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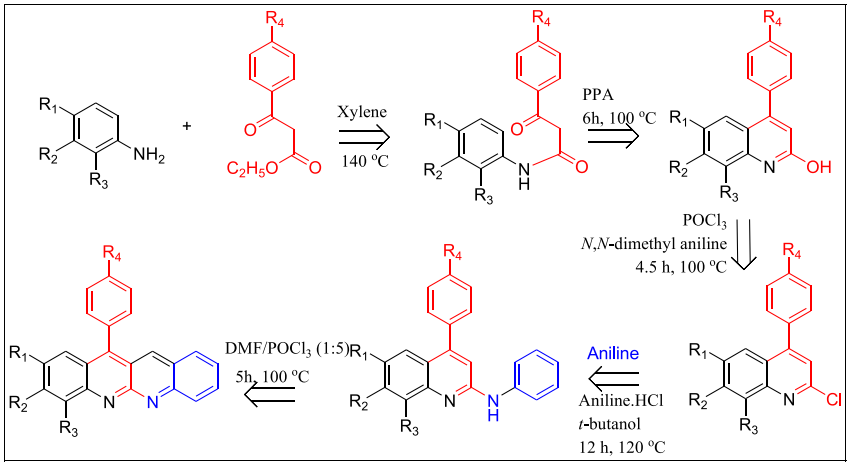

The Vilsmeier Haack heterocyclization of 2-aryl amino-4-phenyl quinolines quinoline yielded the hitherto unknown 5-phenyl-dibenzo[*b*,*g*][1,8]naphthyridines in quantitative yield. The synthesis of aryl amines was achieved by the action of anilines on 2-chloro-4-phenyl quinoline, which in turn was sourced through the combes reaction of benzoyl acetanilides.

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INTRODUCTION

Naphthyridines represent a group of six isomeric heterocyclic systems containing two fused pyridine rings with different mutual arrangement of nitrogen atoms. Among the isomers, [1,8]naphthyridines have received the most attention in the last 15 years as their skeleton is present in a large number of compounds isolated from natural sources. Their importance is reflected in the many routes to these compounds with more than 900 publications, which include 200 patents [1], in the last few years alone. They are the basis for the design of highly efficient antibacterial drugs such as enoxacin [2] and trovafloxacin [3–6]. A part from these, they are used as antitumor and DNA intercalating agents [7,8]. Phenyl-substituted [1,8]naphthyridines are especially reported to possess antitubercular properties [9].

Few methods are reported for the synthesis of dibenzo[*b*, *g*][1,8]naphthyridines [10,11]. Recently, our laboratory focused on the synthesis of dibenzo[*b*,*g*][1,8]naphthyridines through a novel one-step reaction [12] of 2-chloro-3-formyl quinolines with aryl amines as well as 1,2,3,4-tetrahydrodibenzo [*b*,*g*][1,8]naphthyridines [13] from 2-amino-3-formyl quinolines through a Friedlander type condensation reaction.

The Vilsmeier Haack reaction is one of the useful general methods employed for the formylation of various electron-rich aliphatic aromatic and heteroaromatic substrates [14]. Even though it is now used as a powerful tool for the construction of various heterocycles [15], the *in situ* or simultaneous formylation/cyclization process using the same is little explored. As a continuation of this research, we now report the new approaches toward the synthesis of 5-phenyl-dibenzo[b,g][1,8]naphthyridines (**5**) through the heterocyclization of 2-aryl amino-4-phenyl quinolines (**4**) using Vilsmeier reagent in a convenient and efficient process.

RESULTS AND DISCUSSION

The aryl amino quinolines (**4**), 2-chloro-4-Phenyl/(4′-methoxyphenyl)quinolines (**3**) were prepared through ethyl benzoyl acetate and ethyl 4′-methoxybenzoyl acetate, respectively. While the former was used as such, the latter was prepared form *p*-anisic acid using thionyl chloride.

The respective benzoyl acetanilides were prepared by the Knorr reaction [16], where in the benzoyl acetates were condensed with aniline at 140°C in xylene medium.

The attempted cyclization of the benzoyl acetanilides (**1**) to the 2-hydroxy 4-phenyl quinolines (**2**) in acid medium by the Combes process [17,18] gave average yields in sulfuric acid, while maximum yields was obtained through *in situ* prepared and activated polyphosphoric acid. Literature

reveals that K. Hino and co-workers [19] have prepared the 2-hydroxyl-4-(4′-methoxy)phenyl quinoline (**2g**) by the Camps quinoline syntheses route and found them to be antiulcer agents. Their reported yields were 67%. We got a yield of 96% of the same through the benzoyl acetanilide route by the Combes reaction [17,18].

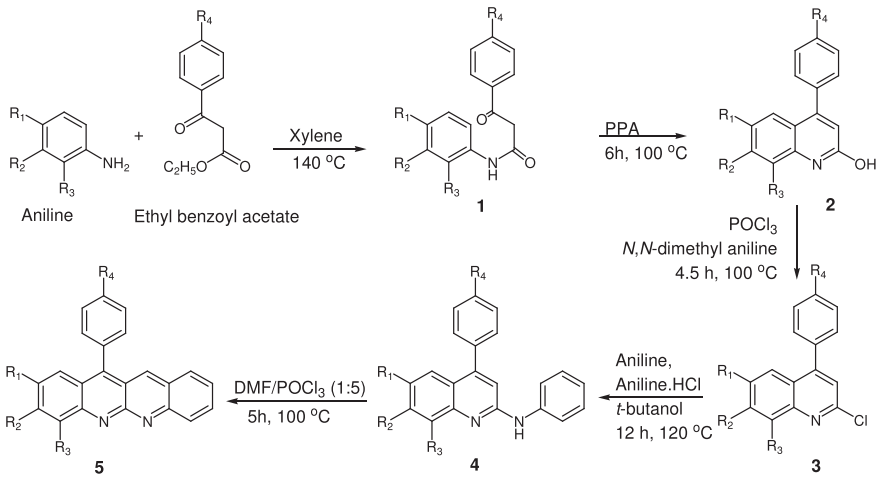
The 2-hydroxy-4-phenyl/(4′-methoxyphenyl)quinoline (**2**) were converted to the precursor 2-chloro-4-phenyl/(4′-methoxyphenyl)quinolines by the action of $POCl_3$ in the presence of *N*,*N*-dimethylaniline. To achieve the 2-aryl amino 4-phenyl/(4′-methoxyphenyl)quinoline, we attempted the condensation of (**3**) with aniline in ethanol medium in the presence of triethyl amine. It did not show any result even after several hours of reflux. On expectation that high temperature would initiate the reaction, keeping the reactants as such, the solvent was changed to *t*-butanol, and the mixture was heated on an oil bath for several hours. Here, some change was observed, but it was not very significant and well pronounced. A pinch of aniline hydrochloride was added to the reaction mixture, and here, after 12 h, the change was well defined and clear; moreover, the starting compound completely disappeared in thin-layer chromatography (TLC) showing that the addition of aniline hydrochloride accelerated the reaction, probably aniline hydrochloride helps in the removal of chlorine during its nucleophilic displacement by the aromatic amine.

Literature cites that 3-formyl-4-(*N*-phenylamino) pyridines [20,21] underwent cyclization easily in the presence of $POCl_3$ to give [1,6] naphthyridines; here, the formyl group was located at the ortho position to the aryl amino group on the pyridine ring.

In our case, it was felt that DMF/$POCl_3$ would be a very good formylating/cyclizing agent, where by both the process could be done simultaneously in a single step in achieving the titled compounds **5** from **4**. Here, the 2-aryl amino 4-phenyl/(4′-methoxyphenyl)quinoline (**4**) and the Vilsmeier adduct prepared in the ratio of [1:1:5 (1-reactant, 1-DMF and 5-$POCl_3$)] gave good results. Here, the $POCl_3$ was added to the reactant in DMF and not the usual way of reactant to the Vilsmeier adduct. A clear change in TLC was observed after heating on a water bath for 4.5–5 h. Further heating resulted in a decrease in the concentration of the observed new compound observed. After the usual work up, the compound was confirmed as the 5-phenyl-dibenzo[*b*,*g*][1,8] naphthyridine (**5**) by the spectral and the analytical technique.

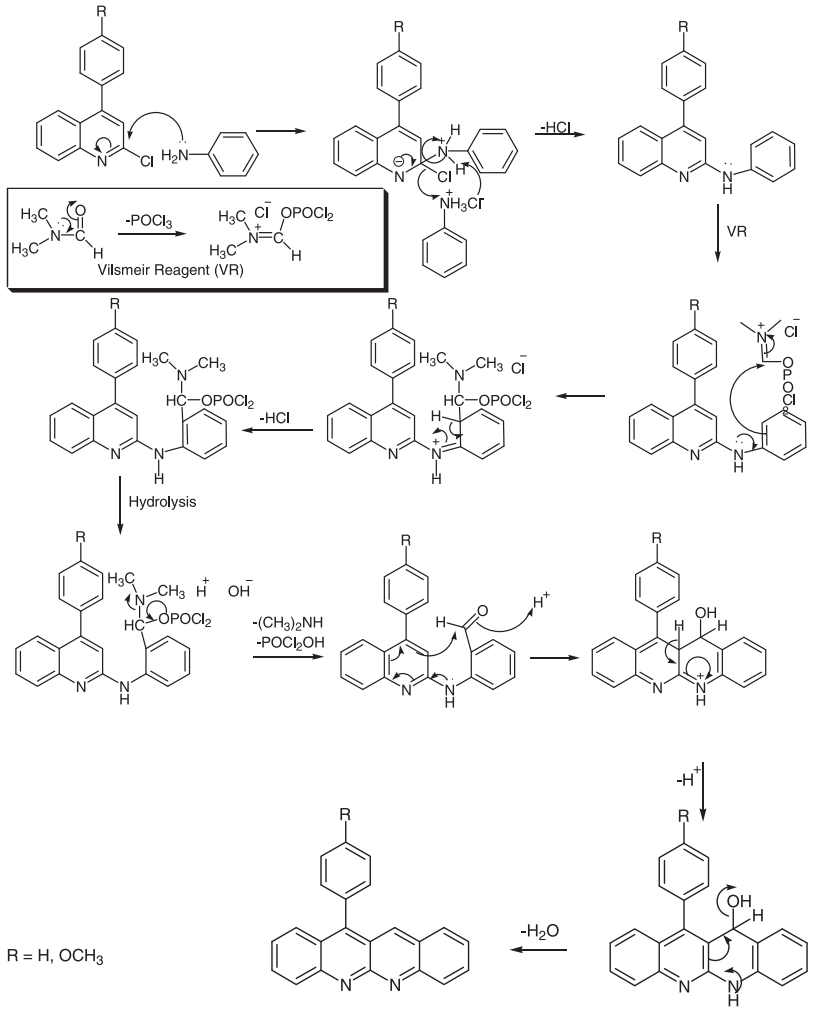
Here, the formylation was expected to take place on the ortho position of the *N*-phenyl ring rather than on the C_3-carbon of the quinoline ring, followed by cyclization with the C_3-proton on the quinoline ring. This could be rather explained on the basis of steric factor, where in the formylation on the quinoline ring is impeded by the 4-phenyl ring, while it is not so on the *N*-aryl side. This conclusion was drawn on the basis of our failed attempts to formylate the 2-hydroxy-4-phenyl quinoline (**2**) as well as the 2-hydroxy-3-dihydro-4-phenyl quinoline on the C_3-position by the Vilsmeier route. We always ended up with the 2-cloro-4-phenyl quinoline in both the attempts. In the reaction we were unable to isolate the aldehyde intermediate confirming that after formylation, the cyclization was simultaneous and immediate under the acidic Vilsmeier Haack reaction condition itself. The reaction was very well generalized and was extended to the other derivatives with moderate to very good yields (Scheme 1).

Scheme 1



(**a**) $R_1 = R_2 = R_3 = R_4 = H$; (**b**) $R_1 = CH_3$, $R_2 = R_3 = R_4 = H$; (**c**) $R_1 = H$, $R_2 = CH_3$, $R_3 = R_4 = H$; (**d**) $R_1 = R_2 = H$, $R_3 = CH_3$, $R_4 = H$; (**e**) $R_1 = CH_3$, $R_2 = H$, $R_3 = CH_3$, $R_4 = H$; (**f**) $R_1 = H$, $R_2 =$ -CH=CH-CH=CH- = R_3, $R_4 = H$; (**g**) $R_1 = R_2 = R_3 = H$, $R_4 = OCH_3$; (**h**) $R_1 = CH_3$, $R_2 = R_3 = H$, $R_4 = OCH_3$; (**i**) $R_1 = H$, $R_2 = CH_3$, $R_3 = H$, $R_4 = OCH_3$; (**j**) $R_1 = R_2 = H$, $R_3 = CH_3$, $R_4 = OCH_3$; (**k**) $R_1 = CH_3$, $R_2 = H$, $R_3 = CH_3$, $R_4 = OCH_3$; (**l**) $R_1 = OCH_3$, $R_2 = R_3 = H$, $R_4 = OCH_3$; (**m**) $R_1 = H$, $R_2 = OCH_3$, $R_3 = H$, $R_4 = OCH_3$; (**n**) $R_1 = R_2 = H$, $R_3 = R_4 = OCH_3$; (**o**) $R_1 = H$, $R_2 =$ -CH=CH-CH=CH- = R_3, $R_4 = OCH_3$

Mechanism. The whole of the reaction can be summarized in the probable mechanism.



EXPERIMENTAL

General. Ethyl-4-methoxy benzoyl acetate was sourced from Aldrich. Other reagents were obtained from local commercial sources. Melting points (mps) were determined on a Boetius microheating table and are uncorrected. TLC were performed on glass plates coated with silica gel-G incorporating 13% $CaSO_4$ as binder. Purification of the crude samples was carried out using chromatographic columns packed with silica gel (60–120 mesh) and neutral alumina. Infrared spectra were recorded on a Perkin-Elmer-597 Infrared Spectrophotometer as KBr pellets. ^{1}H-NMR spectra were recorded on an AMX-400 MHz NMR spectrophotometer using $CDCl_3$ as the solvent and Me_4Si as internal standard, and the chemical shifts are quoted in δ ppm. Mass spectra were recorded on an EI Autospec mass Spectrophotometer. Elemental analyses were performed on Cario-Elmer 1106 and Perkin-Elmer Analyzer. Microanalyses were obtained (C,H,N ± 0.4%).

4-Methoxy-benzoyl chloride. *p*-Anisic acid (25 g, 0.03289 mole) and thionyl chloride (24 mL, 0.03289 mole) were heated on a water bath for about 2 h. Excess thionyl chloride was striped under reduced pressure, and the residue was taken in dry benzene for the next step as such.

Ethyl-4-methoxybenzoyl acetate. Freshly distilled ethylacetoacetate (20 mL, 0.1580 mole) and toluene (30.4 mL) were added and cooled to 5°C in an ice bath. Out of 40 mL of 33% NaOH, 8 mL was added, and pH was raised to 11, and the mixture was stirred. To it, *p*-methoxybenzoylchloride (28 mL, 0.1642 mole) in benzene was added in drops, further the remaining NaOH solution (32 mL) were added, and the reaction mixture was stirred well at 35°C. After half an hour, the aqueous layer was separated, and to it, NaCl (11 g) and NH_4Cl (9.7 g) were added. The aqueous layer was collected and taken in a separating funnel, and to it, 40 mL of benzene was added. The organic layer was separated and dried using anhydrous sodium sulfate. The benzene was distilled under reduced

pressure. The yield of ethyl-4-methoxybenzoyl acetate was (24 mL, 70%).

Benzoyl acetanilide/4-methoxy-benzoyl acetanilide (1a–o). To ethylbenzoyl acetate (3.9 mL, 0.0225 mole)/ethyl 4′-methoxybenzoyl acetate (6 mL, 0.0225 mole) in a round bottom flask, aniline (2.1 mL, 0.0225 mole) was added. To this reaction mixture, xylene (6 mL) was added, and the whole of the reaction mixture was heated at 140°C on an oil bath for 4 h. After the completion of the reaction, the reaction mixture was cooled to allow crystallization to take place. To rinse out the xylene in the flask, 10 mL of petroleum ether was added twice. The product was washed with 30 mL of 1:1 petroleum ether and benzene. The washing was repeated three times. The benzoyl acetanilide/4′-methoxybenzoyl acetanilide was obtained as shining crystals.

2-Hydroxy-4-phenyl) quinoline/2-hydroxy-4-(4′-methoxy-phenyl)quinoline (2a–o). Benzoyl acetanilide (1.5 g, 0.0063 mole)/4′-methoxybenzoylacetanilide (1.50 g, 0.0056 mole) was added to *in situ* prepared and activated polyphosphoric acid 24 mL (15 g P_2O_5 + 4 mL H_3PO_4) taken in a round bottom flask. The reaction mixture was heated on a water bath for about 6 h. After the completion of the reaction, the reaction mixture was poured into crushed ice and stirred well. The precipitated 2-hydroxy-4-methoxy-phenylquinolines were filtered, washed well with water, and dried.

2-Chloro-4-phenylquinolines/2-chloro-4-(4′-methoxphenyl) quinolines (3a–o). 2-Hydroxy-4-phenylquinoline (1 g, 0.0045 mole)/2-hydroxy-4-(4′-methoxyphenyl)quinolines (1 g, 0.0037 mole), phosphorous oxychloride (6.28 mL, 0.08549 mole), and 2 drops of *N*,*N*-dimethyl aniline were taken in a round bottom flask and heated on a water bath for 4.5 h. After the completion of the reaction, the reaction mixture was poured into crushed ice and stirred well. The precipitated 2-chloro-4-phenyl quinolines/2-chloro-4-(4′-methoxyphenyl)quinolines were filtered, washed well with water, and dried. Pure chloro compound was obtained by column chromatography over silica gel using petroleum ether and ethyl acetate (98:2) as eluant.

General procedure for the synthesis of 2-(*N*-phenylamino)-4-phenyl)quinolines/2-(*N*-phenylamino)-4-(4′-methoxyphenyl) quinolines (4a–o). 2-Chloro-4-phenylquinoline (0.5 g, 0.00209 mole)/2-Chloro-4-(4′-methoxyphenyl)quinoline (0.5 mg 0.001855 mole), aniline (0.168 mL, 0.0018 mole), *t*-butanol(5 mL), Et_3N (0.00185 mole), and a pinch of aniline hydrochloride were taken in a round bottom flask fitted with a condenser. The mixture was refluxed on an oil bath for about 12 h at 120°C. After the appropriate time, the excess butanol was removed under reduced pressure. The product was recrystallised from petroleum ether and chloroform (8:2) mixture.

2-(N-phenylamino)-4-phenylquinoline (4a). The compound was obtained as white needles from petroleum ether: chloroform (8:2) (290 mg, 47%); mp 110°C; IR: 3210 (−NH), 1610 (−CN); ^{1}H-NMR δ: (7.95–7.21) (m, 14H, C_5-H, C_6-H, C_7-H, C_8-H and N-Ph H, C_4-PhH), 6.84(s, 1H, C_3-H), 6.65 (bs, 1H, −NH). *Anal.* Calcd for $C_{21}H_{16}N_2$: C, 85.11; H, 5.44; N, 9.45. Found: C, 85.05; H, 5.36; N, 9.43.

6-Methyl-2-(N-phenyl amino)-4-phenylquinoline (4b). The compound was obtained as white flakes from petroleum ether: chloroform (8:2) (400 mg, 65.39%); mp163°C; IR: 3290 (−NH), 1620 (−C═N); ^{1}H-NMR δ: 2.35 (s, 3H, C_6-CH_3), 6.62 (bs, 1H, −NH), 6.75 (s, 1H, C_3-H), 7.1–8.01 (m, 13H, C_5-H, C_7-H, C_8-H, C_4-ArH, 2-*N*-ArH). *Anal*. Calcd for $C_{22}H_{18}N_2$: C, 85.13; H, 5.84; N, 9.03. Found: C, 85.07, H, 5.76, N, 9.04.

7-Methyl-2-(N-phenylamino)-4-phenylquinoline (4c). The compound was obtained as white needles from petroleum ether: chloroform (8:2)(430 mg, 70.3%); mp 115°C; IR: 3225(−NH), 1615 (−C═N); ^{1}H-NMR δ: 2.52 (s, 3H, C_7-CH_3), 6.52 (bs, 1H, −NH), 6.9 (s, 1H, C_3-H), 7.3-7.79 (m, 13H, C_5-H, C_6-H, C_8-H, C_4-ArH, 2-*N*-ArH). *Anal*. Calcd for $C_{22}H_{18}N_2$: C, 85.13; H, 5.84; N, 9.03. Found: C, 85.09; H, 5.82; N, 8.95.

8-Methyl 2-(N-phenylamino)-4-phenylquinoline (4d). The compound was obtained as white needles from petroleum ether: chloroform (8:2)(430 mg, 70.5%); mp 157–58°C; IR: 3145 (−NH), 1608 (−CN); ^{1}H-NMR δ: 2.77(s, 3H, C_8-CH_3), 6.73(bs, 1H, −NH), 6.79(s,1H, C_3-H), 7.02–7.75 (m, 13H, C_5-H, C_6-H, C_7-H, C_4-ArH, 2-*N*-ArH). *Anal*. Calcd for $C_{22}H_{18}N_2$: C, 85.13; H, 5.84; N, 9.03. Found: C, 85.11; H, 5.81; N, 8.99; MS (EI) *m*/*z* (%): 311(30(M + 1), 310(100) M^+, 233(45).

6,8-Dimethyl-2-(N-phenylamino)-4-phenylquinoline (4e). The compound was obtained was white needles from petroleum ether: chloroform (8:2)(420 mg, 77.6%); mp 137°C; IR: 3345 (−NH), 1608.18 (−C═N); ^{1}H-NMR δ: 2.45 (s, 3H, C_6-CH_3), 2.72(s, 3H, C_8-CH_3), 6.60 (bs, 1H, −NH), 6.79 (s,1H, C_3-H), 7.25–8.12 (m, 12H, C_5-H, C_7-H, C_4-ArH, 2-*N*-ArH). *Anal*. Calcd for $C_{22}H_{20}N_2$: C, 85.15; H, 6.21; N, 8.64. Found: C, 85.10; H, 6.19; N, 8.61. See Supporting Information File 1 for full experimental data.

General procedure for the synthesis of 5-phenyl-dibenzo[*b*, *g*][1,8]naphthyridines/5-(4′-methoxyphenyl) dibenzo[*b*,*g*] [1,8] naphthyridines (5a–o). 2-(*N*-phenylamino)-4-phenylquinoline (0.282 g, 0.00092 mole)/2-(*N*-phenylamino)-4-(4′-ethoxyphenyl) quinoline (300 g, 0.00092 mole) and dimethyl-formamide (≈0.1 mL, 0.00092 mole) were taken in a round bottom flask and was added to it phosphorous oxychloride (0.43 mL, 0.0046 mole), drop wise. The reaction mixture was heated on a water bath for 5 h. After the completion of reaction, the whole of the reaction mixture was poured into crushed ice, the precipitated solid was filtered. Neutralization of the mother liquor using sodium bicarbonate yielded a further crop of the precipitate, which together was dried. The mixture was chromatographed over neutral alumina using petroleum ether: ethylacetate (95:5) as eluant, which furnished the pure compound.

5-Phenyl-dibenzo[b,g][1,8]naphthyridine (5a). The compound was obtained as pale yellow crystals in petroleum ether: chloroform (9:1)(210 mg, 74.67%); mp 123°C; IR: 1610 (−C═N); ^{1}H-NMR δ: 7.15–7.95(m, 13H, C_1-H, C_2-H, C_3-H, C_4-H, C_7-H, C_8-H, C_9-H, C_{10}-H, C_5-ArH), 9.01(s, 1H, C_6-H). *Anal*. Calcd for $C_{22}H_{14}N_2$: C, 86.25; H, 4.61; N, 9.14. Found: C, 86.18; H, 4.59; N, 9.15.

3-Methyl-5-phenyl-dibenzo[b,g][1,8]naphthyridin (5b). The compound was obtained as pale yellow crystals in petroleum ether: chloroform (9:1)(240 mg, 77.5%); mp 148°C; IR: 1615 (−C═N); ^{1}H-NMR δ: 2.5(s, 3H, C_3-CH_3), 7.2–7.95 (m, 12H, C_1-H, C_2-H, C_4-H, C_7-H, C_8-H, C_9-H, C_{10}-H, C_5-ArH), 8.95(s, 1H, C_6-H). *Anal*. Calcd for $C_{23}H_{16}N_2$: C, 86.22; H, 5.03; N, 8.75. Found: C, 86.18; H, 4.98; N, 8.72.

2-Methyl-5-phenyl-dibenzo[b,g][1,8]napthyridine (5c). The compound was obtained as pale yellow crystals in petroleum ether: chloroform (9:1) (210 mg, 67.8%); mp 181°C; IR: 1605.2 (−C═N). *Anal*. Calcd for $C_{23}H_{16}N_2$: C, 86.22; H, 5.03; N, 8.75. Found: C, 86.17; H, 4.97; N, 8.71.

1-Methyl-5-phenyl-dibenzo[b,g][1,8]naphthyridine (5d). The compound was obtained as pale yellow crystals in petroleum ether: chloroform (9:1)(250 mg, 80.7%); mp 118°C; IR: 1612.6 (−C═N);

^{1}H-NMR δ: 2.81(s, 3H, C_1-CH_3), 7.27–7.72(m, 12H, C_2-H, C_3-H, C_4-H, C_7-H, C_8-H, C_9-H, C_{10}-H, C_5-ArH), 8.97 (s, 1H, C_6-H). *Anal*. Calcd for $C_{23}H_{16}N_2$: C, 86.22; H, 5.03; N, 8.75. Found: C, 86.15; H, 4.96; N, 8.69.

1,3-Dimethyl-5-phenyl-dibenzo[b,g][1,8]napthyridine (5e). The compound was obtained as pale yellow crystals in petroleum ether: chloroform (9:1)(230 mg, 74.3%); mp 212°C; IR: 1620 (−C═N); ^{1}H-NMR δ: 2.42 (s, 3H, C_3-CH_3), 2.78 (s, 3H, C_1-CH_3), 6.9–8.23 (m, 12H, C_2-H, C_4-H, C_6-H, C_7-H, C_8-H, C_9-H, C_{10}-H and C_5-ArH). *Anal*. Calcd for $C_{24}H_{18}N_2$: C, 86.20; H, 5.42; N, 8.38. Found: C, 86.12; H, 5.37; N, 8.31. See Supporting Information File 2 for full experimental data.

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