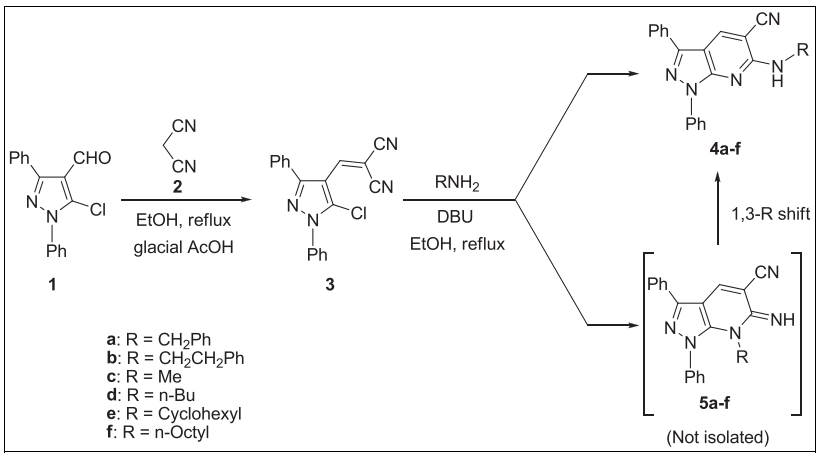
Facile Synthesis of New 6-Alkylamino-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitrile Derivatives

Nadieh Dorostkar-Ahmadi,[a] Abolghasem Davoodnia,[a]* Niloofar Tavakoli-Hoseini,[b] and Hossein Behmadi[a]

[a]Department of Chemistry, Mashhad Branch, Islamic Azad University, Mashhad, Iran
[b]Young Researchers and Elite Club, Mashhad Branch, Islamic Azad University, Mashhad, Iran
*E-mail: adavoodnia@mshdiau.ac.ir; adavoodnia@yahoo.com

Received April 16, 2018
DOI 10.1002/jhet.3285
Published online 00 Month 2018 in Wiley Online Library (wileyonlinelibrary.com).



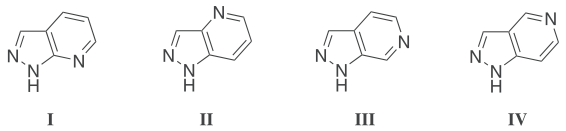
Some new derivatives of 6-alkylamino-1,3-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitrile have been prepared through a Knoevenagel condensation reaction of 5-chloro-1,3-diphenyl-1*H*-pyrazole-4-carbaldehyde with malononitrile in ethanol containing a few drops of glacial acetic acid at reflux temperature followed by cyclization with primary alkyl amines in the presence of 1,8-diazabicyclo[5.4.0] undec-7-ene as catalyst in refluxing ethanol. The products were characterized on the basis of IR, ^{1}H NMR, and ^{13}C NMR spectral and microanalytical data. The ^{1}H NMR data ruled out the formation of the alternative cyclized isomers 6-imino-7-alkyl-1,3-diphenyl-6,7-dihydro-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitriles.

INTRODUCTION

The presence of pyrazole and pyridine motifs, either alone or as a fused ring with other heterocyclic moieties, in a number of biological significant molecules has made them prime targets for scientific research. It has been known that certain pyrazoles possess important biological activities such as analgesics [1], anti-inflammatory [1,2], antimicrobial [3,4], antimalarial [4], antitumor [5,6], anti-hepatitis C virus [6], antiproliferative [7], antioxidants [8], antiangiogenic [9], and antiviral [10] properties. A number of these compounds have been considered as fungicides [11], and pesticides [12], and as the chelating and extracting reagents for different metal ions [13]. Pyrazoles are also not only of interest as important intermediates for the synthesis of fused heterocycles but also as efficient analytical regents in the complexation of transition-metal ions [14]. On the other hand, the pyridine moiety is the base of many bioactive molecules having important biological properties such as anticancer [15], antioxidant [15], anti-inflammatory [16], antimicrobial [17], antitubercular [18], antifungal [19], antileishmanial [20], antiproliferative [21], and anticonvulsant [22] activities. Because of the importance of these heterocycles, we became interested in the synthesis of new compounds formed from a combination of both pyrazole and pyridine scaffolds.

There are various bicyclic pyrazolopyridine scaffolds including pyrazolo[3,4-*b*] pyridine **I**, pyrazolo[4,3-*b*] pyridine **II**, pyrazolo[3,4-*c*] pyridine **III**, and pyrazolo[4,3-*c*] pyridine **IV** (Fig. 1), which are constructed from two fused pyrazole and pyridine rings. Among them, pyrazolo[3,4-*b*] pyridine core **I** has been relatively of more interest because of reported interesting biological properties such as antidiabetic [23], antiproliferative [24], antifungal [25], anticancer [26,27], antibacterial [28], anti-inflammatory [29], antiviral [30], and antimalarial [31] activities. Some of them have also inhibitory activities against fibroblast growth factor receptor kinase [32], TNF-α [33], IL-6 [33], CDK1 [34], and glycogen synthase kinase-3 [35]. A number of methods have already been proposed for the synthesis of pyrazolo[3,4-*b*] pyridines starting from pyrazole or pyridine moiety [36–44].

Taking all these facts into consideration, and in conjunction with our earlier studies of synthesis of



I II III IV

Figure 1. Structures of various pyrazolopyridines.

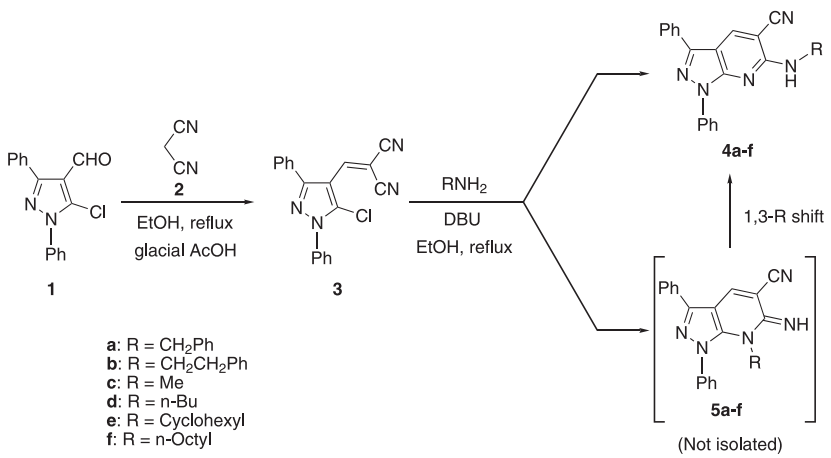
heterocyclic compounds [45–53], herein, we report the synthesis of some new derivatives of 6-alkylamino-1,3-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitrile **4a–f** in synthetically useful yields by Knoevenagel condensation of 5-chloro-1,3-diphenyl-1*H*-pyrazole-4-carbaldehyde **1** with malononitrile **2** in ethanol containing a few drops of glacial acetic acid at reflux temperature, which afforded compound **3**, followed by cyclization with primary alkyl amines in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) as catalyst in refluxing ethanol (Scheme 1). To the authors' knowledge, compounds **4a–f** are new and have not been reported in the literature.

RESULTS AND DISCUSSION

Our synthesis started from 5-chloro-1,3-diphenyl-1*H*-pyrazole-4-carbaldehyde **1** [54] which was converted to 2-((5-chloro-1,3-diphenyl-1*H*-pyrazol-4-yl)methylene) malononitrile **3** [55] when heated with malononitrile **2** in ethanol containing a few drops of glacial acetic acid at reflux temperature. The compound **3** was then allowed to interact with primary alkyl amines in the presence of DBU as catalyst in ethanol under reflux. Monitoring of the reactions with thin-layer chromatography (TLC) showed the formation of a product in each case, which was isolated from the reaction mixture as described in the Experimental section. The structural elucidation of the isolated products was based upon spectral and microanalytical data. For example, the ^{1}H NMR spectrum of the compound isolated from the reaction of benzyl amine with compound **3** in $CDCl_3$ showed a singlet at δ = 9.09 ppm belonging to the CH in pyridine ring and the characteristic signals at δ = 7.30–7.96 ppm for the aromatic protons. Surprisingly, as shown in the expanded view of ^{1}H NMR spectrum (Fig. 2), an A_2X splitting pattern for —NH—CH_2— group is seen which in the NH and CH_2 have been appeared as a broadened triplet at δ = 5.81 ppm and a doublet at δ = 4.89 ppm, respectively, with coupling constant (*J* value) of 5.8 Hz. Such splitting is in accord with structure **4a**, and not **5a**, which in the CH_2 group can be split *via* vicinal coupling with NH group. Such splitting is expected when the proton exchange in NH group is slow. In the Fourier transform infrared spectrum, the absorption bands for NH and cyano groups appeared at 3352 and 2209 cm^{-1}, respectively. Moreover, the characteristic signals in ^{13}C NMR spectrum at δ = 46.02, 69.14, 114.41, 114.87, 126.72, 127.56, 127.63, 127.79, 128.49, 128.76, 129.15, 129.45, 129.51, 131.34, 137.58, 138.65, 144.32, 148.01, 148.81, and 159.39 ppm are in accord with the structure **4a**. Finally, this compound gave satisfactory results of elemental analysis corresponding to the molecular formula $C_{26}H_{19}N_5$. All the other reactions proceed very cleanly to give the corresponding 6-alkylamino-1,3-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitrile in high yield, and no undesirable side-products were observed. The splitting between remained NH group with its vicinal protons rules out the isolation of the alternative cyclized isomers 6-imino-7-alkyl-1,3-diphenyl-6,7-dihydro-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitriles **5a–f** (Experimental section and Supplementary Information).

As illustrated in Scheme 2, the final isolated products **4a–f** can be formed through paths **A**, **B**, or **C**. It is proposed that initial nucleophilic attack of the deprotonated amino group onto the cyano moiety (paths **A** and **B**) or 5-position of pyrazole (path **C**) in compound **3** afforded the intermediates **I** or **II**, respectively.

Scheme 1. Synthesis of novel 6-alkylamino-1,3-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitriles **4a–f**.



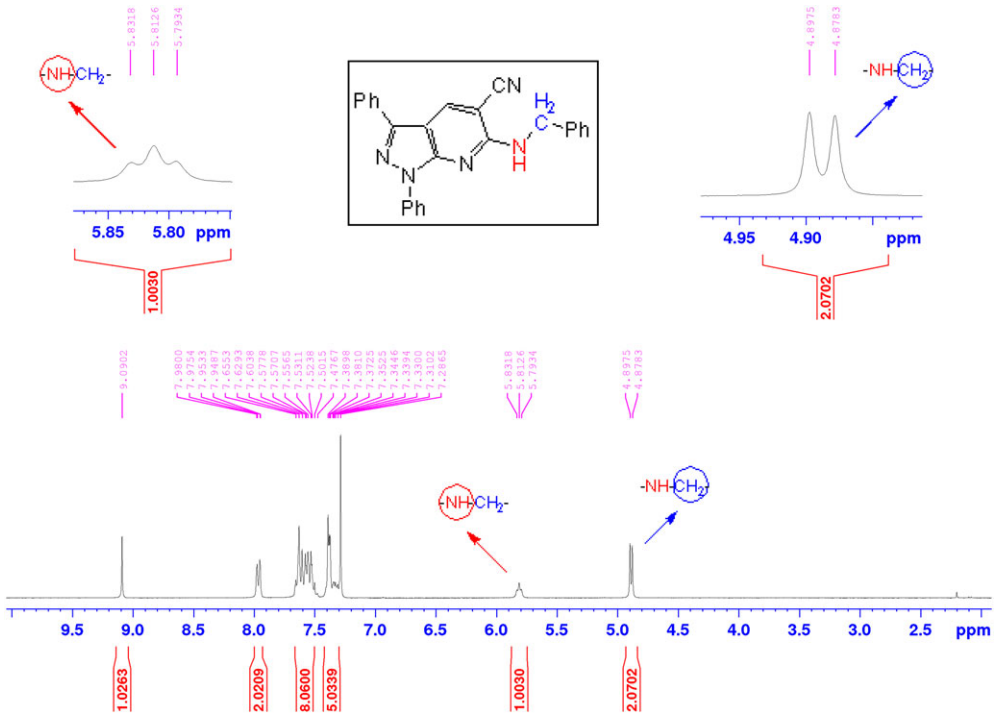
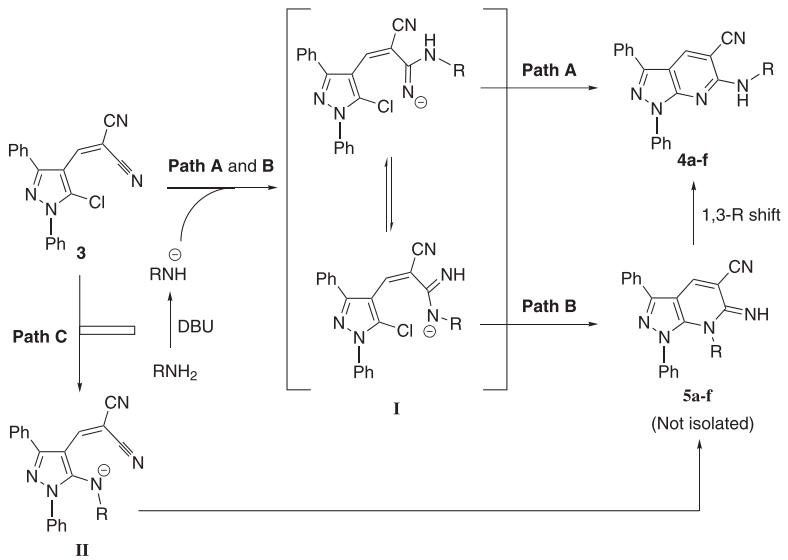


Figure 2. The ^{1}H NMR spectrum of compound **4a** in $CDCl_3$ and its expanded views. [Color figure can be viewed at wileyonlinelibrary.com]

Scheme 2. A plausible mechanism for the formation of compounds **4a–f**.



Subsequent cyclization of the intermediate **I**, in path **A**, affords the final products **4a–f**, whereas in paths **B** and **C**, the alternative cyclization of the intermediate **I** or **II** produces compounds **5a–f**, which finally undergoes 1,3-R migration to give the final products **4a–f**. Under the reaction conditions, however, attempts to isolate compounds **5a–f** failed even after careful monitoring of the reactions.

CONCLUSION

In summary, we have reported the synthesis of new 6-alkylamino-1,3-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitriles **4a–f** in good yields by the Knoevenagel condensation of 5-chloro-1,3-diphenyl-1*H*-pyrazole-4-carbaldehyde **1** with malononitrile **2** in refluxing ethanol containing a few drops of glacial acetic acid

followed by cyclization with primary alkyl amines in the presence of DBU as catalyst. The splitting between remaining NH group with its vicinal protons rules out the formation of the alternative cyclized isomers **5a–f**. A plausible mechanism was also suggested. All new products were characterized on the basis of IR, ^{1}H NMR, and ^{13}C NMR spectra and microanalytical data.

EXPERIMENTAL

Melting points were recorded on a Stuart SMP3 melting point apparatus (Staffordshire, UK). The IR spectra were obtained with KBr disks using a Tensor 27 Bruker spectrophotometer (Billerica, MA). The ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on a Bruker 300 FT spectrometer in $CDCl_3$ as solvent and using tetramethylsilane as internal standard. Elemental analysis was performed on a Thermo Finnigan Flash EA microanalyzer (Milan, Italy).

Synthesis of 2-((5-chloro-1,3-diphenyl-1*H*-pyrazol-4-yl) methylene) malononitrile 3. A mixture of 5-chloro-1,3-diphenyl-1*H*-pyrazole-4-carbaldehyde **1** (1 mmol), and malononitrile (1 mmol) in ethanol (5 mL) in the presence of a few drops of glacial acetic acid as a catalyst was heated under reflux for 5–6 h. The reaction was monitored by TLC. After the completion of the reaction, the mixture was cooled at room temperature, and the precipitate was filtered off. The crude product was recrystallized from ethanol to give the pure compound **3**. 0.31 g (95%), mp 182–184°C; IR: C≡N 2230 cm^{-1}; ^{1}H NMR: δ 7.77 (s, 1H, CH═C (CN)$_2$), 7.52–7.72 (m, 10H, arom-H) ppm; *Anal*. Calcd for $C_{19}H_{11}ClN_4$: C, 68.99; H, 3.35; N, 16.94. Found: C, 69.22; H, 3.17; N, 17.13.

General procedure for the synthesis of 6-alkylamino-1,3-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitriles 4a–f. A mixture of 2-((5-chloro-1,3-diphenyl-1*H*-pyrazol-4-yl)methylene) malononitrile **3** (1 mmol) and a primary alkyl amine (1 mmol) in ethanol (5 mL) in the presence of a few drops of DBU as a catalyst was heated under reflux for 2–3 h. Upon completion, monitored by TLC, the mixture was cooled at room temperature. The crude product was collected and recrystallized from ethanol to afford the pure compounds **4a–f** in high yields.

6-(Benzylamino)-1,3-diphenyl-1H-pyrazolo[3,4-b]pyridine-5-carbonitrile (4a). 0.32 g (80%), mp 194–196°C; IR: NH 3352, C≡N 2209 cm^{-1}; ^{1}H NMR: δ 4.89 (d, 2H, *J* = 5.8 Hz, NCH_2), 5.81 (t br., 1H, *J* = 5.8 Hz, NH), 7.30–7.67 (m, 13H, arom-H), 7.96 (dd, 2H, *J* = 8.0, 1.4 Hz, arom-H), 9.09 (s, 1H, pyridine CH) ppm; ^{13}C NMR: δ 46.02, 69.14, 114.41, 114.87, 126.72, 127.56, 127.63, 127.79, 128.49, 128.76, 129.15, 129.45, 129.51, 131.34, 137.58, 138.65, 144.32, 148.01, 148.81, 159.39 ppm; *Anal*. Calcd for $C_{26}H_{19}N_5$: C, 77.79; H, 4.77; N, 17.44. Found: C, 78.03; H, 4.91; N, 17.25.

6-(Phenethylamino)-1,3-diphenyl-1H-pyrazolo[3,4-b]pyridine-5-carbonitrile (4b). 0.34 g (83%), mp 190–191°C; IR: NH 3352, C≡N 2208 cm^{-1}; ^{1}H NMR: δ 2.99 (t, 2H, *J* = 7.1 Hz, CH_2), 3.90 (q, 2H, *J* = 6.8 Hz, NCH_2), 5.54 (t br., 1H, *J* = 5.6 Hz, NH), 7.23–7.65 (m, 13H, arom-H), 7.97 (dd, 2H, *J* = 8.1, 1.5 Hz, arom-H), 9.09 (s, 1H, pyridine CH) ppm; ^{13}C NMR: δ 36.09, 43.64, 69.02, 114.14, 114.83, 126.63, 126.70, 127.78, 128.74, 128.87, 129.12, 129.13, 129.43, 129.46, 131.38, 137.59, 138.80, 144.31, 147.97, 148.79, 159.49 ppm; *Anal*. Calcd for $C_{27}H_{21}N_5$: C, 78.05; H, 5.09; N, 16.86. Found: C, 77.79; H, 5.20; N, 16.69.

6-(Methylamino)-1,3-diphenyl-1H-pyrazolo[3,4-b]pyridine-5-carbonitrile (4c). 0.28 g (85%), mp 278–280°C; IR: NH 3363, C≡N 2207 cm^{-1}; ^{1}H NMR: δ 3.20 (d, 3H, *J* = 4.8 Hz, NCH_3), 5.49 (q br., 1H, *J* = 4.5 Hz, NH), 7.49–7.66 (m, 8H, arom-H), 7.95 (dd, 2H, *J* = 8.0, 1.6 Hz, arom-H), 9.09 (s, 1H, pyridine CH) ppm; ^{13}C NMR: δ 29.24, 68.90, 114.12, 115.02, 126.67, 127.76, 129.10, 129.12, 129.40, 129.43, 131.38, 137.61, 144.32, 148.00, 148.72, 160.16 ppm; *Anal*. Calcd for $C_{20}H_{15}N_5$: C, 73.83; H, 4.65; N, 21.52. Found: C, 73.62; H, 4.75; N, 21.70.

6-(Butylamino)-1,3-diphenyl-1H-pyrazolo[3,4-b]pyridine-5-carbonitrile (4d). 0.28 g (75%), mp 183–185°C; IR: NH 3351, C≡N 2206 cm^{-1}; ^{1}H NMR: δ 0.99 (t, 3H, *J* = 7.3 Hz, CH_3), 1.46 (sex, 2H, *J* = 7.2 Hz, CH_2), 1.67 (quin, 2H, *J* = 7.3 Hz, CH_2), 3.65 (q, 2H, *J* = 6.3 Hz, NCH_2), 5.46 (t br., 1H, *J* = 5.1 Hz, NH), 7.49–7.65 (m, 8H, arom-H), 7.95 (d, 2H, *J* = 7.1, arom-H), 9.06 (s, 1H, pyridine CH) ppm; ^{13}C NMR: δ 13.86, 20.08, 31.95, 42.13, 68.63, 113.96, 115.08, 126.68, 127.76, 129.10, 129.12, 129.39, 129.43, 131.39, 137.62, 144.37, 147.98, 148.78, 159.72 ppm; *Anal*. Calcd for $C_{23}H_{21}N_5$: C, 75.18; H, 5.76; N, 19.06. Found: C, 75.43; H, 5.64; N, 19.22.

6-(Cyclohexylamino)-1,3-diphenyl-1H-pyrazolo[3,4-b]pyridine-5-carbonitrile (4e). 0.30 g (75%), mp 179–181°C; IR: NH 3389, C≡N 2195 cm^{-1}; ^{1}H NMR: δ 1.20–2.15 (m, 10H, 5CH_2 in cyclohexyl), 4.10–4.26 (m, 1H, CH in cyclohexyl), 5.31 (d br., 1H, *J* = 7.8 Hz, NH), 7.48–7.65 (m, 8H, arom-H), 7.95 (dd, 2H, *J* = 7.9, 1.3 Hz, arom-H), 9.06 (s, 1H, pyridine CH) ppm; ^{13}C NMR: δ 25.03, 25.63, 33.45, 50.84, 68.56, 113.81, 115.07, 126.67, 127.75, 129.09, 129.10, 129.36, 129.40, 131.43, 137.65, 144.43, 147.93, 148.81, 158.99 ppm; *Anal*. Calcd for $C_{25}H_{23}N_5$: C, 76.31; H, 5.89; N, 17.80. Found: C, 76.58; H, 5.80; N, 17.61.

6-(Octylamino)-1,3-diphenyl-1H-pyrazolo[3,4-b]pyridine-5-carbonitrile (4f). 0.30 g (70%), mp 116–118°C; IR: NH 3350, C≡N 2207 cm^{-1}; ^{1}H NMR: δ 0.70–1.90 (m, 15H, $(CH_2)_6CH_3$), 3.50–3.80 (m, 2H, CH_2), 5.48 (s br., 1H, NH), 7.40–8.10 (m, 10H, arom-H), 9.06 (s, 1H, pyridine CH) ppm; ^{13}C NMR: δ 14.13, 22.68, 26.92, 29.25, 29.35, 29.87, 31.83, 42.44, 68.61, 113.95, 115.09, 126.68, 127.76, 129.10, 129.11, 129.39, 129.42, 131.41, 137.63, 144.36, 147.96, 148.78, 159.71 ppm; *Anal*. Calcd

for $C_{27}H_{29}N_5$: C, 76.56; H, 6.90; N, 16.53. Found: C, 76.31; H, 6.78; N, 16.67.

Acknowledgments. This work was supported by Islamic Azad University, Mashhad Branch, Iran.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.