Thermolysis of Chlorovinyl Imines as an Alternate Route for the Synthesis of Pyranoquinolin-3-one and Pyranoacridin-3-one Derivatives

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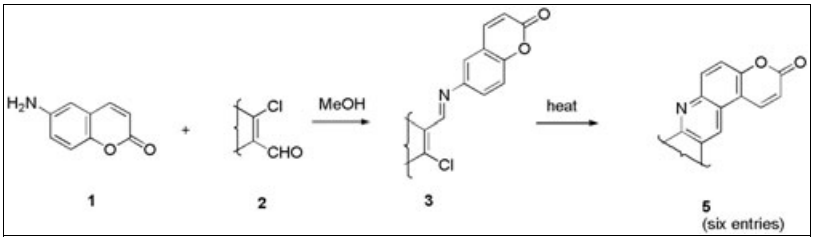

Synthesis of 3*H*-pyrano[3,2-*f*]quinolin-3-one and 3*H*-benzo[*h*]pyrano[3,2-*a*]acridin-3-one derivatives are described by the thermolysis of suitable chlorovinyl imine derivatives. The chlorovinyl imines were obtained by condensation of suitable β-chloro-α,β-unsaturated aldehydes and 6-aminocoumarin in methanol at 15°C. Compounds showed blue fluorescence in alkaline medium.

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INTRODUCTION

The coumarin derivatives fused to azaheterocycles, especially the pyridine nucleus, have been reported to possess various biological activities [1,2] like antiallergic, antidiabetic, analgesic, anti-hypertensive, anti-HIV, and antimalarial properties. This makes such class of compounds important target molecules for synthesis with gradual increase of publications in the last few decades [2]. Thermolysis of anil hydrochlorides has been established as an effective method for the synthesis of various polycyclic azaarenes in the last three to four decades [3]. The anil hydrochloride derivatives are obtained by reaction of one equivalent of β-chloro-α,β-unsaturated aldehyde derivatives and two equivalents of aryl amine. Ray *et al*. observed that reaction of 1-naphthylamine and 1-chloro-3,4-dihydro-2-naphthaldehydes failed to form any anil hydrochlorides but formed the simple Schiff bases (chlorovinyl imine derivatives) [4]. Thermolysis of these chlorovinyl imines also produced desired polycyclic azaarenes in very good yield. The disadvantage of this method is that it is limited to 1-naphthylamines. Other aryl amines produce anil hydrochlorides, rather than chlorovinyl imine derivatives, even when using the amine and chloroaldehydes in 1:1 ratio under normal condition, and one equivalent of chloroaldehyde remained unreacted. Also the Schiff bases prepared by other methods are not so stable and gradually turned into anil derivatives on storage. Advantage of thermolysis of chlorovinyl imines to prepare polycyclic aza arenes (PAAs) is that here one equivalent of amine is saved and sometime may be a better method especially when the aryl amine component is costly or difficult to synthesize. Very recently, we have reported the synthesis of some novel polynuclear pyridocoumarin derivatives [5] by thermolysis of anil hydrochlorides derived from various β-chloro-α,β-unsaturated aldehyde derivatives and two equivalents of 6-aminocoumarin. In our present work, we report a modified method to synthesize pyridocoumarin derivatives by thermolysis of chlorovinyl derivatives derived from various β-chloro-α,β-unsaturated aldehyde derivatives and one equivalent of 6-aminocoumarin. Coumarin-derived fluorescent chemosensors have been extensively applied in a variety of fields. These sensors are effective for detection of many species such as metal ions, anions, biothiols, enzymes, amines and amino acids, chemical warfare agents, proteins, hydroxyl radicals, polymerization and polymeric micelles, DNA and RNA, oxygen, and titania [6a, 6b, 6c, 6d, 6e]. Coumarins, with the structure of benzopyrone [6f], have many advantages including high fluorescence quantum yield, large Stokes shift, excellent light stability, and less toxicity. Therefore coumarins have been widely used in the fields of biology and medicine, perfumes, cosmetics, and fluorescent dyes. By far, coumarin derivatives have been used as fluorescent probes of pH, for detection of nitroxide, nitric oxide, and hydrogen peroxide [6g, 6h, 6i]. Our intention is to check the fluorescence property of these pyridocoumarin derivatives synthesized. The results are discussed later.

RESULT AND DISCUSSIONS

In our present work, we have explored the preparation of chlorovinyl imines and the general method for the

synthesis of pyridocoumarins through thermolysis of chlorovinyl imines. Our first task was to find out an optimal condition to prepare the chlorovinyl imines in the best possible yield because the reaction of such chloroaldehydes with aryl amines (other than 1-naphthylamine derivatives) spontaneously formed anil hydrochlorides instead of chlorovinyl imines [4]. Condensation between 5-methoxy α-tetralone (one equivalent) and 6-aminocoumarin (one equivalent) (Scheme 1) was studied under variation of solvents (chloroform, benzene, MeOH, etc), additives (HCl, Al_2O_3, $NaHCO_3$, etc.), or temperature and time. Results of the detailed studies are summarized in Table 1. It was found that condensation of **1** and **2d** in methanol at 15°C without any acid/base catalyst proved to be the best condition and leads to the desired product in 95% yield within 30 min (Table 1, entry 5). Thus treatment of 6-aminocoumarin (one equivalent) with different β-chloro-α,β-unsaturated aldehyde derivatives (one equivalent) proceeded to give the corresponding novel chlorovinyl imine derivatives. IR spectra of compound **3d** show strong absorption band at 1625.4 and 1732.0 cm^{-1}. The ^1H NMR (300 MHz, $CDCl_3$) data are in conformity with the assigned structure for the chlorovinyl imine **3d** and given later (Fig. 1), and high-resolution mass spectrometry (HRMS) data are also in agreement with the structure of **3d**. In HRMS, two distinct *m*/*z* peaks were observed at 365.9783 for (M^+ + H) and 367.9769 for (M^+ + H + 2) and indicate the presence of chlorine. Reaction of other β-chloro-α,β-unsaturated aldehyde derivatives **2**(**a**–**f**) and 6-aminocoumarin under identical condition produced respectively the chlorovinyl imine derivatives **3**(**a**–**f**) in excellent yields (Table 2). The chlorovinyl imine derivatives were characterized by usual spectroscopic data. When these chlorovinyl imine derivatives were subjected to thermolysis, the desired

Scheme 1. Reaction of chloroaldehyde **2d** with 6-aminocoumarin **1.**

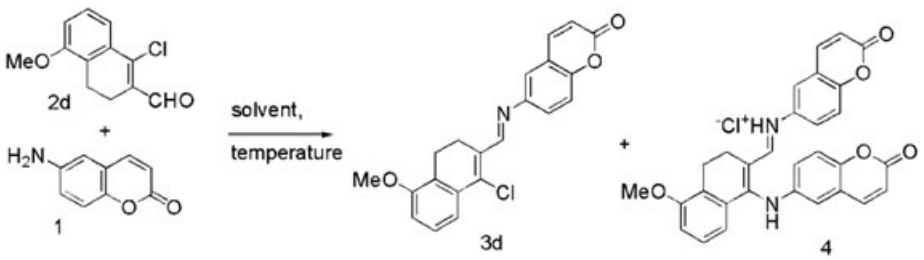


Table 1

Optimization studies in the selective formation of chlorovinyl imine **3d.**

Entry no.	Reactants	Solvent	Additive	Temp (°C)	Time (h)	Product (**3**) (%)	Product (**4**) (%)
1	1[a]:1[b]	Benzene	$NaHCO_3$	Reflux	10	50	—
2	1[a]:1[b]	Benzene	$NaHCO_3$ + Al_2O_3	Reflux	10	52	—
3	1[a]:1[b]	EtOH	$NaHCO_3$	Reflux	12	35	15
4	1[a]:1[b]	EtOH	HCl	15°C	3	—	45
5	**1[a]:1[b]**	**MeOH**	—	15°C	**0.5**	**95**	—
6	1[a]:2[b]	MeOH	—	15°C	0.5	85	5
7	1[a]:1[b]	$CHCl_3$	—	15°C	1	70	—

rt = 15–20°C. Best optimized condition for the reported reaction is shown in bold entry.
[a]**2d**.
[b]**1** in equivalent.

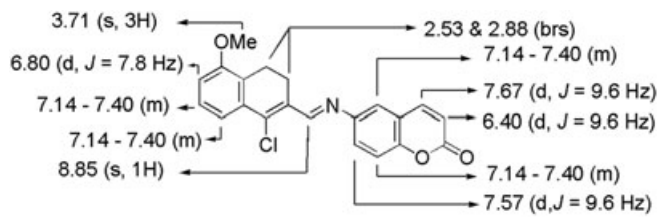
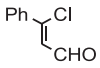
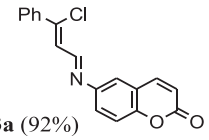
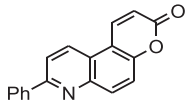
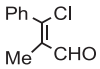
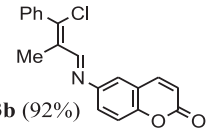
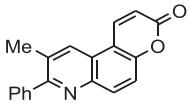
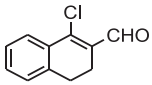
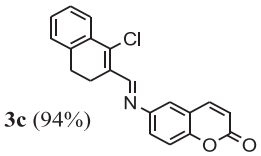
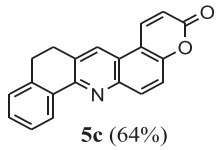
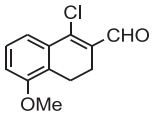
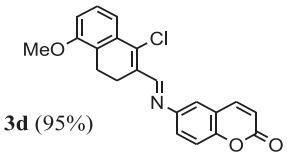
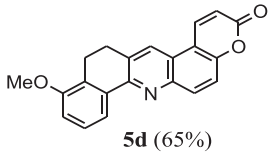
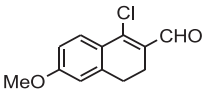
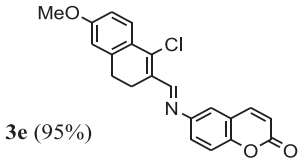
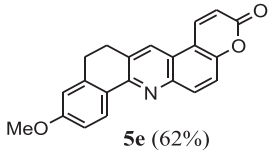
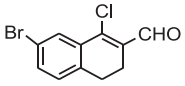
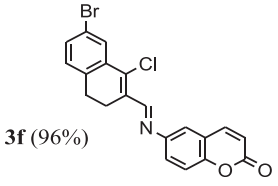
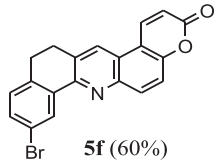


Figure 1. Chemical shifts (δ, ppm) in ^1H NMR spectra of **3d.**

Table 2

Formation of chlorovinyl imine and pyridocoumarin derivatives 3*H*-pyranoquinolin-3-one and 12,13-dihydro-3*H*-benzo[*h*]pyrano[3,2-*a*]acridin-3-one derivatives.

Entry no.	Reactant	Chlorovinyl imine yield	Time (min)	Product(s) yield
1	 **2a**	 **3a** (92%)	30	 **5a** (65%)
2	 **2b**	 **3b** (92%)	60	 **5b** (60%)
3	 **2c**	 **3c** (94%)	50	 **5c** (64%)
4	 **2d**	 **3d** (95%)	30	 **5d** (65%)
5	 **2e**	 **3e** (95%)	30	 **5e** (62%)
6	 **2f**	 **3f** (96%)	50	 **5f** (60%)

PAA **5**(**a**–**f**) in moderate to good yields was produced. Thus compound of **3d** when heated at about 230–260°C for 15–20 min, without any solvent, furnished the novel pyridocoumarin derivative 11-methoxy-12,13-dihydro-3*H*-benzo[*h*]pyrano[3,2-*a*]acridin-3-one (**5d**) in 65% yield after usual work-up (Scheme 2). IR spectra of compound **5d** show strong absorption band at 1628.8 and 1729.0 cm^{-1}. The ^1H NMR (300 MHz, $CDCl_3$) data are in conformity with the assigned structure for the pyridocoumarin **5d** and given later (Fig. 2), and HRMS data are also in agreement with the structure of **5d**. The method is a general one, and other chlorovinyl imines **3**(**a–c**, **e**, **f**) too on thermolysis produced the pyridocoumarin derivatives **5**(**a**–**c**, **e**, **f**), respectively, in 60–65% yields. Scheme 3 represents a plausible mechanism [4] showing the formation of compound **5d** from **3d**.

Fluorescence properties of reference compound 5d. The fluorescence property of **5d** as reference compound (50 μ*M*) was investigated in aqueous methanol at 25°C (λ_{exc} = 350 nm) (Fig. 3). Compound **5d** as such was fluorescence silent in absence of hydroxide ion, although fluorescence intensity of **5d** significantly increased when various concentrations of OH^- (5–100 μ*M*) were added

Scheme 2. Cyclization of chlorovinyl imine **3d** to **5d.**

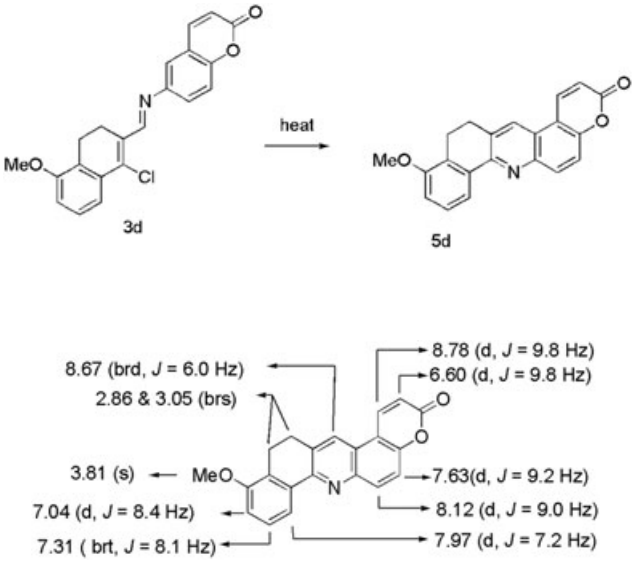


Figure 2. Chemical shifts (δ, ppm) in ^{1}H NMR spectra of **5d.**

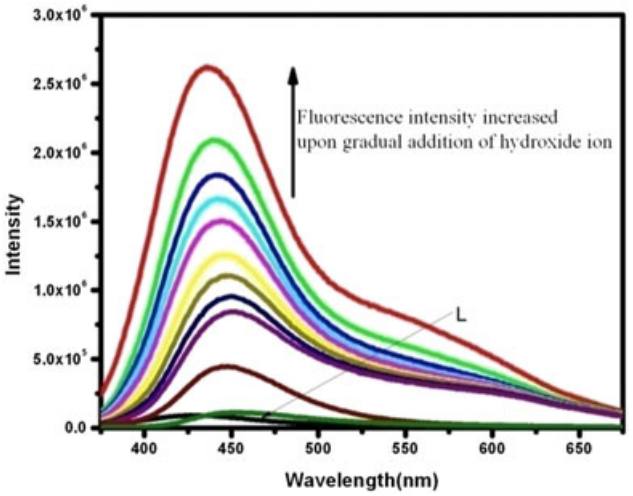
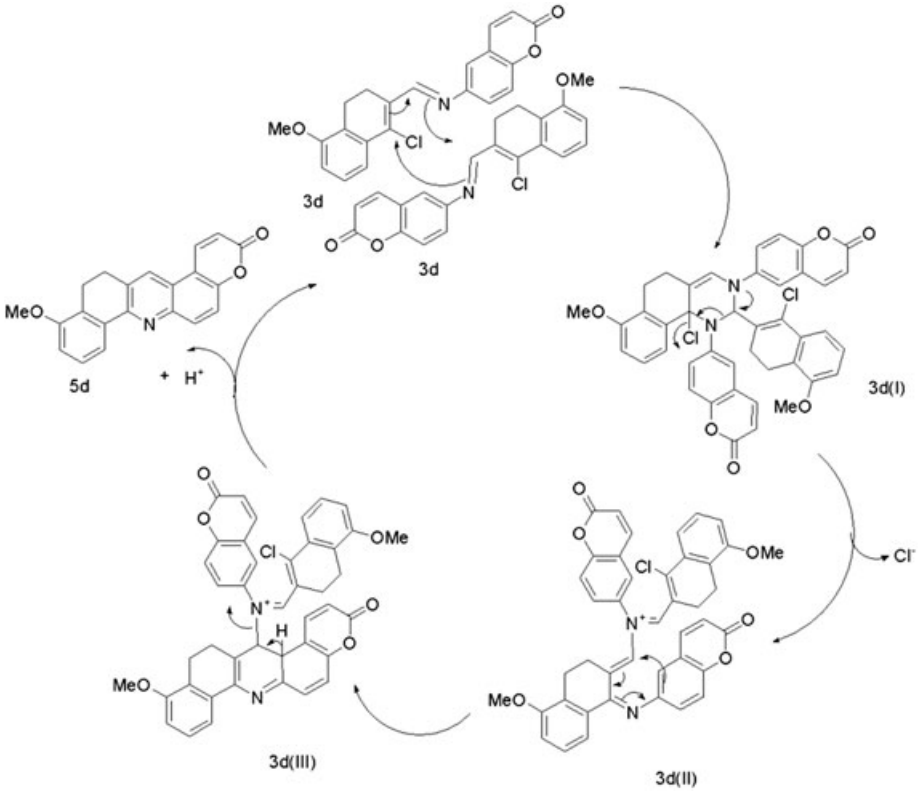


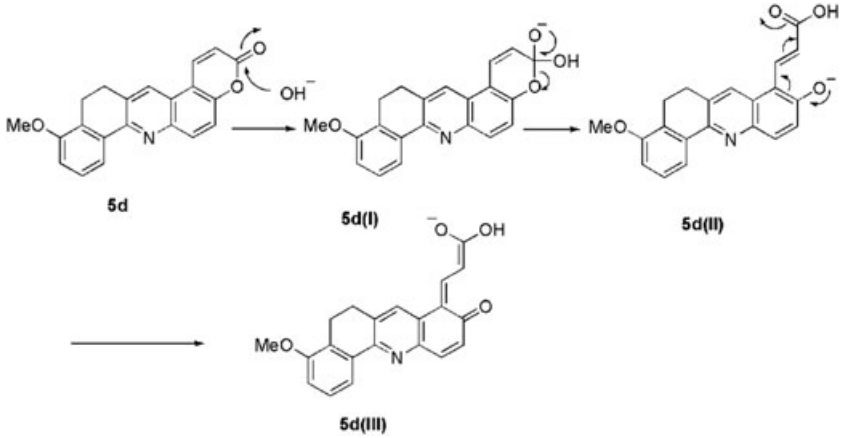
Figure 3. Emission spectra of **5d** (40 m*M*) in the presence of various concentrations of aqueous OH^{-} (λ_{exc} = 350 nm). [Color figure can be viewed at wileyonlinelibrary.com]

(Fig. 1). Upon gradual addition of aqueous OH^{-} solution, increases in pH from 7 to 12 caused a gradual increase in intensity with concomitant little red shift of the band 431 to 438 nm. This enhancement was attributed to the base hydrolysis of lactone ring of coumarin and consequently increased conjugation involved therein (Scheme 4). **5d** has shown selectivity only for hydroxide anion in aqueous methanol over the other anions such as F^{-}, Cl^{-},

Scheme 3. Mechanism of the cyclization from **3d** to **5d.**



Scheme 4. Mechanism of fluorescence enhancement upon addition of OH^- ion.



Br^-, I^-, OAc^-, PO_4^{3-}, NO_3^-, NO_2^-, HSO_4^-, and $H_2PO_4^-$. The change in color of chemosensor **5d** (100 μ*M*) upon addition of hydroxyl ions (10 μ*M*) was clearly visible under visible light through the naked eye, whereas in the presence of the other anions, the ligand solution was colorless. The bright blue fluorescence under UV light was observed only for the mixture of **5d** and OH^- in solution (Fig. 4). This is an interesting feature by which we can detect OH^- without any other instrumental techniques. In conclusion, it is evident that thermolysis of chlorovinyl imines derivatives from various β-chloro-α,β-unsaturated aldehyde derivatives and 6-aminocoumarin may be an alternate route for the synthesis of the pyranoquinolin-3-one and pyranoacridin-3-one derivatives. The process saved the consumption of one equivalent 6-aminocoumarin. The compound shows blue fluorescence in presence of hydroxide ions under UV.

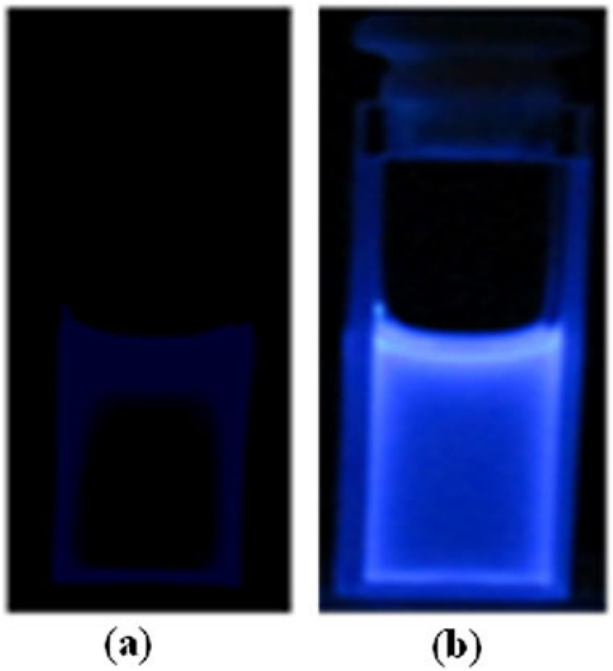


Figure 4. Fluorescence enhancement of **5d** upon addition of OH^- ion: (a) fluorescence silent in absence of hydroxide ion and (b) bright blue fluorescence of **5d** in presence of hydroxide ion under UV light. [Color figure can be viewed at wileyonlinelibrary.com]

EXPERIMENTAL

Preparation of chlorovinyl imines derivatives 3(a–f): general method. A mixture of 6-aminocoumarin (**1**) (290 mg, 1.80 mmol) and the chloroaldehyde **2** (1.80 mmol) in 35–40 mL of MeOH was stirred vigorously at 15°C for 30 min to 1 h (reaction was monitored by thin-layer chromatography) and then cooled to 5–10°C. The yellow to orange solid that separated was filtered, washed with little cold MeOH, and dried under vacuum to obtain the chlorovinyl imines derivatives **3** in 92–96% yield. The chlorovinyl imines were directly used for next step without further purification.

6-((E)-((E)-3-Chloro-3-phenylallylidene)amino)-2H-chromen-2-one (3a). Bright orange solid, yield, 92%; mp 132–133°C (EtOH); IR (KBr) ν_{max}: 1628.8, 1729.0 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) 6.4 (d, 1H, J = 9.6 Hz), 6.44 (d, 1H, J = 9.6 Hz), 6.98 (brs, 1H), 7.07 (m, 1H), 7.22–7.39 (m, 4H), 7.45 (d, 1H, J = 12 Hz), 7.53 (brs, 1H), 7.63 (brs, 1H), 7.9 (brs, 1H) ppm; HRMS [electrospray ionization (ESI), 70 eV]: m/z = 310.1080 (M^+ + H), 312.1128 (M^+ + H + 2) [Calcd. mass for $C_{18}H_{13}ClNO_2$: 310.06 (M^+ + H), 312.06 (M^+ + H + 2)].

6-((E)-((E)-3-Chloro-2-methyl-3-phenylallylidene)amino)-2H-chromen-2-one (3b). Orange solid, yield, 92%; mp 127–128°C (EtOH); IR (KBr) ν_{max}: 1624.0, 1733.8 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 2.42 (s, 3H), 6.47 (d, 1H, J = 9.9 Hz), 7.15 (brs, 1H), 7.24 (m, 1H), 7.32 (d, 1H, J = 8.1 Hz), 7.48 (m, 5H), 7.71 (d, 1H, J = 8.7 Hz), 8.16 (brs, 1H) ppm; HRMS (ESI, 70 eV): m/z = 323.9812

(M^+ + H), 325.9782 (M^+ + H + 2) [Calcd. mass for $C_{19}H_{15}ClNO_2$: 324.08 (M^+ + H), 326.08 (M^+ + H + 2)].

(E)-6-((1-Chloro-3,4-dihydronaphthalen-2-yl)methyleneamino)-2H-chromen-2-one (3c). Light yellow solid, yield, 94%; mp 145–146°C (EtOH); IR (KBr) ν_{max}: 1631.5, 1728.4 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 2.94 (m, 2H), 2.96 (m, 2H), 6.47 (d, 1H, *J* = 9.6 Hz), 7.22–7.26 (m, 1H), 7.26–7.35 (m, 3H), 7.38 (brs, 1H), 7.43 (dd, 1H, *J* = 2.4 and 8.7 Hz), 7.74 (d, 1H, *J* = 9.6 Hz), 7.81 (dd, 1H, *J* = 3.6 and 5.6 Hz), 8.92 (s, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): 23.71, 27.37, 116.93, 117.17, 117.64, 119.25, 125.17, 126.92, 127.48, 130.04, 131.39, 132.34, 138.29, 139.09, 143.10, 143.27, 148.54, 152.46, 159.09, 160.61 ppm; HRMS (ESI, 70 eV): *m*/*z* = 336.1084 (M^+ + H), 338.1131 (M^+ + H + 2) [Calcd. mass for $C_{20}H_{15}ClNO_2$: 336.08 (M^+ + H), 338.08 (M^+ + H + 2)].

(E)-6-((1-Chloro-5-methoxy-3,4-dihydronaphthalen-2-yl) methyleneamino)-2H-chromen-2-one (3d). Light yellow solid, yield, 95%; mp 153–154°C (EtOH); IR (KBr) ν_{max}: 1625.4, 1732.0 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 2.53 (m, 2H), 2.88 (m, 2H), 3.71 (s, 3H), 6.40 (d, 1H, *J* = 9.6 Hz), 6.80 (d, 1H, *J* = 7.8 Hz), 7.14–7.40 (m, 4H), 7.57 (d, 1H, *J* = 9.6 Hz), 7.67 (d, 1H, *J* = 9.6 Hz), 8.85 (s, 1H) ppm; HRMS (ESI, 70 eV): *m*/*z* = 365.9783 (M^+ + H), 367.9769 (M^+ + H + 2) [Calcd. mass for $C_{21}H_{17}ClNO_3$: 366.09 (M^+ + H), 368.09 (M^+ + H + 2)].

(E)-6-((1-Chloro-6-methoxy-3,4-dihydronaphthalen-2-yl) methyleneamino)-2H-chromen-2-one (3e). Yellow solid, yield, 95%; mp 182–184°C (EtOH); IR (KBr) ν_{max}: 1620.0, 1731.8 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 2.91 (m, 4H), 3.86 (s, 3H), 6.46 (d, 1H, *J* = 9.3 Hz), 6.72–6.85 (m, 3H), 7.28 (m, 2H), 7.38 (d, 1H, *J* = 10.5 Hz), 7.73 (d, 1H, *J* = 10.2 Hz), 8.88 (s, 1H) ppm; HRMS (ESI, 70 eV): *m*/*z* = 365.9783 (M^+ + H), 367.9769 (M^+ + H + 2) [Calcd. mass for $C_{21}H_{17}ClNO_3$: 366.09 (M^+ + H), 368.09 (M^+ + H + 2)].

(E)-6-((7-Bromo-1-chloro-3,4-dihydronaphthalen-2-yl) methyleneamino)-2H-chromen-2-one (3f). Yellow solid, yield, 96%; mp 143–144°C (EtOH); IR (KBr) ν_{max}: 1629.8, 1728.4 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 2.50 (m, 2H), 2.79 (m, 2H), 6.50 (d, 1H, *J* = 9.6 Hz), 6.80 (d, 2H, *J* = 7.8 Hz), 6.89 (d, 1H, *J* = 9.3 Hz), 7.1–7.3 (m, 2H), 7.55 (s, 1H), 7.67 (d, 1H, *J* = 9.6 Hz), 8.89 (s, 1H) ppm; HRMS (ESI, 70 eV): *m*/*z* = 414.0125 (M^+ + H), 416.0139 (M^+ + H + 2) [Calcd. mass for $C_{20}H_{14}BrClNO_2$: 413.99 (M^+ + H), 415.99 (M^+ + H + 2)].

Thermal cyclization of chlorovinyl imines: general method for the preparation of pyridocoumarins 5(a–f). Dry chlorovinyl imine derivative **3** (1.0 mmol) was taken in a long-necked, hard glass test tube and heated at about 230–260°C in a salt bath. It was kept at that condition for 15–20 min and then cooled to room temperature. The fused mass was extracted thoroughly with chloroform and washed with water. The organic layer was collected and dried (anhyd. Na_2SO_4), and solvent removed. The crude residue thus obtained was further purified by column chromatography (silica gel/benzene–ethyl acetate mixture, 5:1) to furnish the pyridocoumarin derivative **5** as the only isolable product in 60–65% yield. An analytical sample was prepared by further recrystalization from suitable solvent.

11-Methoxy-12,13-dihydro-3H-benzo[h]pyrano[3,2-a]acridin-3-one (5d). Light brown solid, yield, 65%; mp 222–224°C ($CHCl_3$/pet. ether 60–80°C); IR (KBr) ν_{max}: 1726.8 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 2.86 (m, 2H), 3.05 (brs, 2H), 3.81 (s, 3H), 6.60 (d, 1H, *J* = 9.8 Hz), 7.04 (d, 1H, *J* = 8.4 Hz), 7.31 (brt, 1H, *J* = 8.1 Hz), 7.63 (d, 1H, *J* = 9.2 Hz), 7.97 (d, 1H, *J* = 7.2 Hz), 8.12 (d, 1H, *J* = 9.0 Hz), 8.67 (brd, 1H, *J* = 6.0 Hz), 8.78 (d, 1H, *J* = 9.8 Hz) ppm; HRMS (ESI, 70 eV): *m*/*z* = 330.0885 (M^+ + H) [Calcd. mass for $C_{21}H_{16}NO_3$: 330.11 (M^+ + H)].

(mp and spectral data of the pyridocoumarins **5**(**a**–**c**) and **5**(**e**–**f**) are in agreement with lit. report [5]).

Compound 5a. Colorless solid, yield 65%; mp 212–213°C ($CHCl_3$/pet. ether 60–80°C) (lit mp 212–213°C, ref of [5]).

Compound 5b. Colorless solid, yield 60%; mp 240–242°C ($CHCl_3$/pet. ether 60–80°C) (lit mp 240–242°C, ref of [5]).

Compound 5c. Colorless solid, yield 64%; mp 248–250°C ($CHCl_3$/pet. ether 60–80°C) (lit mp 248–250°C, ref of [5]).

Compound 5e. Light yellowish solid, yield 62%; mp 240–242°C ($CHCl_3$/pet. ether 60–80°C) (lit mp 240–242°C, ref of [5]).

Compound 5f. Light yellow solid, yield 60%; mp 242–244°C ($CHCl_3$/pet. ether 60–80°C) (lit mp 242–244°C, ref of [5]).

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