*N*-(benzyl)diimino)-3,3'-bithiophene **3b** resemble those of derivatives **3a** and



Scheme 2

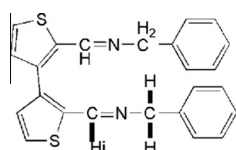


Scheme 3

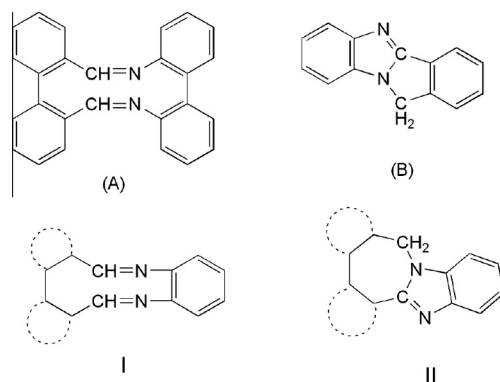
3c, but there is also for the former an additional long-range 4J coupling shown in the schema below (see Scheme 4).

Moreover, besides the H(4), H(5) coupling, a coupling between the most deshielded thiophene proton H(5) and the imine proton (Hi) ($J = 0.93$ Hz, $J = 5.13$ Hz) is observed.

For each compound, series 3, the thiophene protons are easily identifiable, since they resonate between 7.18 and 7.83 ppm with a coupling constant ($J = 5.13$ Hz) as conventionally observed (Hoffman and Gronowitz, 1961) for this kind of protons. In the case of 4,4'-(N-(aryl)diimino)-3,3'-bithiophene 4, thiophene protons appear as doublets from 7.72 to 8.23 ppm, the coupling constant value ($J = 3.5$ Hz) is that commonly observed for thiophene protons α , α' (Hoffman and Gronowitz, 1961). Molecules 3 (3a, 3b, 3c) and 4 (4b, 4c) showed a symmetry plane since the thiophene protons although coupled with one another and sometimes coupled with the imine proton give rise to a unique signal for H(2), H(2') and H(4), H(4') and H(5), H(5'). In contrast, for compound 4a, the signals of protons H(2), H(2') are distinct and the difference in chemical shifts is about 0.4 ppm; this result is in good agreement with the non-symmetrical arrangement. In addition, for each one of the compounds 3 and 4, the imine protons are equivalent, while for 4a a separate signal appears for each imine proton, resonating at about 6.06 and 6.60 ppm. 2-(Anilino)-1-(N-(phenyl)imino)-1H-benzo[3,4-c:4',3'-e]-3,3'-bithiophene 4a', ^1H NMR spectrum shows three doublets whose respective intensities are 1, 1, 2, the first at 7.72 ppm, the second at 7.70 ppm and the third at 7.65 ppm. They have the same coupling constant equal to 3.36 Hz, which corresponds to one of the thiophene protons α ,



Scheme 4



Scheme 5

α' . We concluded that these three doublets are the signals of the thiophene protons H(5'), H(5), H(2) and H(2'). The intensity 1 signal at 6.03 ppm is assigned to N-H proton since it disappears after addition of D_2O . The intensity 1 singlet signal observed at 6.27 ppm is assigned to proton H(6). In the range of 6.55–7.12 ppm, there are complex multiplets corresponding to the 10 phenyl group protons. Compound 5 presents a spectral morphology similar to that of derivative 6. However, by analogy to the diazo-9,20 [a, c, g, i] benzo cyclodecene (A) (Bergmann et al., 1967) and *ortho*-benzylene-2,1 benzimidazole (B) (Amos and Gillis, 1964), the two structural arrangements (I) and (II) may be postulated for derivatives 5 and 6, respectively (Scheme 5). However, according to data from the literature on type (I) derivatives (Bergmann et al., 1967) the intensity 2 signal corresponding to imine protons should be observed at 8.05 ppm, while compound 5 and 6 spectra show the presence of an intensity 2 singlet between 5.29 and 5.20 ppm. Furthermore, the thiophene protons H(2), H(2') of 5 and H(5), H(5') of 6 appear as four separate signals reflecting a total magnetic non-equivalence. Such a result is in total disagreement with a type I symmetry molecule. The characteristics of compounds 5 and 6 are consistent with data from a type II symmetrical arrangement. Elemental analysis data of all the compounds were found to agree with the theoretical values within the limit of $\pm 0.4\%$.

4. Conclusion

In summary, new di-Schiff bases have been prepared by condensation of aromatic amines and 3,3'-bithiophene-2,2' and 4,4'-dicarbaldehyde. The excellent products yields, the, simple starting materials, and the mild reaction conditions are the main advantages of this reaction. This series of heterocyclic derivatives constitute a family of original compounds, of great interest with regard to biological activity.

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