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Synthesis and Characterization of Some New 4,4′-(1,4-Phenylene)dipyrimidine and 6,6′-(1,4-Phenylene)-di(pyridin-2(1*H*)-one) Derivatives

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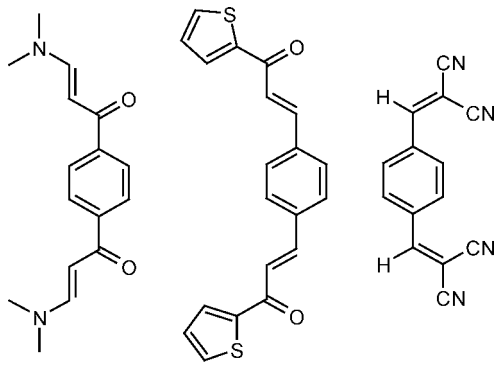
ABSTRACT: *A series of new 4,4′-(1,4-phenylene)-dipyrimidines* **5a–c, 8a–c**, *and* **10a,b** *have been synthesized from the reaction of amidines* **1a–c** *with the dienaminone* **2**, *bis-chalcone* **6**, *or ylidenemalononitrile* **9**. *The reaction of malononitrile and ethyl cyanoacetate with* **2** *gave 6,6′-(1,4-phenylene)di(pyridin-2(1H)-ones) (***15a,b***). The structures of the products were proved by elemental analyses, IR, MS, ^{1}H, and ^{13}C NMR spectroscopy.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:507–512, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20150

INTRODUCTION

The synthesis of pyrimidine and 2-pyridone derivatives is a continuing area of interest because a large number of biologically active molecules that contain these moieties [1–8]. In addition, bis-heterocyclic compounds exhibit various biological activities [9–12] and exert much higher antibacterial activity than heterocyclic compounds [13,14]. However, a literature survey revealed only *p*-(4,4-dipyrimidinyl)-benzene [15], *p*-(4,4-di(4-hydroxy-pyrimidinyl)-benzene [16], and 1,4-bis-(2-pyridone-6-yl)-benzene [17] have been prepared, which require drastic conditions and complex pathways. We have previously described the synthesis of di-(1,2,3,4-tetrahydropyrimidine) derivatives via the acid-catalyzed three-component condensation of benzene-1,4-dicarbaldehye with 1,3-dicarbonyl compounds or ethyl benzoylacetate and (thio)ureas [18,19]. In continuation of our work aimed at developing new approaches for the synthesis of new polyfunctionally substituted heterocyclic [20–23] and bis-heterocyclic compounds [24-26] of expected biological activity, the present procedure is based on the condensation of the dienaminone **2**, bis-chalcone **6**, or bis-ylidenemalononitrile **9**, respectively, with the corresponding reagents which include amidines **1a–c**, malononitrile (**11a**), and ethyl cyanoacetate (**11b**) under basic conditions.



2 **6** **9**

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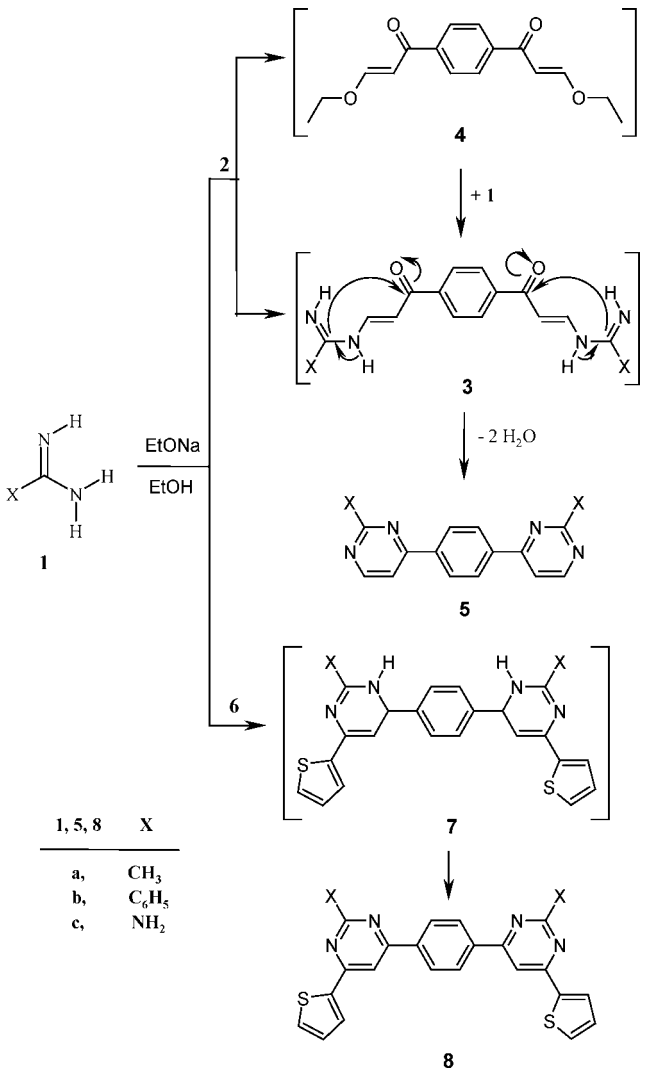
RESULTS AND DISCUSSION

Scheme 1 outlines the synthesis of 4,4′-(1,4-phenylene)-di-2-substituted-pyrimidines **5a–c** from the reaction of the amidine hydrochlorides **1a–c** with 3-dimethylamino-1-[4-(3-dimethylaminoacryloyl)-phenyl]propenone (**2**). The latter compound **2** was obtained from condensation of 1,4-diacetylbenzene with *N*,*N*-dimethylformamidine dimethylacetal [27,28]. For example, the reaction of the dienaminone **2** with 5 equivalents of acetamidine chloride (**1a**) in the presence of 5 equivalents of sodium ethoxide in boiling ethanol (for 18 h) afforded a solid with the empirical formula $C_{16}H_{14}N_4$, and whose mass spectrum showed a strong molecular ion peak, as the base peak at *m/z* 262. The structural assignment of **5a** was confirmed on the basis of its spectroscopic data. The ^{1}H NMR spectrum of **5a** in $CDCl_3$ demonstrated characteristic singlet at δ 2.84 for the two methyl protons, a doublet at δ 7.56 for the two pyrimidine 5-H (J = 5.2 Hz), a singlet at δ 8.24 due to the phenyl protons, and a doublet at δ 8.74 ppm for the two pyrimidines 6-H (J = 5.2 Hz). Moreover, the ^{13}C NMR ($CDCl_3$) of **5a** shows signals at δ 25.29 (2 × CH_3), 113.25 (2 × C-5), 156.84 (2 × C-6), 161.84 (2 × C-4), and 167.26 (2 × C-2), in addition to the phenyl carbons.

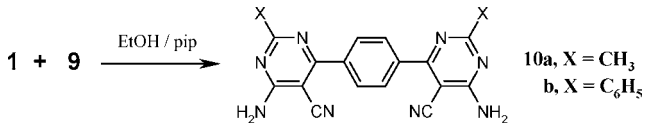
Similarly, compound **2** was cyclized with two different amidine hydrochlorides such as benzamidine hydrochloride (**1b**) and guanidine hydrochloride (**1c**) to give the corresponding dipyrimidines **5b** and **5c**, respectively, in excellent yields (Scheme 1). The molecular formula of compounds **5b,c** is supported by elemental analyses and mass spectra that gave the expected molecular ion peaks. The ^{1}H NMR as well as the IR spectra agreed with the proposed structures **5b,c**. The formation of **5** was rationalized as shown [29] in Scheme 1.

One of the most important characteristics in the reactivity of the enaminones is the displacement of the *N*,*N*-dimethylamino groups by different nucleophiles [30]. One probable route might be attack of the amidine on the enaminone to give **3**, which on subsequent intramolecular cyclization followed by elimination of water that generates the corresponding pyrimidine. The *N*,*N*-dimethylamino groups might also undergo nucleophilic displacement by ethoxide, and the acryloyl derivatives **4** would be formed prior to attack of the amidine and formation of the corresponding pyrimidine. It is known that the condensation of acryloyl derivatives with amidine leads to the corresponding pyrimidine [31].

The reactions of amidines **1a–c** with 3,3′-(1,4-phenylene)-bis[1-(2-thienyl)-2-propen-1-one] **(6)** [32] and sodium ethoxide carried out in abs. ethanol resulted in good yields of 4,4′-(1,4-phenylene)-di-(2-substituted-6-(2-thienyl)-pyrimidine) derivatives **8a–c** (Scheme 1). The structures of dipyrimidines **8a–c** were proven by elemental analyses and spectral data. The gross formula, $C_{22}H_{16}N_6S_2$, of **8c** was confirmed by the mass spectrum, which exhibited the molecular ion at *m/z* 428. The IR spectrum revealed the presence of absorption bands at 3325–3202 cm^{-1} corresponding to the amino group, 3051 cm^{-1} for CH-arom and 1632 cm^{-1} due to C=N. The ^{1}H NMR spectrum of **8c** in DMSO-d_6 showed a four-proton singlet at δ 6.82 assigned for the two amino groups, a two-proton multiplet at δ 7.25 due to the two thiophene rings (4-H), a singlet at δ 7.75 assignable for the two pyrimidine rings (5-H), a two-proton doublet at δ 7.76 due to the two thiophene rings (3-H, J = 6.0 Hz), a two-proton doublet at δ 8.18 due



SCHEME 1

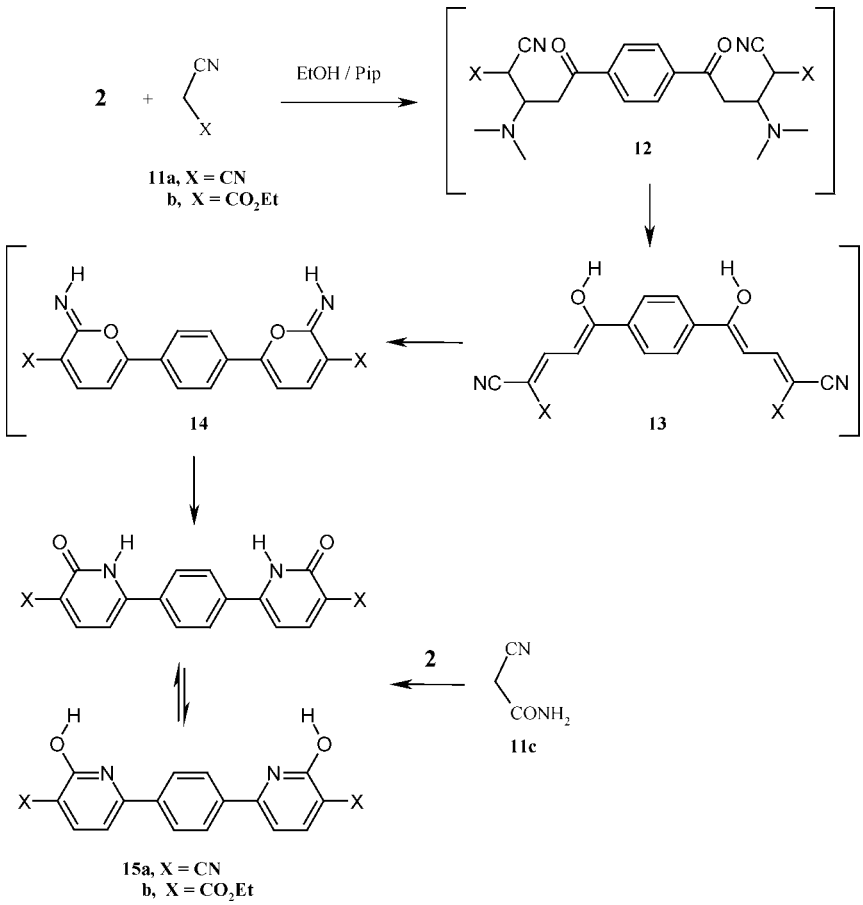


SCHEME 2

to the two thiophene rings (5-H, $J = 6.0$ Hz), and a four-proton singlet at δ 8.24 assigned for the phenyl protons. In its ^{13}C NMR (DMSO-d_6) spectrum, the two pyrimidine rings carbons (C-2, C-4, C-5, and C-6) resonate at $\delta = 169.7$, 165.24, 108.44, and 160.43, respectively; further peaks are at $\delta = 127.0$–138.9 ppm for the thiophene and phenyl carbons. Presumably, the initially formed dihydropyrimidine **7** is air oxidized during the course of the reaction [33] as this intermediate was never observed, and dipyrimidine **8** was the only isolated product.

We have also applied a simple method to the preparation of the fully substituted dipyrimidine **10** (Scheme 2). Thus, the reaction of amidines **1a,b** with 2,2′-(1,4-phenylene)-di(1,1-dicyanoethylene) (**9**) [34] in ethanol containing a catalytic amount of piperidine gave the corresponding 4,4′-(1,4-phenylene)di(6-amino-5-cyano-2-substituted-pyrimidine) **10a,b** and their structures were deduced on the basis of analytical and spectral data. The IR spectra of **10a,b** showed a strong absorption band of the nitrile group at 2217–2218 cm^{-1} and intense bands at 3447–3346 cm^{-1} indicating the presence of stretching vibrations of an amino group. The ^{1}H NMR as well as the mass spectra agreed with the proposed structures of **10a,b**.

The reaction of dienaminone **2** with some active nitriles such as malononitrile and ethyl cyanoacetate was studied. Thus, the reaction of compound **2** with malononitrile (**11a**) or ethyl cyanoacetate (**11b**) at ambient temperature in ethanolic solution and in the presence of piperidine afforded the corresponding 6,6′-(1,4-phenylene)di(pyridin-2(1H)-one) derivatives **15a** and **15b** (Scheme 3). Structures of **15a,b** were based on the correct elemental analyses and spectral data. The IR spectra of **15a,b** were compatible with the assigned structures and revealed the amidic carbonyl absorption bands at



SCHEME 3

1663, 1657 cm^{-1}, respectively. The ^1H NMR of compounds **15a** and **15b** indicated the absence of the dimethylamino group, whereas the spectra revealed two broad singlets at δ 12.20, 12.60 ppm corresponding to the NH-protons. Furthermore, the fragmentation patterns of the mass spectra of **15a** and **15b** showed the molecular ion peak at m/z 314 (M^+, 15%) and m/z 408 (M^+, 23%), respectively which found to be in good agreement with the assigned structure. From this evidence, the mechanistic picture for the pyridone-forming process emerged as shown in Scheme 3. Seemingly, addition of **11** to **2** gives rise to the Michael adduct **12** which subsequently eliminates dimethylamine and undergoes cyclization to the imino-pyran **14** as an intermediate [35]. The presence of piperidine and dimethylamine in the reaction mixture promotes ring transformation of **14** to the corresponding pyridin-2(1H)-one **15**. Further confirmation of structure **15a** was made by comparing with an authentic sample, prepared from the reaction of **2** with cyanoacetamide (**11c**) under the previous conditions, which showed agreement by MP, IR, and ^1H NMR data.

EXPERIMENTAL

All melting points were recorded on a Gallen Kamp apparatus and are uncorrected. IR spectra were obtained from a Perkin-Elmer 880 spectrophotometer. The ^1H NMR spectra were measured in $CDCl_3$ or DMSO-d_6 with a JOEL Lambda 400 (400 MHz) spectrometer using TMS as an internal standard; the ^{13}C NMR spectra were recorded at 100 MHz. The chemical shifts are expressed as δ values (ppm). Mass spectra were determined on a Shimadzu QP 5050A mass spectrometer operating at 70 eV. Reaction monitoring and purity controls of the synthesized compounds were performed by TLC (silica gel, aluminum sheets 60 F_{254}, Merck). The Microanalytical Unit at Chemistry Department, University of Hull, UK performed microanalytical analysis. Compounds **2** [27,28], **6** [32], and **9** [34] were synthesized according to procedures described in the literature.

Synthesis of 4,4′-(1,4-Phenylene)di(2-methylpyrimidine) **5a**

To a stirred solution of **2** (1.2 g, 4.39 mmol) in boiling ethanol (20 mL) was added solution of acetamidine chloride **1a** (2.08 g, 22 mmol, 5 equiv.) in ethanol (20 mL). A solution of Na (0.5 g, 21.95 mmol, 5 equiv.) in ethanol (15 ml) was then slowly added dropwise to the mixture (15 min). The reaction mixture was refluxed for 18 h. The solution was allowed to cool to room temperature and the precipitate was removed by filtration followed by concentration of the filtrate under reduced pressure. The precipitated solid was recrystallized from ethanol to give **5a** (98 mg; 85%). Pale yellow needles; mp 228–230°C; IR (KBr) υ = 3053 (Ar-H), 1624 (C=N); ^1H NMR ($CDCl_3$): δ = 2.84 (s, 6H, 2 CH_3), 7.56 (d, 2H, J = 5.2 Hz, 2 H-5), 8.24 (s, 4H, Ar-H), 8.74 (d, 2H, J = 5.2 Hz, 2 H-6); ^{13}C NMR δ 25.29 (CH_3), 113.25 (C-5), 126.73′ 137.98 (C arom), 156.84 (C-6), 161.84 (C-4), 167.26 (C-2); MS: m/z = 262 (M^+, 100%). Anal. Calcd for $C_{16}H_{14}N_4$ (262.32): C, 73.26; H, 5.38; N, 21.36%. Found: C, 73.31; H, 5.33; N, 21.42%.

4,4′-(1,4-Phenylene)di(2-phenyl-pyrimidine) **5b**. This compound was prepared in 78% isolated yield by condensation of **2** with benzamidine chloride **1b** using the procedure described for the synthesis of **5a**; pale yellow needles; mp >300°C; IR (KBr) υ = 3051 (Ar-H), 1622 (C=N); ^1H NMR (DMSO-d_6): δ = 7.43 (t, 4H, J = 8.8 Hz, 2 H-3′, 2H-5′), 7.43–7.53 (m, 2H, 2H-4′), 7.85 (d, 2 H, J = 5.2 Hz, 2H-5), 8.29 (dd, 4H, J = 8.8 Hz, 2H-2′, 2H-6′), 8.58 (s, 4H, Ar-H), 8.96 (d, 2H, J = 5.2 Hz, 2H-6); MS: m/z = 386 (M^+, 25%). Anal. Calcd for $C_{26}H_{18}N_4$ (386.46): C, 80.81; H, 4.69; N, 14.50%. Found: C, 80.91; H, 4.71; N, 14.45%.

4,4′-(1,4-Phenylene)di(2-amino-pyrimidine) **5c**. This compound was prepared in 83% isolated yield by condensation of **2** with guanidine hydrochloride **1c** using the procedure described for the synthesis of **5a**; pale yellow needles; mp >300°C; IR (KBr) υ = 3308–3191 (NH_2), 3049 (Ar-H), 1634 (C=N); ^1H NMR (DMSO-d_6): δ = 6.95 (s, 4H, 2 NH_2), 7.78 (d, 2H, J = 5.2 Hz, 2H-5), 8.34 (s, 4H, Ar-H), 8.74 (d, 2H, J = 5.2 Hz, 2 H-6); MS: m/z = 264 (M^+, 42%). Anal. Calcd for $C_{14}H_{12}N_6$ (264.29): C, 63.63; H, 4.58; N, 31.80%. Found: C, 63.65; H, 4.61; N, 31.83%.

4,4′-(1,4-Phenylene)di(2-methyl-6-(2-thienyl)-pyrimidine) **8a**. This compound was prepared in 89% isolated yield by condensation of **6** with acetamidine chloride **1a** using the procedure described for the synthesis of **5a**; pale yellow needles; mp 268–270°C; IR (KBr) υ = 3052 (Ar-H), 1631 (C=N); ^1H NMR (DMSO-d_6): δ = 2.80 (s, 6H, 2 CH_3), 7.20 (m, 2H, 2-thiophene H-4), 7.72 (d, 2H, J = 6.0 Hz, 2-thiophene H-3), 7.79 (s, 2H, 2 H-5), 8.12 (d, 2H, J = 6.0 Hz, 2-thiophene H-5), 8.24 (s, 4H, Ar-H); MS: m/z = 426 (M^+, 20%). Anal. Calcd for $C_{24}H_{18}N_4S_2$ (426.57): C, 67.58; H, 4.25; N, 13.13; S, 15.03%. Found: C, 67.63; H, 4.22; N, 13.21; S, 15.09%.

4,4′-(1,4-Phenylene)di(2-phenyl-6-(2-thienyl)-pyrimidine) **8b**. This compound was prepared in 81% isolated yield by condensation of **6** with benzamidine chloride **1b** using the procedure described for the synthesis of **5a**; pale yellow needles; mp 286–288°C; IR (KBr) υ = 3052 (Ar-H), 1632 (C=N); ^{1}H NMR (DMSO-d_6): δ = 7.30 (m, 2H, 2-thiophene H-4), 7.43 (t, 4H, J = 8.8 Hz, 2H-3′, 2H-5′), 7.43–7.60 (m, 2H, 2H-4′), 7.84 (d, 2H, J = 6.0 Hz, 2-thiophene H-3), 7.84 (s, 2H, 2H-5), 8.12 (d, 2H, J = 6.0 Hz, 2-thiophene H-5), 8.30 (dd, 4H, J = 8.8 Hz, 2H-2′, 2H-6′), 8.34 (s, 4H, Ar-H); MS: m/z = 550 (M^+, 45%). Anal. Calcd for $C_{34}H_{22}N_4S_2$ (550.71): C, 74.16; H, 4.03; N, 10.17; S, 11.64%. Found: C, 74.11; H, 4.12; N, 10.21; S, 11.63%.

4,4′-(1,4-Phenylene)di(2-amino-6-(2-thienyl)-pyrimidine) **8c**

This compound was prepared in 85% isolated yield by condensation of **6** with guanidine hydrochloride **1c** using the procedure described for the synthesis of **5a**; pale yellow needles; mp 271–273°C; IR (KBr) υ = 3325–3202 (NH_2), 3051 (Ar-H), 1632 (C=N); ^{1}H NMR (DMSO-d_6): δ = 6.82 (s, 4H, 2 NH_2), 7.25 (m, 2H, 2-thiophene H-4), 7.75 (s, 2H, 2H-5), 7.76 (d, 2H, J = 6.0 Hz, 2-thiophene H-3), 8.18 (d, 2H, J = 6.0 Hz, 2-thiophene H-5), 8.24 (s, 4H, Ar-H), ^{13}C NMR δ 108.44 (C-5), 127.0–138.9 (C-3-C-5 thiophene + C-arom), 143.56 (thiophene C-2), 160.43 (C-6), 165.24 (C-4), 169.7 (C-2); MS: m/z = 428 (M^+, 20%). Anal. Calcd for $C_{22}H_{16}N_6S_2$ (428.54): C, 61.66; H, 3.76; N, 19.61; S, 14.96%. Found: C, 61.71; H, 3.82; N, 19.62; S, 14.94%.

Synthesis of bisnitriles **10a,b**

A mixture of 2,2′-(1,4-phenylene)di(1,1-dicyanoethylene) (**9**) (0.01 mol) and amidine **1a,b** (0.03 mol) in methanol containing a catalytic amount of piperidine was heated under reflux for 7 h. The solid product was collected by filtration and recrystallized from DMF/EtOH.

4,4′-(1,4-Phenylene)di(6-amino-5-cyano-2-methylpyrimidine) **10a**. Yellow needles (yield: 84%), mp >300°C; IR (KBr) υ = 3437–3382 (NH_2), 3049 (Ar-H), 2217 (CN), 1624 (C=N); ^{1}H NMR (DMSO-d_6): δ = 2.46 (s, 6H, 2 CH_3), 7.90 (bs, 4H, 2NH_2), 8.40 (s, 4H, Ar-H); MS: m/z = 342 (M^+, 45%). Anal. Calcd for $C_{18}H_{14}N_8$. (342.37): C, 63.15; H, 4.12; N, 32.73%. Found: C, 63.22; H, 4.14; N, 32.71%.

4,4′-(1,4-Phenylene)di-6-amino-5-cyano-2-phenylpyrimidine **10b**. Yellow needles (yield: 78%), mp >300°C; IR (KBr) υ = 3447–3346 (NH_2), 3052 (Ar-H), 2218 (CN), 1622 (C=N); ^{1}H NMR (DMSO-d_6): δ = 7.40 (t, 4H, J = 8.8 Hz, 2 H-3′, 2H-5′), 7.44–7.54 (m, 2H, 2H-4′), 7.90 (bs, 4H, 2 NH_2), 8.30 (dd, 4H, J = 8.8 Hz, 2H-2′, 2H-6′), 8.50 (s, 4H, Ar-H); MS: m/z = 466 (M^+, 54%). Anal. Calcd for $C_{28}H_{18}N_8$ (466.51): C, 72.09; H, 3.89; N, 24.02%. Found: C, 72.11; H, 3.91; N, 24.11%.

Synthesis of 6,6′-(1,4-Phenylene)di(3-cyano-pyridin-2(1H)-one) **15a**

To a stirred solution of **2** (0.5 g, 1.84 mmol) in boiling ethanol (20 mL), 0.29 g **11a** (4.39 mmol) and a few drops of piperidine were added. The reaction mixture was refluxed for 6 h. The solid product was collected by filtration, washed with ethanol, and recrystallized from EtOH (yield: 69%). mp >300°C; IR (KBr) υ = 3309 (NH), 3049 (Ar-H), 2227 (CN), 1663 (CO), 1621 (C=N); ^{1}H NMR (DMSO-d_6): δ = 5.82 (d, 2H, J = 8 Hz, H-5), 7.75 (d, 2H, J = 8 Hz, H-4), 7.90 (s, 4H, Ar-H), 12.20 (brs, 2H, 2 NH, disappears with D_2O); MS: m/z = 314 (M^+, 15%). Anal. Calcd for $C_{18}H_{10}N_4O_2$ (314.31): C, 68.79; H, 3.21; N, 17.83.Found: C, 68.83; H, 3.31; N, 17.92%.

Compound **15a** was also obtained in 52% yield when **2** reacted with cyanoacetamide (**11c**) in a molar ratio 1:2 under the previous conditions.

6,6′-(1,4-Phenylene)di(3-ethoxycarbonyl-pyridin-2(1H)-one) **15b**. This compound was prepared in 67% isolated yield by reaction of **2** with ethyl cyanoacetate (**11b**) using the procedure described for the synthesis of **15a**; mp >300°C; IR (KBr) υ = 3357 (NH), 3048 (Ar-H), 1725 (CO), 1657 (ring CO), 1623 (C=N); ^{1}H NMR (DMSO-d_6): δ = 1.75 (t, 6H, 2CH_3), 4.03 (q, 4H, 2CH_2), 6.78 (d, 2H, J = 8 Hz, H-5), 7.90 (s, 4H, Ar-H), 8.10 (d, 2H, J = 8 Hz, H-4), 12.60 (brs, 2H, 2NH, disappears with D_2O); MS: m/z = 408 (M^+, 23%). Anal. Calcd for $C_{22}H_{20}N_2O_6$ (408.41): C, 64.70; H, 4.94; N, 6.86. Found: C, 64.81; H, 5.02; N, 6.82.

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