Dyes and Pigments 86 (2010) 266–270

Contents lists available at ScienceDirect

Dyes and Pigments

journal homepage: www.elsevier.com/locate/dyepig

The synthesis of highly fluorescent heterocyclic compounds: Pyrido[2′,1′:2,3]imidazo[4,5-*b*]quinoline-12-yl cyanides

Mohammad Rahimizadeh*, Mehdi Pordel, Mehdi Bakavoli, Hossein Eshghi

Department of Chemistry, School of Sciences, Ferdowsi University of Mashhad, Mashhad 91775-1436, Iran

ARTICLE INFO

Article history:
Received 27 September 2009
Received in revised form
11 January 2010
Accepted 13 January 2010
Available online 1 February 2010

Keywords:
Quinolines
Imidazo[1,2-*a*]pyridine
Dye
Absorption
Emission
Fluorescence

ABSTRACT

The syntheses and fluorescence properties of novel derivatives of pyrido[2′,1′:2,3]imidazo[4,5-*b*]quinoline are described. The key intermediates (4-substituted)(3-hydroxyimino-2,3-dihydroimidazo[1,2-*a*]pyridin-2-yliden)methyl cyanides were synthesized by reacting the imidazo[1,2-*a*]pyridine with arylacetonitriles via nucleophilic substitution of hydrogen. The compounds were deep violet in colour. Acylation of hydroxyl group led to heterocyclization and gave highly fluorescent pyrido[2′,1′:2,3]imidazo[4,5-*b*]quinoline.

1. Introduction

Fluorescent heterocyclic compounds are of interest in many disciplines such as emitters for electroluminescence devices [1], molecular probes for biochemical research [2], in traditional textile and polymer fields [3], fluorescent whitening agents [4] and photo conducting materials [5]. Quinolines are well-known fluorescent compounds of high quantum yield and have attracted much attention owing to their potential high technology applications [6–9] including fluorescence probes in chemosensors [10,11]. Quinolines are, in general, comparatively easy to prepare, and numerous derivatives have been described for potential use as biologically active materials [12,13]. Several classical synthetic methods can be used to prepare quinolines, including the Skraup synthesis that involves heating aniline with glycerol and nitrobenzene in the presence of sulfuric acid [14], the Combes quinoline synthesis [15], Friedländer synthesis [16] and other methods [17–19]. Imidazo[1,2-*a*]pyridine derivatives are also well-known fluorescent compounds, as exemplified by imidazo[1,2-*a*]pyridinium salts, which are used to prepare styryl dyes [20] and imidazopyridine-7-nitrofurazan conjugates which have been utilized as fluorescent probes for the localization and function of the peripheral benzodiazepine receptor (PBR) [21]. Based on these aspects, it was decided to synthesize a heterocyclic system that contained both imidazo[1,2-*a*]pyridine and quinoline rings and to study their fluorescence properties. Continuing the authors' study of the nucleophilic substitution reactions of hydrogen [22–25], it was decided to use these reactions in the synthesis of novel derivatives of (3-hydroxyimino-2,3-dihydroimidazo[1,2-*a*]pyridin-2-yliden)-2-phenylacetonitrile **3a**–**e** as key intermediates. In this paper, a new method for the synthesis of several novel, highly fluorescent derivatives of pyrido[2′,1′:2,3]imidazo[4,5-*b*]quinolines **4a**–**e**, is described.

2. Experimental

2.1. Equipment and materials

Melting points were recorded on an Electrothermal type 9100 melting point apparatus. The IR spectra were obtained on a 4300 Shimadzu spectrometer and only noteworthy absorptions are listed. The ^{1}H NMR (500 MHz) and the ^{13}C NMR (125 MHz) spectra were recorded on a Bruker Avance DRX-500 Fourier transformer spectrometer. Chemical shifts are reported in ppm downfield from TMS as internal standard; coupling constance *J* are given in Hertz. The mass spectra were scanned on a Varian Mat CH-7 at 70 eV. Elemental analyses were performed on a Thermo Finnigan Flash EA microanalyzer. Absorption spectra were recorded on an Agilent

* Corresponding author. Tel.: +98 511 879 7022; fax: +98 511 879 6416.
E-mail address: rahimizh@yahoo.com (M. Rahimizadeh).

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doi:10.1016/j.dyepig.2010.01.013

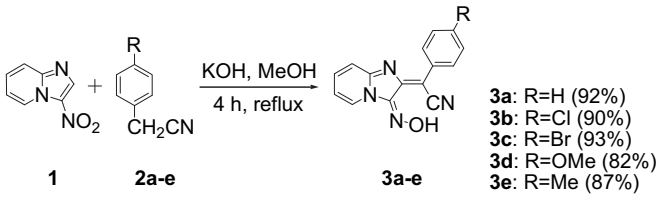

Fig. 1. Synthesis of new dyes **3a**–**e**.

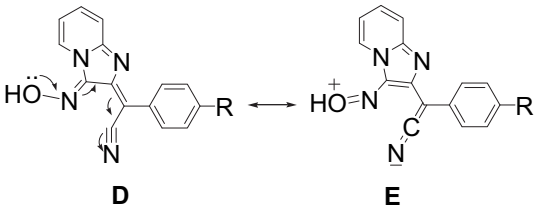

Fig. 3. A resonance from hydroxyl to cyanide group.

8453 spectrophotometer. Fluorescence spectra were recorded using Shimadzu RF-1501 spectrofluorophotometer. UV–vis and fluorescence scans were recorded from 350 to 700 nm. The fluorescence quantum yields (Φ_F) of compounds **4a**–**e** were determined *via* comparison methods, using fluorescein as a standard sample in 0.1 M NaOH and MeOH solution [26]. All measurements were carried out at room temperature. Compound **1** was obtained according to the published methods [27]. Other reagents were commercially available.

2.2. General procedure for the synthesis of *3a–e* from *1* to *2a–e*

Compounds **1** (10 mmol) and **2a**–**e** (12 mmol) were added with stirring to a solution of KOH (20 g, 357 mmol) in methanol (80 mL). The mixture was refluxed with stirring for 4 h, and then poured into water. After neutralization with dilute HCl solution, the precipitate was collected by filtration, washed with water, followed by EtOH and acetone, and then air dried to give pure **3a**–**e**.

2.2.1. 2-(3-Hydroxyimino-2,3-dihydroimidazo[1,2-a]pyridin-2-yliden)-2-phenylacetonitrile (*3a*)

Compound **3a** was obtained as dark red powder, yield (2.41 g, 92%), mp 195–197 °C; ^{1}H NMR (DMSO-d_6) δ 6.58 (dd, J = 8.6 Hz, J' = 7.0 Hz, 1H), 7.03 (d, J = 9.2 Hz, 1H), 7.13–7.72 (m, 4H), 8.13 (d, J = 8.4 Hz, 2H), 9.03 (d, J = 7.0 Hz, 1H), 13.35 (br s, 1H); IR (KBr): 3450 cm^{-1} (OH), 2196 cm^{-1} (CN). MS (m/z) 262 (M$^+$). Anal. Calcd for $C_{15}H_{10}N_4O$ (262.3): C, 68.69; H, 3.84; N, 21.36. Found: C, 68.35; H, 3.70; N, 21.14.

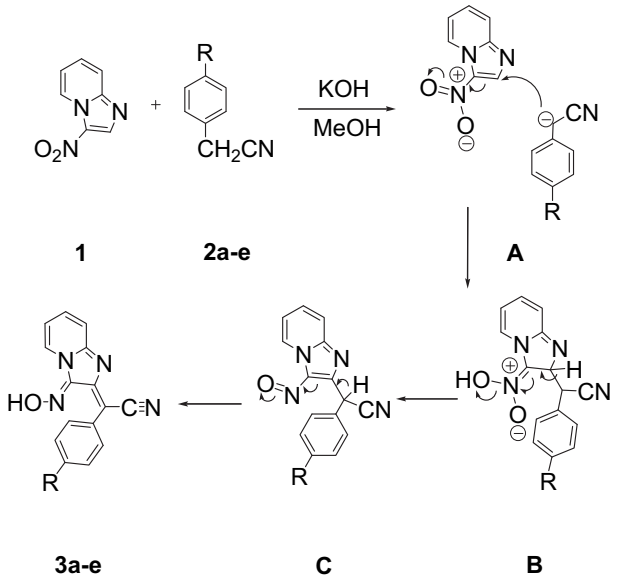

Fig. 2. Proposed reaction mechanism for the synthesis of prepared dyes **3a**–**e**.

2.2.2. (4-Chlorophenyl)(3-hydroxyimino-2,3-dihydroimidazo[1,2-a]pyridin-2-yliden)methyl cyanide (*3b*)

Compound **3b** was obtained as dark red powder, yield (2.66 g, 90%), mp 217–219 °C; ^{1}H NMR (DMSO-d_6) δ 6.58 (dd, J = 8.6 Hz, J' = 6.8 Hz, 1H), 7.08 (d, J = 8.9 Hz, 1H), 7.44 (d, J = 8.7 Hz, 2H), 7.62 (dd, J = 8.9 Hz, J' = 8.6 Hz, 1H), 8.16 (d, J = 8.7 Hz, 2H), 9.01 (d, J = 6.8 Hz, 1H), 13.51 (br s, 1H); IR (KBr): 3455 cm^{-1} (OH), 2195 cm^{-1} (CN). MS (m/z) 296 (M$^+$), 298 (M$^+$ + 2). Anal. Calcd for $C_{15}H_9ClN_4O$ (296.7): C, 60.72; H, 3.06; N, 18.88. Found: C, 60.41; H, 2.91; N, 18.63.

2.2.3. (4-Bromophenyl)(3-hydroxyimino-2,3-dihydroimidazo[1,2-a]pyridin-2-yliden)methyl cyanide (*3c*)

Compound **3c** was obtained as dark red powder, yield (3.17 g, 93%), mp 219–220 °C; ^{1}H NMR (DMSO-d_6) δ 6.60 (dd, J = 8.6 Hz, J' = 6.9 Hz, 1H), 7.08 (d, J = 9.1 Hz, 1H), 7.55–7.70 (m, 3H), 8.10 (d, J = 8.7 Hz, 2H), 9.01 (d, J = 6.9 Hz, 1H), 13.51 (br s, 1H); IR (KBr): 3455 cm^{-1} (OH), 2195 cm^{-1} (CN). MS (m/z) 341 (M$^+$), 343 (M$^+$ + 2). Anal. Calcd for $C_{15}H_9BrN_4O$ (341.1): C, 52.81; H, 2.66; N, 16.42. Found: C, 52.53; H, 2.51; N, 16.69.

2.2.4. (3-Hydroxyimino-2,3-dihydroimidazo[1,2-a]pyridin-2-yliden)(4-methoxyphenyl)methyl cyanide (*3d*)

Compound **3d** was obtained as dark red powder, yield (2.39 g, 82%), mp 175–177 °C; ^{1}H NMR (DMSO-d_6) δ 3.76 (s, 3H), 6.50 (dd, J = 8.6 Hz, J' = 6.8 Hz, 1H), 7.02 (d, J = 8.8 Hz, 2H), 7.12 (d, J = 9.2 Hz, 1H), 7.52 (dd, J = 9.2 Hz, J' = 8.6 Hz, 1H), 8.07 (d, J = 8.8 Hz, 2H), 8.95 (d, J = 6.8 Hz, 1H), 13.27 (br s, 1H); IR (KBr): 3450 cm^{-1} (OH), 2197 cm^{-1} (CN). MS (m/z) 292 (M$^+$). Anal. Calcd for $C_{16}H_{12}N_4O_2$ (292.3): C, 65.75; H, 4.14; N, 19.17. Found: C, 65.40; H, 3.99; N, 18.86.

2.2.5. (3-Hydroxyimino-2,3-dihydroimidazo[1,2-a]pyridin-2-yliden)(4-methylphenyl)methyl cyanide (*3e*)

Compound **3e** was obtained as dark red powder, yield (2.40 g, 87%), mp 173–175 °C; ^{1}H NMR (DMSO-d_6) δ 2.31 (s, 3H), 6.54 (dd, J = 8.6 Hz, J' = 6.9 Hz, 1H), 7.06 (d, J = 9.2 Hz, 1H), 7.23 (d, J = 8.2 Hz, 2H), 7.58 (dd, J = 9.2 Hz, J' = 8.6 Hz, 1H), 8.03 (d, J = 8.2 Hz, 2H), 8.97 (d, J = 6.9 Hz, 1H), 13.34 (br s, 1H); IR (KBr): 3450 cm^{-1} (OH), 2197 cm^{-1} (CN). MS (m/z) 276 (M$^+$). Anal. Calcd for $C_{16}H_{12}N_4O$

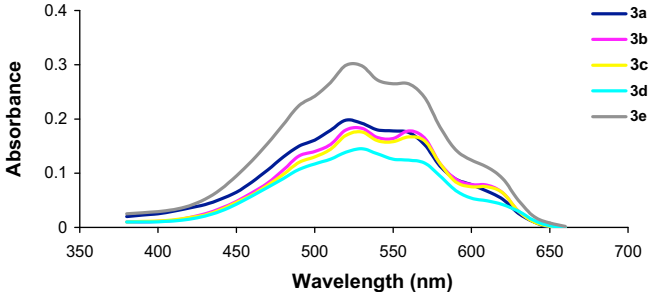

Fig. 4. Visible absorption spectrum of compounds **3a**–**e** in dilute (3×10^{-5} M) ethanol solution.

Table 1
Spectroscopic properties of dyes **3a**–**e** in EtOH solvent.

Dye	3a	3b	3c	3d	3e
λ_{max} (nm)[a]	525	527	528	530	527
$\varepsilon \times 10^{-3}$ (M^{-1} cm^{-1})[b]	10.35	9.85	9.35	7.65	15.60

[a] Wavelengths of maximum absorbance (λ_{max}).
[b] Extinction coefficient.

(276.3): C, 69.55; H, 4.38; N, 20.28. Found: C, 69.21; H, 4.23; N, 20.19.

2.3. General procedure for the synthesis of *4a–e* from *3a–e*

Acetyl chloride (0.55 g, 7 mmol) was added with stirring to a solution of **3a**–**e** (5 mmol) in dry pyridine (50 mL). After the addition was completed, the mixture was refluxed for 4 h. The pyridine was evaporated under reduced pressure. The resulting solid residue was boiled in EtOH for 5 min. The precipitated solid product was collected by filtration and washed well with water and acetone to give pure compounds **4a**–**e**.

2.3.1. Pyrido[2′,1′:2,3]imidazo[4,5-b]quinolin-12-yl cyanide (*4a*)

Compound **4a** was obtained as shiny yellow needles (pyridine), yield (1.09 g, 90%), mp 280–282 °C; ^{1}H NMR ($CDCl_3$) δ 6.92–7.12 (m, 1H), 7.62–7.92 (m, 4H), 8.18–8.55 (m, 2H), 8.97 (d, $J = 6.8$ Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$): δ 102.53, 107.34, 109.35, 118.25, 120.59, 122.34, 129.47, 135.43, 136.75, 137.50, 145.33, 146.34, 147.90, 148.51, 149.12 ppm; IR (KBr): 2198 cm^{-1} (CN). MS (m/z) 244 (M^+). Anal. Calcd for $C_{15}H_8N_4$ (244.3): C, 73.76; H, 3.30; N, 22.94. Found: C, 73.41; H, 3.20; N, 22.71.

2.3.2. 3-Chloropyrido[2′,1′:2,3]imidazo[4,5-b]quinolin-12-yl cyanide (*4b*)

Compound **4b** was obtained as shiny yellow needles (pyridine), yield (1.28 g, 92%), mp 325–327 °C; ^{1}H NMR ($CDCl_3$) δ 6.98–7.22 (m, 1H), 7.72–7.93 (m, 3H), 8.28 (d, $J = 8.8$ Hz, 1H), 8.53 (d, $J = 1.9$ Hz, 1H), 8.96 (d, $J = 6.9$ Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$): δ 102.31, 107.21, 109.41, 118.40, 121.39, 122.21, 131.18, 133.01, 135.27, 136.83, 137.91, 146.02, 147.21, 148.09, 149.20 ppm; IR (KBr): 2199 cm^{-1} (CN). MS (m/z) 278 (M^+), 280 ($M^+ + 2$). Anal. Calcd for $C_{15}H_7ClN_4$ (278.7): C, 64.64; H, 2.53; N, 20.10. Found: C, 64.29; H, 2.41; N, 19.88.

2.3.3. 3-Bromopyrido[2′,1′:2,3]imidazo[4,5-b]quinolin-12-yl cyanide (*4c*)

Compound **4c** was obtained as shiny yellow needles (pyridine), yield (1.50 g, 93%), mp 331–332°C; ^{1}H NMR ($CDCl_3$) δ 7.02–7.19 (m, 1H), 7.65–7.89 (m, 3H), 8.30 (d, $J = 8.8$ Hz, 1H), 8.51 (d, $J = 1.9$ Hz, 1H), 8.95 (d, $J = 6.9$ Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$): δ 103.50, 109.11, 109.63, 118.31, 119.81, 121.70, 133.91, 133.99, 136.14, 137.61, 139.32, 146.09, 147.00, 148.56, 149.35 ppm; IR (KBr): 2199 cm^{-1} (CN). MS (m/z) 323 (M^+), 325 ($M^+ + 2$). Anal. Calcd for $C_{15}H_7BrN_4$ (323.1): C, 55.75; H, 2.18; N, 17.34. Found: C, 55.43; H, 2.03; N, 17.16.

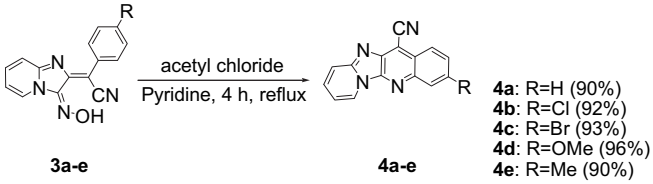

Fig. 5. Synthesis of new fluorescent compounds **4a**–**e**.

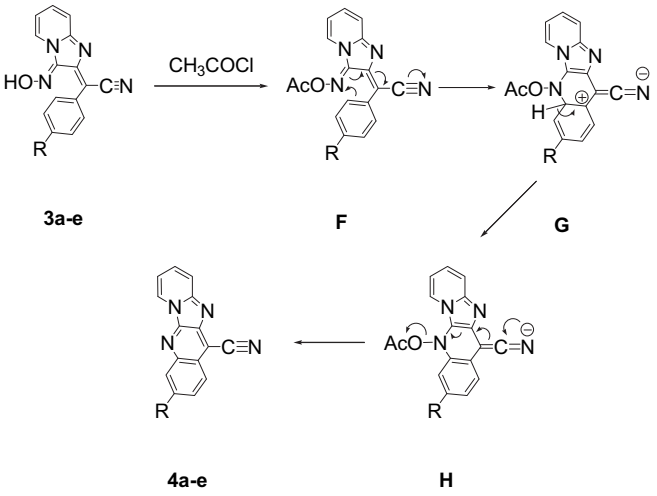

Fig. 6. Proposed reaction mechanism for the synthesis of prepared compounds **4a**–**e**.

2.3.4. 3-Methoxypyrido[2′,1′:2,3]imidazo[4,5-b]quinolin-12-yl cyanide (*4d*)

Compound **4d** was obtained as shiny orange needles (pyridine), yield (1.31 g, 96%), mp 305–307 °C; ^{1}H NMR ($CDCl_3$) δ 4.04 (s, 3H), 6.93–7.12 (m, 1H), 7.47 (dd, $J = 9.4$ Hz, $J' = 2.4$ Hz, 1H), 7.60 (d, $J = 2.4$ Hz, 1H), 7.66–7.8 (m, 2H), 8.31 (d, $J = 9.4$ Hz, 1H), 8.91 (d, $J = 6.9$ Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 56.54, 103.81, 109.69, 110.74, 113.51, 118.91, 123.12, 133.02, 133.90, 137.69, 139.00, 143.57, 146.65, 147.50, 149.14, 160.30 ppm; IR (KBr): 2198 cm^{-1} (CN). MS (m/z) 274 (M^+). Anal. Calcd for $C_{16}H_{10}N_4O$ (274.3): C, 70.07; H, 3.67; N, 20.43. Found: C, 69.71; H, 3.50; N, 20.19.

2.3.5. 3-Methylpyrido[2′,1′:2,3]imidazo[4,5-b]quinolin-12-yl cyanide (*4e*)

Compound **4e** was obtained as shiny yellow needles (pyridine), yield (1.16 g, 90%), mp 300–301 °C; ^{1}H NMR ($CDCl_3$) δ 2.64 (s, 3H), 6.88–7.07 (m, 1H), 7.48–7.73 (m, 3H), 8.06 (d, $J = 1.9$ Hz, 1H), 8.28 (d, $J = 8.6$ Hz, 1H), 8.91 (d, $J = 6.4$ Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 22.23, 102.91, 109.50, 110.11, 117.34, 127.45, 131.54, 133.26, 133.35, 136.54, 137.19, 142.56, 143.50, 146.13, 147.12, 149.07 ppm; IR (KBr): 2197 cm^{-1} (CN). MS (m/z) 258 (M^+). Anal. Calcd for $C_{16}H_{10}N_4$ (258.3): C, 74.41; H, 3.90; N, 21.69. Found: C, 74.23; H, 3.79; N, 21.43.

3. Results and discussion

The new (4-substituted)(3-hydroxyimino-2,3-dihydroimidazo[1,2-*a*]pyridin-2-yliden)methyl cyanides **3a**–**e** were synthesized via the nucleophilic substitution of hydrogen of 3-nitro-imiadazo[1,2-*a*]pyridine **1** with arylacetonitriles **2a**–**e** in basic MeOH solution [28] in excellent yields (Fig. 1). When *R* is an electron withdrawing group such as NO_2 group, the yield of the reaction is very low, since the corresponding conjugated base is a weak

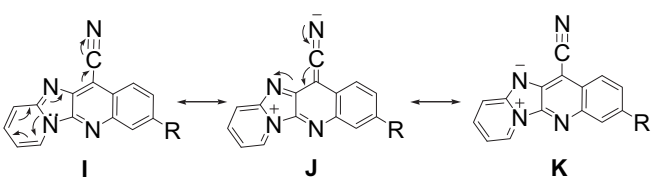

Fig. 7. The resonance structures of compounds **4a**–**e**.

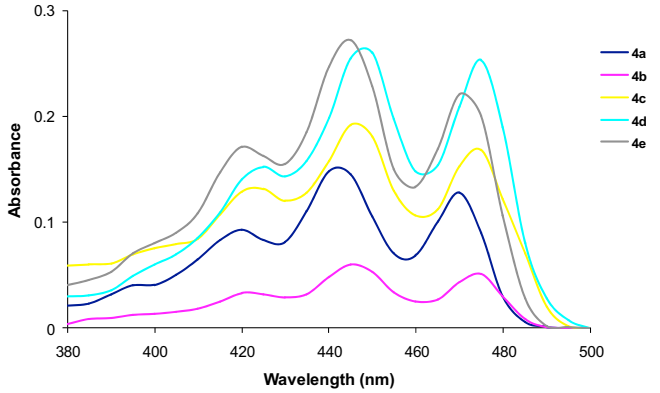

Fig. 8. Visible absorption spectrum of compounds **4a**–**e** in dilute (1×10^{-6} M) chloroform solution.

nucleophile. A proposed mechanism to explain the formation of compounds **3a**–**e** is shown in Fig. 2.

In spectral data of compounds **3a**–**e**, the presence of one exchangeable peak at ranging 13.39–13.51 ppm in the ^{1}H NMR spectrum, a weak absorption band at ranging 2195–2198 cm^{-1} and a broad absorption band at ranging 3450–3455 cm^{-1} assignable to CN and OH group in the IR spectrum together with microanalytical data strongly support the uncyclized structure of compounds **3a**–**e**.

As depicted in Fig. 3, in these compounds, there is a strong resonance from OH (electron-donor) to CN (electron-acceptor) group which can be witnessed in their absorption spectra in visible (violet) area. Formation of two aromatic rings (in form B) is the driving force for this fully extended conjugation.

Fig. 4 shows the visible absorption spectrum of compounds **3a**–**e** in dilute (3×10^{-5} M) ethanol solution. Characteristics of absorption spectra for **3a**–**e** in ethanol are presented in Table 1. Values of extinction coefficient (ε) were calculated as the slope of the plot of absorbance *vs* concentration, and have a precision of the order of 5%. Absorbance intensity and extinction coefficient (ε) in compound **3e**, with a methyl group attached to the *para* position of the benzene ring, were the biggest values.

Acylation of compounds **3a**–**e** with acetyl chloride gave the new pyrido[2′,1′:2,3]imidazo[4,5-b]quinolines **4a**–**e**, in excellent yields, *via* the intramolecular electrophilic aromatic substitution (Fig. 5). The possible mechanism for this transformation is shown in Fig. 6. Other reagent such as $SOCl_2$ and $POCl_3$ act in same way but the yield of reaction is not as good as acetyl chloride.

The structural assignments of compounds **4a**–**e** were based on the analytical and spectral data. For example, the ^{1}H NMR spectrum

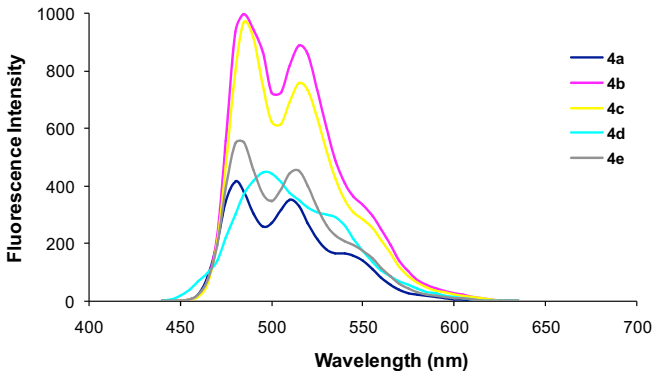

Fig. 9. Emission spectrum of compounds **4a**–**e** in dilute (1×10^{-6} M) chloroform solution.

Table 2
Photophysical data for absorption (abs) and fluorescence (flu) of **4a**–**e**.

Dye	4a	4b	4c	4d	4e
λ_{max} (nm)	443	446	447	448	445
$\varepsilon \times 10^{-4}$ (M^{-1} cm^{-1})	13.50	6.00	19.30	26.50	27.40
λ_{flu} (nm)	480	485	485	495	485
Φ_F[a]	0.57	0.90	0.87	0.61	0.70

[a] Quantum yield.

of **4d** revealed the absence of the signal at δ 13.27 ppm assignable to one exchangeable proton (OH group) and two doublet signals at δ 7.02 ppm and δ 8.07 ppm attributed to four aromatic protons of *p*-methoxyphenyl ring of compound **3d** and presence of the doublet of doublet signal at δ 7.47 ppm ($J = 9.4$ Hz and $J' = 2.4$ Hz), the doublet signal at δ 7.60 ppm with meta J (2.4 Hz) and the doublet signal at δ 8.31 ppm with ortho J (9.4 Hz) assignable to three aromatic protons of methoxyphenyl ring. Moreover, the FT-IR spectrum of **4d** in KBr did not show the absorption band at 3450 cm^{-1} corresponding to OH group. Furthermore, the mass spectrum of this product shows the molecular ion m/z 274 (M^+), confirming its presumed structure. Analytical data are also in accordance with the proposed structure for compound **4d**. These compounds were highly fluorescent. When a heteronitrogen is singly bonded to carbon atoms in a heterocycle, as in pyrrole rings (e.g. indole, carbazole), the transitions involving the non-bonding electrons have properties similar to those of π–π^* transitions. In fact, the non-bonding orbital is perpendicular to the plane of the ring, which allows it to overlap the *p* orbitals on the adjacent carbon atoms. The relatively high fluorescence quantum yield of the synthetic compounds **4a**–**e** can be explained in the same way (Fig. 7).

The fluorescence absorption and emission spectra of compounds **4a**–**e** were recorded at the concentration of 10^{-6} M in chloroform as the solvent. The fluorescence excitation (λ_{ex}) wavelength at 271 nm (λ_{ex}/nm) was used for all compounds **4a**–**e**. Figs. 8 and 9 show the visible absorption and emission spectra of compounds **4a**–**e**. The λ_{max}, λ_{flu} and fluorescence quantum yield (Φ_F) data are presented in Table 2.

4. Conclusion

In conclusion, we have described a new and efficient synthetic method for the synthesis of new tetracyclic ring systems containing a quinoline nucleus which have strong fluorescence emission. The precursors of these compounds, (3-hydroxyimino-2,3-dihydroimidazo[1,2-*a*]pyridin-2-yliden)-2-phenylacetonitrile derivatives **3a**–**e** were prepared *via* nucleophilic substitution of hydrogen on 3-nitro-imidazo[1,2-*a*]pyridine.

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