GUANIDINE-ANNELATED HETEROCYCLES XVI. A PRACTICAL SYNTHESIS OF 7-CHLORO-4-PHENYLAMINO[1,2,4]TRIAZOLO-QUINAZOLIN-5(4H)-ONES

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Abstract - The triazolo[2,3-α]quinazolin-5(4H)-ones 9a-c can be prepared via Dimroth rearrangement by treatment of 5 with representative carboxylic acid orthoesters 6a-c each heating at 40 °C more elevated temperature than the preparation of their analogous triazolo[4,3-α] isomers 7a-c.

In previous reports, 4-phenylamino[1,2,4]triazolo[4,3-α]- and [2,3-α]quinazolin-5(4H)-ones 1,2 starting from isatoic anhydride, were performed as satisfactory antihypertensive agents. In order to study the synthesis of triazolo[2,3-α]quinazolin-5(4H)-ones via Dimroth rearrangement, 5-chloroisatoic anhydride (1) was employed as starting material to develop a cluster of new compounds and the key intermediate 5, as shown in Scheme 1, according to a known procedure. 2 At first, compound 5 was treated with representative carboxylic acid orthoesters, namely triethyl orthoformate, orthoacetate and orthopropionate (6a-c) (Scheme 2). Thus, reaction of 5 with an excess amount of triethyl orthoformate (6a) neatly under reflux at ca. 145 °C gave 7a in 51% yield as pink granules. The 1H NMR spectrum of 7a shows two singlet peaks at 9.11 and 9.54 ppm, which can be assigned to a NH proton of the 4-phenylamino group and a H-1 proton, respectively. The structure of 7a is also confirmed by its mass spectrum, m/z 311 (M+, 100%).

Scheme 1. Preparation of key intermediate 5 starting from 5-chloroisatoic anhydride (1).
Treatment of 5 with 6b and 6c under resembling conditions afforded the corresponding 1-methyl- and 1-ethyl-7-chloro-4-phenylamino[1,2,4]triazolo[4,3-a]quinazolin-5(4H)-one (7b,c) in 38% and 2% yield, respectively. The $^1$H NMR spectrum of 7b shows a three-proton singlet at $\delta$ 2.85 which can be assigned to the C1-methyl protons. Similarly, the $^1$H NMR spectrum of 7c shows a three-proton triplet at $\delta$ 1.31 and a two-proton quartet at $\delta$ 3.23 which can be assigned to the methyl and methylene protons of C1-ethyl group, respectively. It is interesting to note that the yields of these compounds decreased dramatically with the increase in carbon number of the 1-substituents, namely, 7a (R = H) in 51%, 7b (R = Me) in 38%, and 7c (R = CH$_2$Me) in 2%, perhaps due to the spatial hindrance and less benzene ring electron density, caused by chloro electron-withdrawing group, indirectly for the triazolo ring cyclization, comparing with their 4-phenylamino[1,2,4]triazolo[4,3-a]quinazolin-5(4H)-one counterparts (R = H, 82%; Me, 64%; CH$_2$Me, 53%). Further treatment of 5 with 6c stirring at 80, 100 and 120 °C for 9 h provided only trace 1-ethyl product 7c at 100 and 120 °C conditions, observed by TLC.

Scheme 2. Synthesis of 7-chloro-4-phenylamino[1,2,4]triazolo[4,3-a]- and [2,3-a]quinazolin-5(4H)-ones. Cyclocondensation of 5 with 6a-c was proposed to form the target compounds 7a-c in an angular fusion type in comparison with our study of linearly fused quinazolinone derivatives. These cyclized products were assigned in different fusion types and the assignments were made mainly based on the $^1$H NMR spectral analysis. The $^1$H NMR spectra showed that the NH proton at N-1 in the linear fusion type of those 3-substituted 1H-1,2,4-triazolo[3,4-b]quinazolin-5-ones exhibited much downfield protons (e.g. 11.22, 11.62 and 13.20 ppm), and the similar phenomena were also observed by Shawali and Sayed. On the other hand, the $^1$H NMR spectrum of corresponding NH proton of our cyclized products 7a-c each was observed at $\delta$ 9.10-9.20, which received almost no influence after the ring fusion and should be reasonably assigned to the NH proton of phenylamino side chain in 7a-c. Consequently, the linear isomers, 3-substituted 8-chloro-4-phenyl-1H-[1,2,4,5]tetrazino[6,1-b]quinazolin-6(4H)-ones (8a-c), were not yielded in these cyclocondensation reactions. Compounds 7a-c were neatly heating in pyridine or xylene at reflux under stringent conditions ca. 180 °C, but only starting material was recovered, as determined by $^1$H NMR spectroscopy. Thus compound 5 was brought into reaction with the corresponding cyclizing agents, e.g. 6a neatly, 6b in pyridine and 6c in
xylene by heating intentionally at more elevated temperature \(ca.~180~^\circ C\). The products were isolated in different crystalline form with different melting point and \(R_f\) value as compared with those of the corresponding 7a-c. It then suggested that the Dimroth rearrangement occurred actually under the above reaction conditions and structures of these products might be reasonably assigned to the corresponding 2-substituted 7-chloro-4-phenylamino[1,2,4]triazolo[2,3-a]quinazolin-5(4H)-one derivatives (9a-c). Nevertheless, this assignment was furthermore effectively confirmed by their spectral analyses.

All synthetic products were investigated by spectral, elemental analyses as well as high resolution mass spectrometry (HRMS). Differentiation of the structures of compounds 9a-c with those of compounds 7a-c was made especially on the basis of \(^1\)H NMR spectra. All signals of aromatic protons on the phenyl and quinazolinone ring of 9a-c and 7a-c clustered at \(\delta\) 6.76-7.24, 7.55-8.16 and at \(\delta\) 6.75-7.15, 7.83-8.21, respectively in the same region as expected. On the other hand, a signal of proton at C(2) of 9a appeared in relatively higher field at 9.21 ppm in contrast to that of the proton at C(1) of 7a at 9.54 ppm. Similar phenomena were also observed in those of compounds 9b,c in comparison with compounds 7b,c. Such a fact should originate from the location of the substituents in compounds 7a-c, which were exposed within the deshielding environment of the condensed quinazolinone nucleus. Thus, signals of these protons shifted to lower field, while those of the corresponding protons in compounds 9a-c remained in relatively higher field due to their extension on the opposite direction just beyond this region (Table 1).

**Table 1.** Differential spectral analysis of 7-chloro-4-phenylamino[1,2,4]triazolo[4,3-a]- and [2,3-a]quinazolin-5(4H)-ones

<table>
<thead>
<tr>
<th>Compd No.</th>
<th>R</th>
<th>(\delta) ppm</th>
<th>Compd No.</th>
<th>R</th>
<th>(\delta) ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>7a</td>
<td>H</td>
<td>9.54 (s)</td>
<td>9a</td>
<td>H</td>
<td>9.21 (s)</td>
</tr>
<tr>
<td>7b</td>
<td>Me</td>
<td>2.85 (s)</td>
<td>9b</td>
<td>Me</td>
<td>2.35 (s)</td>
</tr>
<tr>
<td>7c</td>
<td>Et</td>
<td>1.31 (t), 3.23 (q)</td>
<td>9c</td>
<td>Et</td>
<td>1.28 (t), 2.76 (q)</td>
</tr>
</tbody>
</table>

As to the phenylamino group, it expected little or no deshielding effect on the substituents of 9a-c, especially when a hydrogen bonding was formed between NH and C=O groups, which kept the aromatic nucleus extending more apart down- or upwards from the heterotricyclic plane. However, the benzene ring of phenylamino group of compounds 8a-c should have much stronger shielding effect on the substituents of 8a-c than those of 9a-c, especially the C3-H of 8a. The \(^1\)H NMR spectra of 1,2,4-triaryl-1H-imidazoles showed a singlet at \(\delta\) 7.58-8.02 which were assigned to be the C-5 proton next to phenylamino group based on Gust’s reports. Therefore, the consistent chemical shifts of these
two groups of compounds 7a-c and 9a-c demonstrate another unambiguous evidence showing these two type compounds not in a linear fusion model (8a-c). As usual, these two groups of compounds 7a-c and 9a-c were also investigated by mass spectral analyses, showing their expected molecular ions and different cleavage pattern. The identity of these compounds was examined by the elemental analyses or HRMS, and both of which showed that the expected compounds were presented definitely.

**Proposed Mechanisms**

The synthetic routes and structural assignment for 1-substituted 7-chloro-4-phenylamino[1,2,4]triazolo[4,3-a]quinazolin-5(4H)-ones (7a-c) and their corresponding 2-substituted 7-chloro-4-phenylamino[1,2,4] triazolo[2,3-a]quinazolin-5(4H)-one derivatives (9a-c) were described above in detail. A number of heterocycles, like pyrimidine,7-10 quinazoline3,11-15 as well as 1,3-benzothiazine16-18 ring systems, might undergo the Dimroth rearrangement under the action of acid or base at elevated temperature. However, the phenomenon of synthesizing compounds 9a-c via Dimroth rearrangement by treating reaction mixture only at elevated temperature was rarely studied.

**Scheme 3.** A proposed mechanism for preparation of 9a-c from the key intermediate 5.

So far there are three different proposed mechanisms possible for Dimroth rearrangement catalyzed by heat alone. A new method for preparation of a series of pyrazolo[4,3-e][1,2,4]triazolo[1,5-c]pyrimidines was reported by Mezheritsky and Starikova,19 which was carried out under severe conditions on heating their melts and gave the unexpected isomers via Dimroth rearrangement reaction. Thus our synthetic reaction might occur under the resembling mechanism to produce compounds 9a-c (Scheme 3). It is suggested that the initial step of the key intermediate 5 to triazole isomers 9a-c rearrangement involves tandem migration of hydride and a 1-[1,1-diethoxyalkyl] group (10 → 11). The N-1,1-diethoxyalkyl-N-2-quinazolinonylhydrazine (11) undergoes cleavage of the N-N bond with the intermediate formation of the tight ionic compound (12) of the resonance-stabilized anion and the aminium cation, which then transfers to the N-amine 13. The subsequent cyclocondensation of 13 affords the final products 9a-c. This proposed mechanism was also observed by treatment of 2-hydrazino-3,4-dihydro-2H-1,3-benzothiazin-4-one with trifluoroacetic anhydride in DMF (dimethylformamide) at 4 °C to give only the open-chain
2-trifluoroacetylhydrazino-3,4-dihydro-2H-1,3-benzothiazin-4-one, which was further heated in DMF or DMSO (dimethyl sulfoxide) under reflux to provide the ring-closure 2-trifluoromethyl-1,2,4-triazolo[5,1-b][1,3]benzothiazin-9-one.\(^{16}\)

![Scheme 4](image)

**Scheme 4.** Synthetic mechanism via the approach to the desired intermediate 14.

As shown in Scheme 4, 1-amino-6-chloro-2-imino-3-phenylamino-2,3-dihydroquinazolin-4(1H)-one (14) was thought to be the reaction intermediate for Dimroth rearrangement according to previous reports;\(^{11,15,20}\) however, treatment of 5 (mp = 198.5-199.5 °C) in xylene under reflux at ca. 210 °C for 10 h failed to give the desired intermediate 14 and most of starting material was obtained unchanged in 71% yield. The last one of these possible mechanisms for preparation of compounds 9a-c through isomerization of compounds 7a-c might be depicted in Scheme 5 according to Miller and Rose’s study.\(^7\) This proposed mechanism might involve a reactive zwitterionic intermediate 16 and the isomerization of triazole seems to proceed through a sequence of ring opening, rotation and ring closure reactions. Such reactions would be favored by the capacity of the triazole and benzene systems in the intermediate 16 to delocalize ions and relocate the triazole binding site to release the steric strain of side chain of compounds 7a-c. But treating compounds 7a-c each in pyridine or xylene under reflux at ca. 180 °C for 10 h failed to produce the expected corresponding compounds 9a-c.

![Scheme 5](image)

**Scheme 5.** An unlikely mechanism for synthesis of 9a-c via refluxing of 7a-c at 180 °C.

Therefore, the possible proposed mechanism for the synthesis of compounds 9a-c via Dimroth rearrangement catalyzed by heat alone might be depicted in Scheme 3, which indicates intermediates 10-13, the open-chain intermediates for cyclocondensation of 5 and corresponding carboxylic acid orthoesters 6a-c, as essential intermediates for the Dimroth rearrangement to form triazolo[2,3-a]quinazolin-5(4H)-ones 9a-c, the isomers of compounds 7a-c.
EXPERIMENTAL

All melting points were determined on electrothermal apparatus Büchi B-540 and were uncorrected. The UV and IR spectra were measured with Shimadzu UV-160A and FTIR-8700 spectrophotometer, respectively. The $^1$H NMR spectra were recorded on a Varian Gemini-300 RT spectrometer. The mass spectra were conducted on a Micromass TRIO-2000 GC-MS spectrometer. The HRMS were measured on a Finnigan MAT-95XL spectrometer. The elemental analyses were performed on a Perkin Elmer 240 EA.

7-Chloro-4-phenylamino[1,2,4]triazolo[4,3-a]quinazolin-5(4H)-one (7a). A mixture of 5 (0.5 g, 1.65 mmol) and triethyl orthoformate (6a) (1.46 g, 9.9 mmol) was heated under reflux (ca. 145 °C) for 5 h. After cooling, the precipitate was collected and recrystallized from EtOAc to give 7a (0.26 g, 51%), $R_f$ 0.18 (CHCl$_3$/acetone = 9:1), as pink granules, mp 252.5-253.5 °C. UV $\lambda_{\text{max}}$ nm (EtOH) (log $\varepsilon$): 273 (5.10); IR (KBr) cm$^{-1}$: 3250 (N-H), 3158 (C-H), 3138 (C-H), 1690 (C = O), 1602 (C=N/C=C), 1564 (C=N/C=C), 1240 (C-N), 753 (C-Cl); $^1$H-NMR (DMSO-$d_6$): $\delta$ 6.80 (2H, d, $J$ = 8.2 Hz, H-2', H-6'), 6.81 (1H, t, $J$ = 8.2 Hz, H-4'), 7.15 (2H, t, $J$ = 8.2 Hz, H-3', H-5'), 8.06 (1H, dd, $J$ = 2.4, 8.7 Hz, H-8), 8.13 (1H, d, $J$ = 2.4 Hz, H-6), 8.26 (1H, d, $J$ = 8.7 Hz, H-9), 9.11 (1H, s, NH), 9.54 (1H, s, H-1); EIMS 70 eV m/z (rel. int): 313 [M]$^+$ (33), 311 [M]$^+$ (100), 220 [MH-NHC$_6$H$_5$]$^+$ (54), 91 [NC$_6$H$_5$]$^+$ (35); Anal. Calcd for C$_{15}$H$_{10}$N$_5$OCl: C, 57.79; H, 3.23; N, 22.46. Found: C, 57.94; H, 3.17; N, 22.12.

7-Chloro-1-methyl-4-phenylamino[1,2,4]triazolo[4,3-a]quinazolin-5(4H)-one (7b). A mixture of 5 (0.5 g, 1.65 mmol) and triethyl orthoacetate (6b) (1.34 g, 8.3 mmol) in pyridine (5 mL) was heated under reflux (ca. 140 °C) for 15 h. After cooling, the reaction mixture was treated with EtOH (50 mL) and allowed to stand at 4 °C overnight. The precipitate was collected and recrystallized from EtOAc to give 7b (0.21 g, 38%), $R_f$ 0.10 (CHCl$_3$/acetone = 9:1), as pale yellow granules, mp 263.0-263.8 °C. UV $\lambda_{\text{max}}$ nm (EtOH) (log $\varepsilon$): 269 (4.97); IR (KBr) cm$^{-1}$: 3260 (N-H), 3028 (C-H), 3010 (C-H), 1678 (C=O), 1570 (C=N/C=C), 1250 (C-N), 751 (C-Cl); $^1$H-NMR (DMSO-$d_6$): $\delta$ 2.85 (3H, s, Me), 6.76 (2H, d, $J$ = 8.0 Hz, H-2', H-6'), 6.80 (1H, t, $J$ = 8.0 Hz, H-4'), 7.15 (2H, t, $J$ = 8.0 Hz, H-3', H-5'), 7.99 (1H, dd, $J$ = 2.4, 9.0 Hz, H-8), 9.10 (1H, s, NH); EIMS 70 eV m/z (rel. int): 313 [M]$^+$ (33), 311 [M]$^+$ (100), 220 [MH-NHC$_6$H$_5$]$^+$ (54), 77 [C$_6$H$_5$]$^+$ (100); Anal. Calcd for C$_{16}$H$_{12}$N$_5$OCl: C, 58.98; H, 3.71; N, 21.50. Found: C, 58.71; H, 3.77; N, 21.31.

7-Chloro-1-ethyl-4-phenylamino[1,2,4]triazolo[4,3-a]quinazolin-5(4H)-one (7c). A solution of 5 (0.4 g, 1.4 mmol) and triethyl orthopropionate (6c) (1.1 g, 6.2 mmol) in xylene (5 mL) was heated under reflux (130-140 °C) for 9 h. After cooling, evaporation of all solvents gave 0.45 g of red brown solids. Chromatography (EtOAc/n-hexane = 1:1) of crude product gave 7c, 11 mg (2%) $R_f$ 0.32 (CHCl$_3$/acetone = 9:1) as colourless solid, mp 172-173 °C, and 9c 0.298 g (64.1%). $^1$H-NMR (DMSO-$d_6$): $\delta$ 1.31 (3H, t, $J$ = 7.5 Hz, Me), 3.23 (2H, q, $J$ = 7.5 Hz, CH$_2$), 6.75 (2H, d, $J$ = 7.5 Hz, H-2', H-6'), 6.82 (1H, t, $J$ = 7.5 Hz, H-4'), 7.10 (2H, t, $J$ = 7.5 Hz, H-3', H-5'), 7.83 (1H, dd, $J$ = 2.4, 9.0 Hz, H-8), 8.16 (1H, d, $J$ = 9.0 Hz,
H-9), 8.21 (1H, d, J = 2.4 Hz, H-6), 9.20 (1H, s, NH); EIMS 70 eV m/z (rel. int): 341 [M]+ (9), 339 [M]+ (27), 248 [M-NC6H5]+ (15), 92 [C6H5NH]+ (100); HRMS found (M+) 339.0878, C17H14N5OCl requires (M) 339.0887.

7-Chloro-4-phenylamino[1,2,4]triazolo[2,3-a]quinazolin-5(4H)-one (9a). A mixture of 5 (253 mg, 0.84 mmol) and triethyl orthoformate (6a) (2.23 g, 15 mmol) was heated under reflux (ca. 180 °C) for 5 h. After cooling, the precipitate was collected and recrystallized from EtOAc to give 9a (117 mg, 45%), Rf 0.51 (CHCl3/acetone = 9:1), as red brown granules, mp 197-198 °C (dec.). IR (KBr) cm -1: 3355 (N-H), 3132 (C-H), 1715 (C=0), 1578 (C=N/C=C), 1217 (C-N), 756 (C-Cl); 1H-NMR (DMSO-d6): δ 6.78 (2H, d, J = 8.5 Hz, H-2', H-6'), 6.90 (1H, t, J = 8.5 Hz, H-4'), 7.24 (2H, t, J = 7.6 Hz, H-3', H-5'), 7.58 (1H, dd, J = 2.5, 7.6 Hz, H-8), 7.78 (1H, d, J = 2.5 Hz, H-6), 8.16 (1H, d, J = 7.6 Hz, H-9), 9.21 (1H, s, H-2), 9.63 (1H, s, NH); EIMS 70 eV m/z (rel. int): 313 [M]+ (7), 312 [MH]+ (45), 311 [M]+ (22), 154 [ClC6H3CONH2]+ (100); HRMS found (M+) 311.0574, C15H10N5OCl requires (M) 311.0574.

7-Chloro-2-methyl-4-phenylamino[1,2,4]triazolo[2,3-a]quinazolin-5(4H)-one (9b). A mixture of 5 (1.5 g, 5 mmol) and triethyl orthoacetate (6b) (2.43 g, 15 mmol) in pyridine (9 mL) was heated under reflux (ca. 180 °C) for 16 h. After cooling, the reaction mixture was treated with EtOH (35 mL) and allowed to stand at 4 °C overnight. The precipitate was collected and recrystallized from EtOH to give 9b (0.94 g, 63%), Rf 0.75 (CHCl3/acetone = 9:1), as pale yellow needles, mp 219-220 °C. UV λ max nm (EtOH) (log ε): 339 (5.05), 288 (5.48), 278 (5.45); IR (KBr) cm-1: 3556 (N-H), 3050 (=C-H), 2940 (C-H), 1698 (C=O), 1635 (C=N/C=C), 1600 (C=N/C=N), 1265 (C-N), 758 (C-Cl); 1H-NMR (DMSO-d6): δ 2.35 (3H, s, Me), 6.77 (2H, d, J = 7.5 Hz, H-2’, H-6’), 6.89 (1H, t, J = 7.5 Hz, H-4’), 7.22 (2H, t, J = 7.5 Hz, H-3’, H-5’), 7.56 (1H, d, J = 8.7 Hz, H-9), 7.74 (1H, d, J = 8.7 Hz, H-8), 8.14 (1H, s, H-6), 9.54 (1H, s, NH); 13C-NMR (DMSO-d6): δ 10.3 (Me), 112.7 (C-2’, C-6’), 118.8 (C-9a), 121.1 (C-9), 125.4 (C-4’), 127.3 (C-7), 128.0 (C-6), 129.4 (C-3’, C-5’), 134.3 (C-8), 145.6 (C-5a), 146.4 (C-3a), 147.4 (C-1’), 153.8 (C-2), 154.6 (C-5); EIMS 70 eV m/z (rel. int): 326 [MH]+ (25), 325 [M]+ (13), 154 [ClC6H3CONH2]+ (100); HRMS found (M+) 325.0730, C16H12N5OCl requires (M) 325.0730.

7-Chloro-2-ethyl-4-phenylamino[1,2,4]triazolo[2,3-a]quinazolin-5(4H)-one (9c). A solution of 5 (0.74 g, 2.4 mmol) and triethyl orthopropionate (6c) (1.3 g, 7.5 mmol) in xylene (6 mL) was heated under reflux (ca. 180 °C) for 9 h. After cooling, the reaction mixture was allowed to stand at 4 °C overnight. The precipitate was collected and recrystallized from EtOH to give 9c (0.43 g, 51%), Rf 0.80 (CHCl3/acetone = 9:1), as pale yellow granules, mp 127-127.5 °C. UV λ max nm (EtOH) (log ε): 339 (5.05), 288 (5.48), 278 (5.45); IR (KBr) cm-1: 3556 (N-H), 3050 (C-H), 2940 (C-H), 1698 (C-O), 1635 (C=N/C=C), 1600 (C=N/C=N), 1265 (C-N), 1265 (C=N/C=C), 1532 (C=N/C=C), 1250 (C-N), 1208 (C=N/C=C), 1250 (C-N), 1218 (C=N/C=C), 1250 (C-N), 1218 (C=N/C=C), 1250 (C-N), 1208 (C-N), 1188 (C-9a), 121.1 (C-9), 125.4 (C-4’), 127.3 (C-7), 128.0 (C-6), 129.4 (C-3’, C-5’), 134.3 (C-8), 145.6 (C-5a), 146.4 (C-3a), 147.4 (C-1’), 153.8 (C-2), 154.6 (C-5); EIMS 70 eV m/z (rel. int): 326 [MH]+ (25), 325 [M]+ (13), 154 [ClC6H3CONH2]+ (100); HRMS found (M+) 325.0730, C16H12N5OCl requires (M) 325.0730.

7-Chloro-2-ethyl-4-phenylamino[1,2,4]triazolo[2,3-a]quinazolin-5(4H)-one (9d). A solution of 5 (0.74 g, 2.4 mmol) and triethyl orthopropionate (6c) (1.3 g, 7.5 mmol) in xylene (6 mL) was heated under reflux (ca. 180 °C) for 9 h. After cooling, the reaction mixture was allowed to stand at 4 °C overnight. The precipitate was collected and recrystallized from EtOH to give 9d (0.43 g, 51%), Rf 0.80 (CHCl3/acetone = 9:1), as pale yellow granules, mp 127-127.5 °C. UV λ max nm (EtOH) (log ε): 339 (4.82), 287 (5.28); IR (KBr) cm-1: 3232 (N-H), 3048 (=C-H), 3010 (=C-H), 2940 (C-H), 1678 (C=O), 1632 (C=N/C=C), 1604 (C=N/C=C), 1250 (C-N), 1218 (C-N), 752 (C-Cl); 1H-NMR (DMSO-d6): δ 8.20 (1H, t, J = 7.5 Hz, CH3), 6.76 (2H, d, J = 7.7 Hz, H-2’, H-6’), 6.88 (1H, t, J = 7.7 Hz, H-4’), 7.22 (2H, d, J = 7.7 Hz, H-3’, H-5’), 7.75 (1H, d, J = 8.9 Hz, H-9), 7.73 (1H, dd, J = 2.5, 8.9 Hz, H-8), 8.15 (1H, d, J = 7.7 Hz, H-9), 7.73 (1H, dd, J = 2.5, 8.9 Hz, H-8), 8.15 (1H, d, J = 7.7 Hz, H-9), 7.73 (1H, dd, J = 2.5, 8.9 Hz, H-8), 8.15 (1H, d, J = 7.7 Hz, H-9), 7.73 (1H, dd, J = 2.5, 8.9 Hz, H-8), 8.15 (1H, d, J = 7.7 Hz, H-9), 7.73 (1H, dd, J = 2.5, 8.9 Hz, H-8), 8.15 (1H, d, J = 7.7 Hz, H-9), 7.73 (1H, dd, J = 2.5, 8.9 Hz, H-8), 8.15
(1H, d, J = 2.5 Hz, H-6), 9.51 (1H, s, NH); EIMS 70 eV m/z (rel. int): 341 [M]+ (13), 339 [M]+ (41), 248 [M-NC₆H₅]+ (10), 92 [C₆H₅NH]+ (100); Anal. Calcd for C₁₇H₁₄N₅OCl: C, 60.09; H, 4.15; N, 20.61. Found: C, 60.15; H, 4.25; N, 20.52.

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