

## Synthesis and spectroscopic characterization of pyrrole-2,3-diones and their following reactions with 1,2-aromatic diamines

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**Abstract:** 4-aryloxy-5-aryl-2,3-furandiones and *N,N*-dialkyl urea combine with loss of water yielding the pyrrole-2,3-dione derivatives in moderate yields (47-68%). Then, these compounds were converted into 2(1*H*)-quinoxalinones with various 1,2-phenylenediamines. The structures and characterizations of the synthesized compounds were established by the <sup>1</sup>H- and <sup>13</sup>C-NMR, IR and elemental analysis.

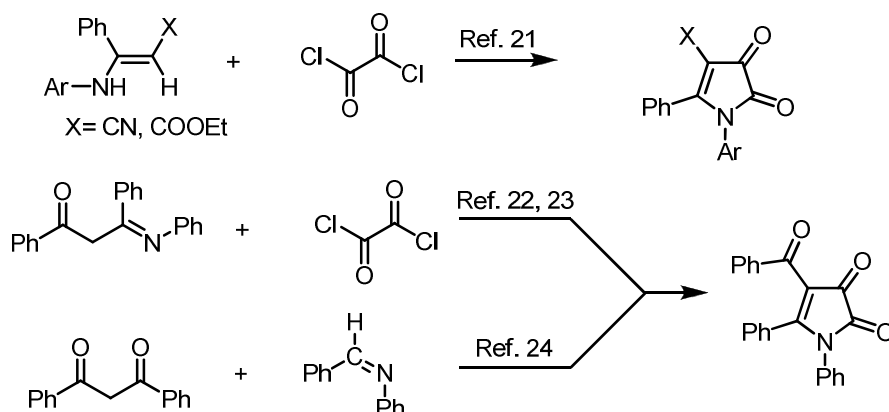
**Keywords:** Furan-2,3-dione; Pyrrole-2,3-dione; Quinoxaline; 1,2-phenylenediamine.

### 1. Introduction

The importance of substituted pyrroles in organic chemistry as building blocks for the synthesis of various representatives of the pyrrole family is well-known. They are commonly found as structural motifs in bio-active molecules such as porphyrins, alkaloids and co-enzymes.<sup>1,2</sup> In view of their importance, there is a continuing interest in developing versatile synthetic routes.<sup>3-5</sup> Generally, pyrroles have been prepared by Knorr reactions,<sup>6</sup> Hantzsch pyrrole synthesis<sup>7</sup> or 1,3-dipole addition of azomethine ylides with alkynes.<sup>8-13</sup> Pyrrole-2,3-dione derivatives which belong to the group of  $\alpha$ -oxo  $\gamma$ -lactames, have proved to be good synthons for different heterocycles. Pyrrole-2,3-diones have been the subject of extensive studies in the recent past.<sup>14-19</sup> There are reports about experimental and theoretical studies of pyrrole-2,3-diones.<sup>20</sup> 1-aryl-4-cyano-5-phenyl-1*H*-pyrrole-2,3-diones and 1-aryl-4-methoxycarbonyl-5-phenyl-1*H*-pyrrole-2,3-diones have been synthesized in 75–94% yield by the reaction of oxalyl chloride with 3-phenyl-3-arylamino propenenitriles and ethyl 3-phenyl-3-arylamino prop-2-enoates, respectively.<sup>21</sup> On the other hand, 4-benzoyl-1,5-diphenyl-2,3-dihydro-1*H*-pyrrole-2,3-dione has been prepared by reaction of the imine of dibenzoylmethane with aniline and oxalyl chloride.<sup>22,23</sup> Red-coloured 4-aryloxy-1,5-diaryl-2,3-dihydro-1*H*-pyrrole-2,3-diones have been obtained by treatment of the 4-aryloxy-5-aryl-2,3-dihydro-1*H*-furan-2,3-dione with Schiff bases at 60–70 °C.<sup>24</sup> 1-(*N,N*-alkylcarbonyl)-4-aryloxy-5-aryl-1*H*-pyrrole-2,3-diones were synthesized from the cyclocondensation reactions of *N,N*-alkylurea with 4-aryloxy-5-aryl-2,3-dihydrofuran-2,3-dione.<sup>20</sup>

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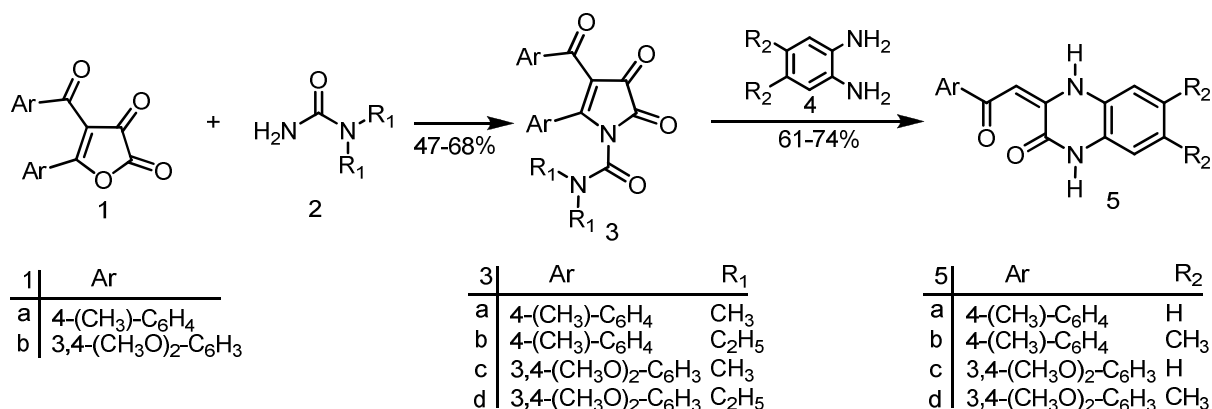
## Synthesis and spectroscopic characterization of pyrrole-2,3-diones



**Scheme 1.** Some synthesis reactions of various pyrrole-2,3-dione

Quinoxaline derivatives constitute the basis of many insecticides, fungicides, herbicides, as well as being important in human health and as receptor antagonists. Although rarely described in nature, synthetic quinoxaline moiety is a part of number of antibiotics such as echinomycin, levomycin and actinomycin which are known to inhibit the growth of Gram-positive bacteria and also active against various transplantable tumours. In addition, quinoxaline derivatives are reported for their application in dyes, efficient electroluminescent materials, organic semiconductors and DNA cleaving agents. These are useful as intermediates for many target molecules in organic synthesis and also as synthons.<sup>25-27</sup> Many synthetic routes have been developed for the synthesis of quinoxaline derivatives. Most common method is the condensation of aromatic 1,2-diamine with 1,2-dicarbonyl compound in refluxing ethanol or acetic acid.<sup>28</sup>

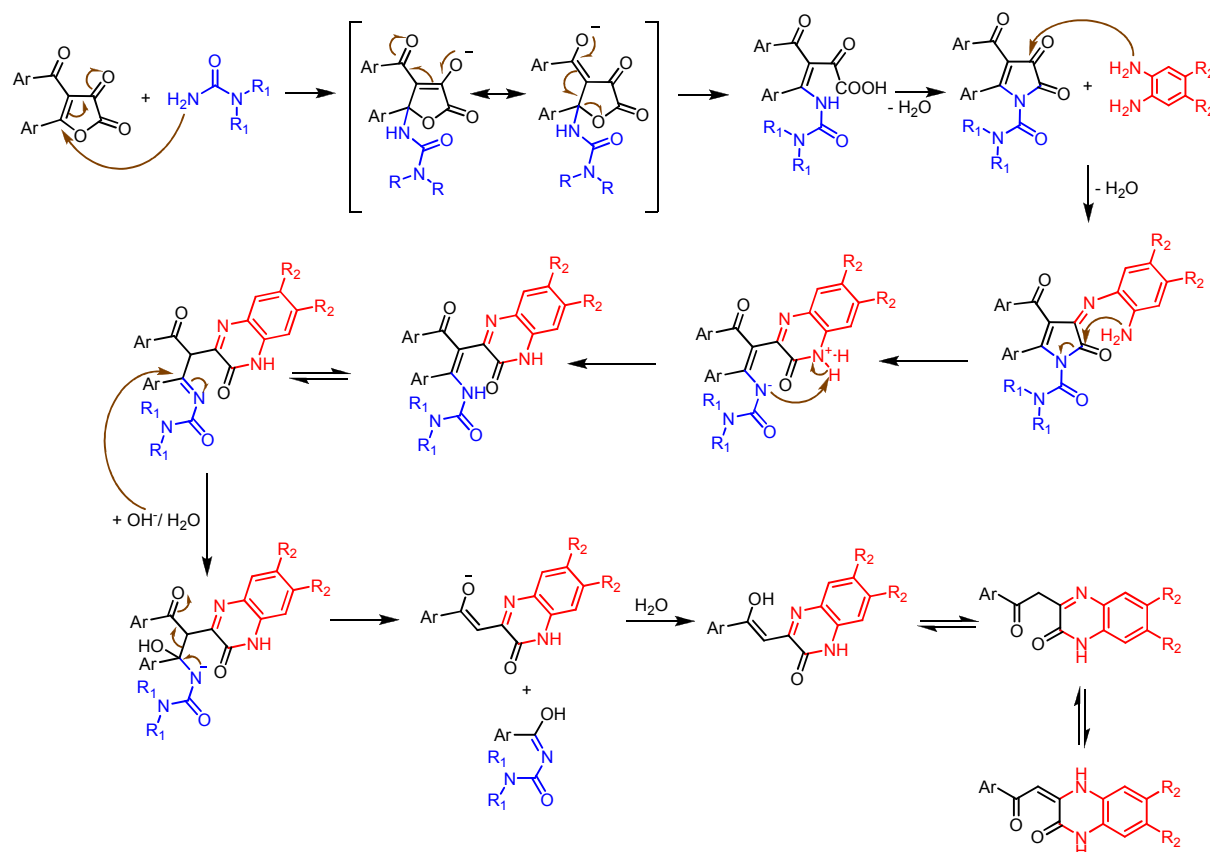
In this study, the reactions of 4-(4-methylbenzoyl)-5-(4-methylphenyl)-2,3-furandione (**1a**) and 4-(3,4-dimethoxybenzoyl)-5-(3,4-dimethoxyphenyl)-2,3-furandione (**1b**) with *N,N*-disubstituted urea derivatives **2** were investigated. The reactions afforded the 1*H*-pyrrole-2,3-dione. Then the following reactions of these compounds were performed with 1,2-phenylenediamines lead to 2(1*H*)-quinoxalinone derivatives (see Scheme 2). The structures of the compounds synthesized were characterized by IR, <sup>1</sup>H-, <sup>13</sup>C-NMR and elemental analysis studies.



**Scheme 2.** Synthesis of pyrrole-2,3-diones and their reaction with *o*-phenylenediamines

## 2. Results and discussion

As shown in Scheme 2, the novel pyrrole-2,3-dione compounds **3a-d** were synthesized by the cyclocondensation reactions of 4-aryl-5-aryl-furan-2,3-dione (**1a,b**) with asymmetric dialkylurea derivatives **2a,b** in 47-68% yields. The compounds **3** which include *p*-methyl or 3,4-dimethoxy groups in their structures, are new compounds synthesized in our laboratories by us and could be use as the substrates.



**Scheme 3.** Formation of pyrrol-2,3-diones and quinoxalin-2-ones

Firstly the reaction was carried out by Micheal-type attack of nitrogen atom of  $\text{NH}_2$  group of the urea derivative on C-5 in the furan-dione ring. Later molecule of water was eliminated and compound **3** was obtained. Nucleophilic addition of aromatic-1,2-diamines (**4**) to pyrrole-2,3-dione (**3a-d**) lead to quinoxalin-2-ones (**5a-d**) in 61-74% yields. These compounds arise from the sequential attacks of *o*-phenylenediamines at the C-3 and C-2 atom of pyrroledione, respectively, followed by elimination of water and pyrrole ring opening. And basic hydrolysis of this intermediate provide the final product **5**. A possible reaction scenario is outlined in Scheme 3.

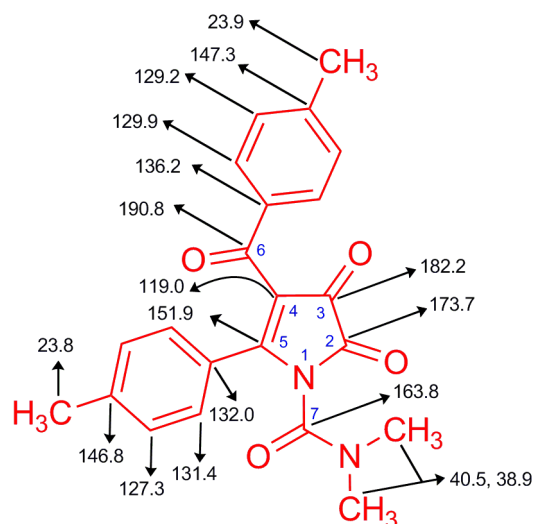
The compounds **5a** and **5c** are not novel.<sup>29-31</sup> These compounds were synthesized earlier. But the synthesis method describing in this work is new for these compounds.

There are four different carbonyl groups in compounds **3**. Each of them has different electronic environment. So, the infrared spectra are excellent evidences for identification of these groups. Compound **3a** showed absorption bands at 1774, 1722, 1701 and 1652  $\text{cm}^{-1}$  due to carbonyl compounds.

The  $^1\text{H-NMR}$  spectra of the  $\text{CDCl}_3$  solution of **3a** exhibited four singlets identified as methyl ( $\delta = 3.37, 3.17, 2.50, 2.44$ ). The signals of aromatic protons were observed as AA'BB' system at  $\delta$

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7.98-7.25 ppm. The structure of the compound **3a** was verified from  $^{13}\text{C}$ -NMR data compared with those of very close analogue <sup>20</sup> ( $\delta$  in bracket) obtained from a similar reaction employing 4-benzoyl-5-phenyl-2,3-dihydro-2,3-furandione instead of 4-(4-methylbenzoyl)-5-(4-methylphenyl)-2,3-dihydro-2,3-furandione: aroyl carbon at  $\delta$  190.80 (187.13), C<sub>3</sub> 182.20 (180.25), C<sub>2</sub> 173.67 (162.81), -NCON- 163.80 (160.94), C<sub>5</sub> 151.87 (151.00), aromatic carbons  $\delta$  147.31-127.25 (139.93-128.58), C<sub>4</sub> 119.02 (117.65), N-CH<sub>3</sub> 40.47, 38.94 (37.98, 37.74).



**Figure 1.** The  $^{13}\text{C}$ -NMR signals of **3a**

In the IR spectrum of **5a** characteristic absorption bands at 3244 ( $\text{NH} \rightleftharpoons \text{OH}$ ) and 1679 (s,  $\text{C}=\text{O}$ ) were observed.  $^1\text{H}$ -NMR spectrum of **5a** shows that  $\text{NH} \rightleftharpoons \text{OH}$  protons appears at 13.85 and 10.00 ppm, thus indicating a rapid and intense exchange process among several tautomeric forms. The  $^{13}\text{C}$ -NMR signals of **5a** showed two carbonyl carbons at  $\delta$  190.42 ( $\text{CO}$ , aroyl) and 157.40 ( $\text{C}=\text{O}$ ), a methyl carbon attached phenyl ring at  $\delta$  21.59. In the  $^{13}\text{C}$ -NMR spectra of **5a** aromatic signals of quinoxaline and phenyl moiety were clearly observed. Other spectral and analytical data of all synthesized compounds are in good agreement with their proposed structures.

### 3. Conclusion

In conclusion, the compounds synthesized are significant preliminary compounds due to the fact that original pyrrole-dione derivatives include 4-methylphenyl or 3,4-dimethoxyphenyl groups and reactive lactam groups in their structures. We think about that these compounds may be important from a medicinal point of view as well as their widespread biological significance.

### 4. Experimental

Melting points are uncorrected and recorded on Electrothermal 9200 digital melting point apparatus. Microanalyses were performed on a Leco-932 CHNS-O Elemental Analyser. A Jasco 460 FT-IR Spectrophotometer were used for IR spectra ( $4000\text{-}400\text{ cm}^{-1}$ ), using KBr pellets. The  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectra were measured with Varian XL Gemini 200 MHz or Bruker Avance III 400 MHz spectrometers and the chemical shifts were recorded in ppm units. The reactions were followed by TLC (Silica gel, aluminium sheets 60 F<sub>254</sub>, Merck). Solvents and all other chemical reagents were purchased from Merck, Sigma, Aldrich and Fluka. Solvents were dried by refluxing with the appropriate drying agents and distilled before use. The compounds **1** were prepared according to published method.<sup>32,33</sup>

#### 4.1. General Method for the Synthesis of Pyrrole-2,3-diones (3)

Corresponding *N,N*-disubstituted urea **2** (1 mmol) was added to magnetically stirred solution of 2,3-furandione derivatives **1** (1 mmol) in distilled benzene (30 mL). The mixture was refluxed for 4-6 hours. After the solvent was removed by evaporation, the oily residue was treated with dry diethyl ether to obtain crude pyrrole-2,3-dione **3**. Then, the crude products was purified with recrystallization from toluene or 2-propanol.

**1-(*N,N*-dimethylcarbonyl)-4-(4-methylbenzoyl)-5-(4-methylphenyl)-1*H*-pyrrole-2,3-dione (3a):** Yield 47% with a reflux time of 6 h, (recrystallized from toluene); mp 131 °C. IR (KBr, cm<sup>-1</sup>): = 3097-2900 cm<sup>-1</sup> (b, C-H, aromatic and aliphatic), 1774, 1722, 1701, 1652 (s, C=O), 1605-1440 (m, C=C, C=N, aromatic rings); <sup>1</sup>H-NMR δ (200 MHz, CDCl<sub>3</sub>): 7.98-7.25 (m, 8H, aromatic H), 3.37 (s, 3H, N-CH<sub>3</sub>), 3.17 (s, 3H, N-CH<sub>3</sub>), 2.50 (s, 3H, CH<sub>3</sub>), 2.44 ppm (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR δ (50 MHz, CDCl<sub>3</sub>): 190.8 (C<sub>6</sub>, aroyl), 182.2 (C<sub>3</sub>), 173.7 (C<sub>2</sub>), 163.8 (C<sub>7</sub>), 151.9 (C<sub>5</sub>), 147.3, 146.8, 136.2, 132.0, 131.4, 129.9, 129.2, 127.3 (aromatic C), 119.0 (C<sub>4</sub>), 40.5, 38.9 (N-CH<sub>3</sub>), 23.9, 23.8 ppm (CH<sub>3</sub>). Elemental Analysis: Calculated for C<sub>22</sub>H<sub>20</sub>N<sub>2</sub>O<sub>4</sub> (376.14): C, 70.20; H, 5.36; N, 7.44. Found: C, 69.94; H, 5.44; N, 7.41.

**1-(*N,N*-diethylcarbonyl)-4-(4-methylbenzoyl)-5-(4-methylphenyl)-1*H*-pyrrole-2,3-dione (3b):** Yield 49% with a reflux time of 4.5 h, (recrystallized from toluene); mp 137 °C. IR (KBr, cm<sup>-1</sup>): 3038-2875 cm<sup>-1</sup> (b, C-H, aromatic and aliphatic), 1777, 1718, 1697, 1648(s, C=O), 1605-1430 (m, C=C, C=N, aromatic ring); <sup>1</sup>H-NMR δ (400 MHz, CDCl<sub>3</sub>): 8.11-7.17 (m, 8H, aromatic H), 3.34 (q, *J* = 7.5 Hz, 2H, N-CH<sub>2</sub>), 3.18 (q, *J* = 7.4 Hz, 2H, N-CH<sub>2</sub>), 2.35 (s, 3H, CH<sub>3</sub>), 2.31 (s, 3H, CH<sub>3</sub>), 1.28 (t, *J* = 7.5 Hz, 3H, CH<sub>3</sub>), 1.05 ppm (t, *J* = 7.4 Hz, 3H, CH<sub>3</sub>). <sup>13</sup>C-NMR δ (100 MHz, CDCl<sub>3</sub>): 189.2 (C<sub>6</sub>, aroyl), 185.5 (C<sub>3</sub>), 161.7 (C<sub>2</sub>), 159.3 (C<sub>7</sub>), 152.1 (C<sub>5</sub>), 143.1, 138.7, 132.90, 132.8, 129.8, 129.4, 129.0, 127.2 (aromatic C), 92.5 (C<sub>4</sub>), 42.0 (N-CH<sub>2</sub>), 21.7 (CH<sub>3</sub>), 13.3 ppm (CH<sub>3</sub>). Elemental Analysis: Calculated for C<sub>24</sub>H<sub>24</sub>N<sub>2</sub>O<sub>4</sub> (404.17): C, 71.27; H, 5.98; N 6.93. Found: C, 71.16; H, 5.81; N, 6.82.

**1-(*N,N*-dimethylcarbonyl)-4-(3,4-dimethoxybenzoyl)-5-(3,4-dimethoxyphenyl)-1*H*-pyrrole-2,3-dione (3c):** Yield 68% with a reflux time of 4 h, (recrystallized from 2-propanol); mp 146 °C. IR (KBr, cm<sup>-1</sup>): ν = 3083-2847 (b, C-H, aromatic and aliphatic), 1771, 1710, 1698, 1655(s, C=O), 1597-1451 (C=C and C=N, aromatic rings), 1268 (C-O-C). <sup>1</sup>H-NMR δ (400 MHz, CDCl<sub>3</sub>): 7.62-6.76 (m, 6H, aromatic), 3.99, 3.88, 3.78, 3.63 (4 × s, 12H, OCH<sub>3</sub>), 3.38, 3.14 (2 × s, 6H, N-CH<sub>3</sub>). <sup>13</sup>C-NMR δ (100 MHz, CDCl<sub>3</sub>): 187.7 (C<sub>6</sub>, aroyl), 177.7 (C<sub>3</sub>), 170.0 (C<sub>2</sub>), 157.0 (C<sub>7</sub>), 154.5 (C<sub>5</sub>), 153.9, 150.3, 149.3, 149.2, 129.9, 125.9, 122.3, 120.2, 117.1, 111.3, 111.1, 110.7 (aromatic C), 92.1 (C<sub>4</sub>), 56.2, 56.1, 56.0, 55.7 (4 × OCH<sub>3</sub>), 38.4, 37.0 ppm (2 × N-CH<sub>3</sub>). Elemental analysis: Calculated for C<sub>24</sub>H<sub>24</sub>N<sub>2</sub>O<sub>8</sub> (468.46): C, 61.53; H, 5.16; N, 5.98. Found: C, 61.57; H, 5.36; N, 5.67.

**1-(*N,N*-diethylcarbonyl)-4-(3,4-dimethoxybenzoyl)-5-(3,4-dimethoxyphenyl)-1*H*-pyrrole-2,3-dione (3d):** Yield 54% with a reflux time of 6 h, (recrystallized from 2-propanol); mp 190 °C. IR (KBr, cm<sup>-1</sup>): ν = 3075-2843 (b, aromatic and aliphatic C-H stretching), 1766 (s), 1717 (s), 1699 (s), 1650 (s, C=O groups), 1594-1459 (C=C and C=N aromatic rings), 1266 (C-O-C stretching). <sup>1</sup>H-NMR δ (400 MHz, CDCl<sub>3</sub>): 7.63-6.70 (m, 6H, aromatic), 3.99, 3.95, 3.88, 3.70 (4 × s, 12H, OCH<sub>3</sub>), 3.28 (q, 4H, N-CH<sub>2</sub>), 1.17 (t, 6H, CH<sub>3</sub>). <sup>13</sup>C-NMR δ (100 MHz, CDCl<sub>3</sub>): 187.8 (C<sub>6</sub>, aroyl), 184.6 (C<sub>3</sub>), 160.8 (C<sub>2</sub>), 157.2 (C<sub>7</sub>), 153.8 (C<sub>5</sub>), 149.3, 149.2, 149.1, 129.9, 126.0, 122.8, 121.0, 111.1, 111.1, 110.7, 110.1, 109.7 (aromatic C), 91.7 (C<sub>4</sub>), 56.1, 55.8 (4 × OCH<sub>3</sub>), 43.6, 41.4 (2 × N-CH<sub>2</sub>), 13.9, 12.4 ppm (CH<sub>3</sub>). Elemental analysis: Calculated for C<sub>26</sub>H<sub>28</sub>N<sub>2</sub>O<sub>8</sub> (496.51): C, 62.89; H, 5.68; N, 5.64. Found: C, 62.57; H, 5.62; N, 5.24.

#### 4.2. General Method for the Synthesis of Quinoxalines 5a,d

Equimolar amounts of 2,3-pyrrolediones **3** and 1,2-phenylenediamines **4** in ethanol (30 mL) were heated, under reflux, for 15 min. Then 1 mL 2N NaOH solution was added and stirred at room temperature for 1 day. By adding 1N HCl solution, the acidity of reaction mixture was adjusted pH=4. Later it was kept in refrigerator for 1 hour. The obtained solid product was filtered off and washed with acetic acid and diethyl ether for purification.

**3-[2-(4-methylphenyl)-2-oxoethylidene]quinoxaline-2(1H)-one (5a)**: Yield 69%; mp: 247 °C. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu = 3244$  (NH  $\rightleftharpoons$  OH), 3042-2860 (C-H, aromatic and aliphatic), 1679 (C=O), 1606-1407 (C=C and C=N, aromatic rings).  $^1\text{H-NMR}$   $\delta$  (400 MHz,  $\text{CDCl}_3$ ): 13.85, 10.00 (b, NH  $\rightleftharpoons$  OH), 8.00-7.20 (m, 8H, aromatic), 7.00 (s, 1H, =CH-), 2.45 (s, 3H,  $\text{CH}_3$ ).  $^{13}\text{C-NMR}$   $\delta$  (100 MHz,  $\text{CDCl}_3$ ): 190.4 (Ar-C=O), 157.4 (C=O, amide), 157.0 (C-3), 144.6, 142.7, 136.3, 129.3, 127.6, 125.6, 125.1, 124.8, 123.8, 116.1, 115.6 (aromatic C), 91.0 (=CH-), 21.6 ( $\text{CH}_3$ ). Elemental analysis: Calculated for  $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_2$  (278.31 g/mol): C, 73.37; H, 5.07; N, 10.07. Found: C, 73.51; H, 4.98; N, 9.98.

**6,7-dimethyl-3-[2-(4-methylphenyl)-2-oxoethylidene]quinoxaline-2(1H)-one (5b)**: Yield 61%; mp: 315 °C. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu = 3154$  (NH  $\rightleftharpoons$  OH), 3101-2853 (C-H, aromatic and aliphatic), 1684 (C=O), 1601-1458 (C=C and C=N, aromatic rings).  $^1\text{H-NMR}$   $\delta$  (400 MHz,  $\text{CDCl}_3$ ): 13.90, 10.71, 9.23 (b, NH  $\rightleftharpoons$  OH), 8.00-6.86 (m, 6H, aromatic), 6.77 (s, 1H, =CH-), 2.47, 2.39, 2.33 (3  $\times$  s, 9H,  $\text{CH}_3$ ). Elemental analysis: Calculated for  $\text{C}_{19}\text{H}_{18}\text{N}_2\text{O}_4$  (306.36 g/mol): C, 74.49; H, 5.92; N, 9.14. Found: C, 74.53; H, 5.86; N, 8.87.

**3-[2-(3,4-dimethoxyphenyl)-2-oxoethylidene]quinoxaline-2(1H)-one (5c)**: Yield 74%; mp: 270 °C. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu = 3268$  (NH  $\rightleftharpoons$  OH), 3071-2780 (C-H, aromatic and aliphatic), 1681 (C=O), 1615-1472 (C=C and C=N, aromatic rings), 1268 (C-O-C).  $^1\text{H-NMR}$   $\delta$  (400 MHz,  $\text{CDCl}_3$ ): 13.77, 10.97, 9.44 (b, NH  $\rightleftharpoons$  OH), 7.97-6.90 (m, 7H, aromatic), 6.88 (=CH-), 3.94, 3.87 (2  $\times$  s, 6H,  $\text{OCH}_3$ ).  $^{13}\text{C-NMR}$   $\delta$  (100 MHz,  $\text{CDCl}_3$ ): 189.8 (Ar-C=O), 157.2 (C=O, amide), 154.6 (C-3), 155.1, 148.2, 143.8, 136.1, 127.6, 125.7, 124.8, 123.6, 121.8, 116.0, 115.3, 110.3 (aromatic C), 91.0 (=CH-), 56.1, 56.0 (2  $\times$   $\text{OCH}_3$ ). Elemental analysis: Calculated for  $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_4$  (324.33 g/mol): C, 66.66; H, 4.97; N, 8.64. Found: C, 66.69; H, 5.00; N, 8.58.

**6,7-dimethyl-3-[2-(3,4-dimethoxyphenyl)-2-oxoethylidene]quinoxaline-2(1H)-one (5d)**: Yield 65%; mp: 253 °C. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu = 3291$  (NH  $\rightleftharpoons$  OH), 3056-2853 (C-H, aromatic and aliphatic), 1673 (C=O), 1615-1466 (C=C and C=N, aromatic rings), 1268 (C-O-C).  $^1\text{H-NMR}$   $\delta$  (400 MHz,  $\text{CDCl}_3$ ): 13.84, 10.41, 9.00 (b, NH  $\rightleftharpoons$  OH), 7.86-6.86 (m, 5H, aromatic), 6.76 (s, 1H, =CH-), 4.04, 3.99 (2  $\times$  s, 6H,  $\text{OCH}_3$ ), 2.33, 2.28 (2  $\times$  s, 6H,  $\text{CH}_3$ ). Elemental analysis: Calculated for  $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_4$  (352.38 g/mol): C, 68.17; H, 5.72; N, 7.95. Found: C, 67.67; H, 5.51; N, 7.73.

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