Thermolysis of *N*-Aryl Enaminoimine Hydrochloride Derivatives: A Short and General Method for the Synthesis of Pyranoquinolin-3-one and Pyranoacridin-3-one Derivatives

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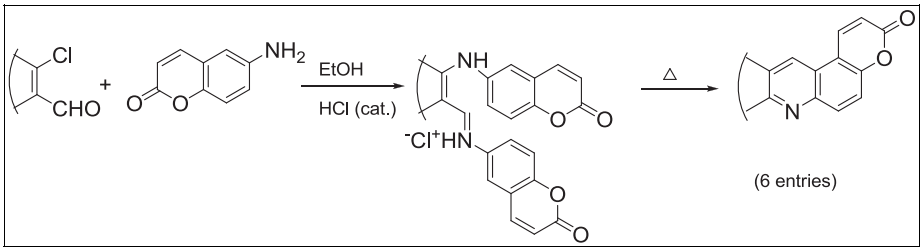

A short synthesis of 3H-pyrano[3,2-f]quinolin-3-one, 3H-acenaphtho[1,2-b]pyrano-[3,2-f]quinolin-3-one, and 3H-benzo[h]pyrano[3,2-a]acridin-3-one derivatives are described via thermolysis of suitable enaminoimine hydrochloride derivatives.

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INTRODUCTION

Coumarins fused to heteroaromatic rings constitute an important class of non-natural and natural product. Many natural and synthetic coumarin derivatives [1–12] fused with naphthalene, aza/oxa/thia-heterocycles have been reported to possess significant biological activities including anti HIV activity, cytotoxity and anticancer activity, antimicrobial activity, as well as activity against malignant dermatoses. Chromenoquinoline derivatives [13] have been reported to act as human progesterone receptor and have been used therapeutically. Although coming across the literature, we found a gradual increase on the studies towards the synthesis of pyridocoumarin derivatives since 1999 and in the last decade, a large number of synthesis [14–27] of such compounds have been reported by various groups especially by Majumdar *et al*. [15,20,25–27]. As a part of our ongoing studies towards the synthesis of novel coumarin derivatives anchored with heterocyclic rings, we undertook the challenge towards the synthesis of some novel polynuclear pyridocoumarin derivatives such as 3H-pyrano[3,2-f]quinolin-3-one, 3H-acenaphtho[1,2-b]pyrano [3,2-f]quinolin-3-one, and 3H-benzo[h]pyrano[3,2-a]acridin-3-one derivatives. For quite some time, we, in collaboration with the group of Ray *et al*, are working on the synthesis of polycyclic azaarenes via thermoysis of *N*-aryl enaminoimine hydrochloride derivatives (anil hydrochlorides) [28–31]. Such anil hydrochloride derivatives are obtained by reaction of suitable β-chloro-α,β-unsaturated aldehyde derivatives and aryl amines. The method has several advantages such as easy availability of starting materials, simple procedure, short time reaction and generally high yield, and above all the potential to introduce to various substitutions in both aryl amine and chloroaldehyde as part of the substrates. We have used this pathway to achieve the synthesis of our target compounds.

RESULT AND DISCUSSIONS

Retro synthesis (Figure 1) showed that the required enaminoimine hydrochlorides (**3**) may be a precursor for the synthesis of pryidocoumarins (**4**) and such enaminoimine hydrochlorides can easily be obtained from β-chloroacrolein derivatives (**2**) and 6-aminocoumarin (**1**). A number of pyranoquinolin-3-one and pyranoacridin-3-one derivatives have been synthesized using these pathway. Herein, we report our results.

The required choroaldehydes **2(a–f)** were prepared via Vilsmeier–Haack reaction from the corresponding ketones and $POCl_3$/DMF by standard-reported procedure. When these chloroaldehydes were treated with two equivalents of 6-aminocoumarin (**1**) in ethanol containing catalytic conc, HCl produced the anil hydrochlorides (**3**) in excellent yields. Thus, 1-chloro-6-methoxy-3,4-dihydronaph- thalen-2-aldehyde (**2d**) (1.0 equivalent) on treatment with 6-aminocoumarin (**1**) (~2.0 equivalents) in EtOH in presence of HCl (cat.) at room temperature furnished the intermediate *N*-aryl enaminoimine hydrochloride derivative **3d**, as a dark red solid, in 95% yield. Brief heating of the anil hydrochloride derivative (**3d**) at about 250–260 °C for 3–5 min without any solvent furnished the pyridocoumarin derivative **4d** (10-methoxy-12,13-dihydro-3H-benzo[h]pyrano[3,2-a]acridin-3-one), in 52% yield, as the only isolable product after usual work up. The spectral and analytical data of compound **4d** is in well agreement with the assigned structure. We were

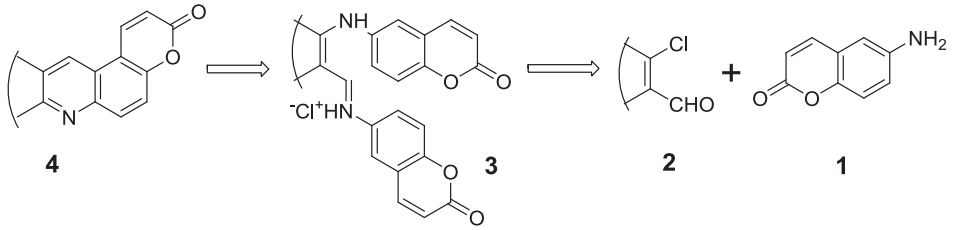


Figure 1. Retrosynthesis of pyridocoumarin derivatives.

unable to isolate the other possible isomeric product (**5d**) formed (if any) (Scheme 1).

The method is a general one. A series of novel pyridocoumarin derivatives **4(a–f)** have been synthesized (in 50–58% yield) via thermal cyclization at 220–320 °C of different anil hydrochloride derivatives **3(a–f)**. These anil hydrochlorides were in turn prepared by reaction of various chloroaldehydeds **2(a–f)** and 6-aminocoumarin (**1**) (table-1). In most of the cases, the major/isolated product is formed via cyclization of anil derivative at C_5-position of the coumarin moiety. However, in few occasions, we were able to isolate the isomeric products that arise via cyclization at C_7-position of the coumarin ring (entries no. 1 and 2). The results have been summarized in Table 1.

The dihydropyrido coumarin **4(c–e)** when aromatized with DDQ in refluxing chlorobenzene at ~140 °C for 12–14 h furnished the fully aromatic 3H-benzo[h]pyrano [3,2-a]acridine-3-one derivatives **5(c–e)** in excellent yield (Scheme 2)

In conclusion, we have achieved the synthesis of several 3H-pyrano[3,2-f]quinolin-3-one, 3H-acenaphtho[1, 2-b]pyrano[3,2-f]quinolin-3-one, and 3H-benzo[h]pyrano[3, 2-a]acridin-3-one derivatives via an efficient, short, and simple method.

EXPERIMENTAL

All melting points are uncorrected and were checked with one-side open glass capillary using a sulphuric acid bath. Solvents were dried following standard literature procedure. ^{1}H nmr spectra were recorded on Brucker 500 MHz (at Chemgen Pharma, Kolkata) and Brucker 200 MHz (at I.I.T Kharagpur). ^{13}C nmr spectra were recorded, respectively, in 50 or 125 MHz nmr spectrometer (Brucker), respectively. ESI mass spectra were recorded on a micro mass Q-TOF mass spectrometer (serial no. YA 263) at IACS, Kolkata; IR spectral data was obtained from JASCO FT/IR680 PLUS spectrometer. Reactions were monitored with TLC on pre-coated plates of silica gel GF_{245} (Merck) and visualizing under UV light and/or by charring solution.

Preparation of anil hydrochlorides 3(a–f): General method. To a mixture of 6-aminocoumarin (**1**) (580 mg, 3.60 mmol) and the choroaldehyde **2(a–f)** (1.8 mmol) in 35–40 mL of EtOH, catalytic amount of conc. HCl was added with the help of capillary tube. The mixture was stirred vigorously at room temperature for 2–4 h and then cooled to 5–10 °C. The red to dark red solid separated was filtered, washed with little cold ethanol, and dried under vacuum to obtain the anil hydrochloride derivatives **3(a–f)** in 81–96% yield. The anil hydrochlorides were directly used for next step without further purification. ^{1}H nmr of the sample could not be recorded because of poor solubility of these salts.

Compound **3a**: Red solid, yield 81%, mp 218–220 °C (d); IR (KBr) ν_{max}: 1726.94 (very strong), 1644.02 (m), 1629.55 (m), 3442.31 (broad) cm^{-1}. Compound **3b**: Orange red solid, yield

Scheme 1. (i) EtOH, HCl (cat.), 30–35 °C, 3 h. (ii) 250–260 °C, 3–4 mins.

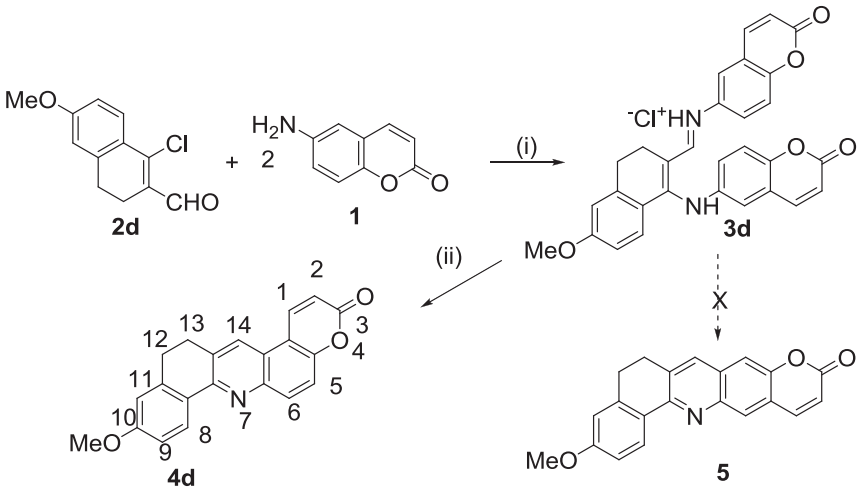
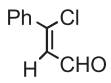
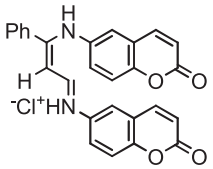
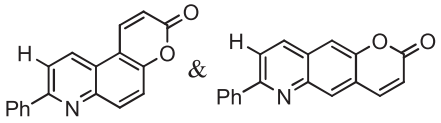
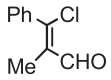
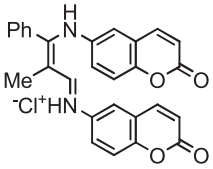
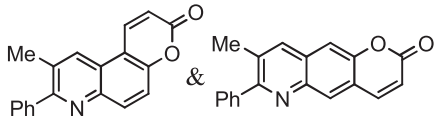
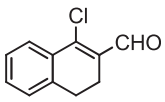
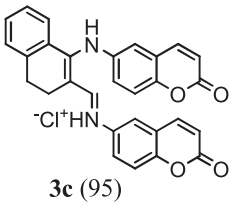
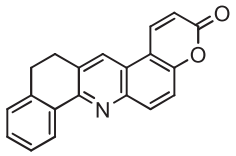
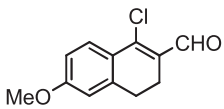
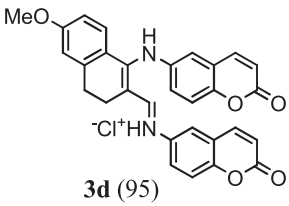
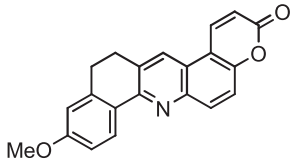
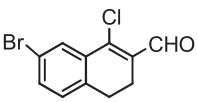
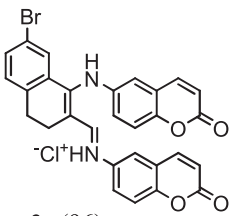
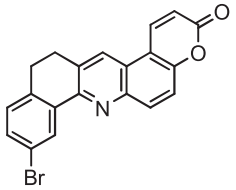
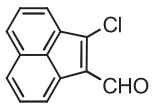
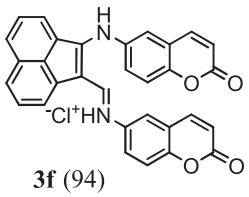
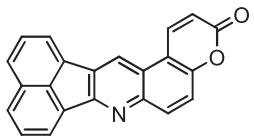
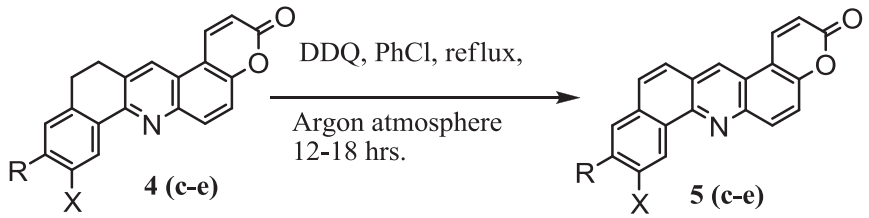


Table 1

Formation of anilhydrochloride, 3H-pyranoquinolin-3-one, 3H-acenaphtho[1,2-b]-pyrano[3,2-f]quinolin-3-one, and 12,13-dihydro-3H-benzo[h]pyrano [3,2-a]acridin-3-one derivatives.

Entry no.	Chloroaldehyde	Anil hydrochloride (% of isolated yield)	Heating temp. (°C)	Product (s) (% of isolated yield)
1	 **2a**	 **3a** (81)	220–240	 **4a** (45) **4a'** (10)
2	 **2b**	 **3b** (82)	220–240	 **4b** (46) **4b'** (10)
3	 **2c**	 **3c** (95)	250–260	 **4c** (55)
4	 **2d**	 **3d** (95)	250–260	 **4d** (52)
5	 **2e**	 **3e** (96)	250–260	 **4e** (50)
6	 **2f**	 **3f** (94)	300–320	 **4f** (50)

Scheme 2. Aromatization of compounds **4(c–e)**.



c. R = X = H; **d**. R = OMe, X = H;
e. R = H, X = Br

82%, mp 180–182 °C (d); IR (KBr) ν_{max}: 1710.55 (strong), 1729.83 (m), 1745.26 (s), 3290.93 (m), 3442.32 (br) cm^{-1}. Compound **3c**: Red solid, yield 95%, mp 235–336 °C (d); IR (KBr) ν_{max}: 1719.23 (strong), 1735.62 (s), 1750.83 (m), 3090.37 (br) cm^{-1}. Compound **3d**: Red solid, yield 95%, mp 168–170 °C (d); IR (KBr) ν_{max}: 1724.05 (strong), 3421.11 (br) cm^{-1}. Compound **3e**: Red solid, yield 96%, mp 180–182 °C (d); IR (KBr) ν_{max}: 1728.05 (strong), 3402.01 (broad) cm^{-1}. Compound **3f**: Deep red solid, yield 94%, mp 275–277 °C (d); IR (KBr) ν_{max}: 1721.16 (strong), 3403.05 (broad) cm^{-1}.

Thermal cyclization of anil hydrochlorides; preparation of pyridocoumarins 4(a–f): General method. An amount of 1.0 mmol of anil hydrochloride derivative **3 (a–f)** was taken in a long-necked hard glass test tube and heated at about 220–320 °C for 3–5 min in a salt bath. The fused mass was extracted thoroughly with $CHCl_3$ and washed with water. The organic layer was collected, 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 **4(a–f)** in 50–58% yield. An analytical sample was prepared by recrystalization from suitable solvents.

Compound **4a**: Colorless solid, yield 45%, mp 212–213 °C ($CHCl_3$ / pet. ether 60–80 °C). ^{1}H nmr (200 MHz, $CDCl_3$) δ: 6.65 (d, 1H, *J* = 10.0 Hz), 7.49–7.58 (m, 3H), 7.72 (d, 1H, *J* = 9.2 Hz), 8.08 (d, 1H, *J* = 8.8 Hz), 8.19 (d, 2H, *J* = 7.2 Hz), 8.36 (d, 1H, *J* = 9.2 Hz), 8.44 (d, 1H, *J* = 9.6), 8.61 (d, 1H, *J* = 8.8 Hz) ppm; IR (KBr) ν_{max}: 1721.16 (strong) cm^{-1}; hrms (ESI, 70 eV); *m/z*: 274.0864 (M^++H) [calculated mass for $C_{18}H_{12}NO_2$: 274.0868 (M^++H)]. Compound **4a**: Colorless solid, yield 10%, mp 210–212 °C ($CHCl_3$/pet. ether 60–80 °C) ^{1}H nmr (200 MHz, $CDCl_3$) δ: 6.56 (d, 1H, *J* = 9.6 Hz), 7.51–7.59 (m, 4H), 7.74 (s, 1H), 7.95 (d, 1H, *J* = 9.6 Hz), 8.00 (d, 1H, *J* = 8.8 Hz), 8.19 (d, 2H, *J* = 8.0 Hz), 8.33 (d, 1H, *J* = 8.8 Hz) ppm; IR (KBr) ν_{max}: 1722.12 (strong) cm^{-1}; hrms (ESI, 70 eV); *m/z*: 274.0860 (M^++H) [calculated mass for $C_{18}H_{12}NO_2$: 274.0866 (M^++H)]. Compound **4b**: Colorless solid, yield 46%, mp 240–242 °C ($CHCl_3$ / pet. ether 60–80 °C) ^{1}H nmr (200 MHz, $CDCl_3$) δ: 2.57(s, 3H), 6.62 (d, 1H, *J* = 9.7 Hz), 7.46–7.53 (m, 3H), 7.59 (m, 2H), 7.64 (d, 1H, *J* = 9.2 Hz), 8.27 (d, 1H, *J* = 9.2 Hz), 8.39 (s, 1H), 8.45 (d, 1H, *J* = 9.7 Hz) ppm. ^{13}C nmr (75 MHz, $CDCl_3$) 21.01, 112.06, 116.12, 119.79, 123.32, 128.41, 128.57, 128.82, 130.76, 131.32, 134.26, 138.30, 139.89, 143.60, 153.59, 160.19, 160.49 ppm; IR (KBr) ν_{max}: 1727.91 (strong) cm^{-1}; hrms (ESI, 70 eV); *m/z*: 288.1013 (M^++H) [calculated mass for $C_{19}H_{14}NO_2$: 288.1025 (M^++H)]. Compound **4b**: Colorless solid, yield 10%, mp 240–242 °C ($CHCl_3$/pet. ether 60–80 °C). ^{1}H nmr (500 MHz, $CDCl_3$) δ: 2.508(s, 3H), 6.38 (d, 1H, *J* = 9.5 Hz), 6.50 (br s, 1H), 7.14 (d, 1H, *J* = 2.5 Hz), 7.49 (d, 1H, *J* = 6.5 Hz), 7.50–7.53 (m, 2H), 7.60–7.62 (m, 2H), 7.65 (d, 1H, *J* = 9.0 Hz), 8.40 (s, 1H) ppm; IR (KBr) ν_{max}: 1727.01 (strong) cm^{-1}. Compound **4c**: Colorless solid, yield 58%, mp 248–250 °C ($CHCl_3$/pet. ether 60–80 °C). ^{1}H nmr (400 MHz, $CDCl_3$) δ: 3.05 (br t, 2H, *J* = 7.0 Hz), 3.21 (br t, 2H, *J* = 7.0 Hz), 6.60 (d, 1H, *J* = 9.7 Hz), 7.29 (br d, 1H, *J* = 7.1 Hz), 7.38–7.46 (m, 2H), 7.63 (d, 1H, *J* = 9.3 Hz), 8.27 (d, 1H, *J* = 9.2 Hz), 8.29 (s, 1H), 8.42 (d, 1H, *J* = 9.7 Hz), 8.55 (br d, 1H, *J* = 7.4 Hz) ppm. ^{13}C nmr (125 MHz, $CDCl_3$) δ: 28.11, 29.19, 112.40, 116.05, 119.64, 123.56, 125.96, 127.52, 127.75, 128.10, 130.18, 132.52, 133.94, 134.44, 138.44, 139.17, 144.64, 153.31, 153.42, 160.65 ppm. IR (KBr) ν_{max}: 1732.73 (strong) cm^{-1}; hrms (ESI, 70 eV); *m/z*: 300.0688 (M^++H) [calculated mass for $C_{20}H_{14}NO_2$: 300.1025 (M^++H)].

Compound **4d**: Light yellowish solid, yield 52%, mp 240–242 °C ($CHCl_3$/pet. ether 60–80 °C). ^{1}H nmr (400 MHz, $CDCl_3$) δ: 3.02 (br t, 2H, *J* = 7.0 Hz), 3.20 (br t, 2H, *J* = 7.0 Hz), 3.98 (s, 3H), 6.60 (d, 1H, *J* = 9.6 Hz), 6.81 (d, 1H, *J* = 2.0 Hz), 6.98 (dd, 1H, *J* = 2.4 and 8.6 Hz), 7.26 (d,1H, *J* = 9.2 Hz), 8.26 (s, 1H), 8.26 (br s, 1H) 8.42 (d, 1H, *J* = 10.0 Hz), 8.50 (d, 1H, *J* = 8.4Hz) ppm. ^{13}C nmr (75 MHz, $CDCl_3$) δ: 28.41, 29.19, 55.34, 112.37, 113.01, 113.19, 115.9, 119.39, 123.06, 126.96, 127.39, 127.63, 131.85, 134.18, 138.42, 140.98, 144.66, 153.08, 153.37, 160.80, 161.74 ppm. IR (KBr)ν_{max}: 1730.8 (strong sharp) cm^{-1}; hrms (ESI, 70 eV); *m/z*: 330.1122 (M^++H) [calculated mass for $C_{21}H_{16}NO_3$: 330.1085 (M^++H)]. Compound **4e**: Light yellow solid, yield 50%, mp 242–244 °C ($CHCl_3$ /pet. ether 60–80 °C).^{1}H nmr (200 MHz, $CDCl_3$) δ: 3.00 (br t, 2 H, *J* = 7.2 Hz), 3.22 (br t, 2H, *J* = 4.0 Hz), 6.62 (d, 1H, *J* = 9.8 Hz), 7.32 (s, 1H), 7.42–7.52 (m, 2H), 7.65 (dd, 1H, *J* = 3.4 and 9.0 Hz), 8.29 (br d, 2H, *J* = 6.8), 8.43 (dd, 1H, *J* = 2.2 Hz and 9.6 Hz) ppm.; IR (KBr) ν_{max}: 1717.3 (strong sharp) cm^1; hrms (ESI, 70 eV); *m/z*: 378.0156 (M^++H), 380.0078 (M^++2 + H) [calculated mass for $C_{20}H_{13}BrNO_2$: 378.0130 (M^++H), 380.0109 (M^++2 + H)]. Compound **4f**: Light yellow solid, yield 50%, mp 249–251 °C (benzene/EtOAc). ^{1}H nmr (500 MHz, $CDCl_3$) δ: 6.66 (d, 1H, *J* = 9.9 Hz), 7.68 (d, 1H, *J* = 8. 9 Hz), 7.78 (br t, 1H, *J* = 7.3 Hz), 7.84 (br t, 1H, *J* = 7.2 Hz), 8.03 (d, 1H, *J* = 8.1 Hz), 8.07 (d, 1H, *J* = 8.2 Hz), 8.13 (d, 1H, *J* = 7.0 Hz), 8.38 (d, 1H, *J* = 9.0 Hz), 8.45 (d, 1H, *J* = 6.9 Hz), 8.59 (d, 1H, *J* = 9.4 Hz) , 8.90 (s, 1H) ppm; IR (KBr) ν_{max}: 1715.23 (strong sharp) cm^{-1}; hrms (ESI, 70 eV); *m/z*: 322.0921 (M^++H) [calculated mass for $C_{22}H_{12}NO_2$: 322.0868 (M^++H)].

Aromatization of dihydropyrido coumarin derivatives 4(c–e); preparation of 5(c–e): General method. A mixture of 0.30 mmol of dihydropyrido coumarin derivatives **4(c–e)** and 0.31 mmol of DDQ in 25 ml chlorobenzene was refluxed for 12–14 h under argon atmosphere. The chlorobenzene was removed under reduced pressure and the residue was taken up in excess of chloroform and filtered a short path of silica gel. Removal of the solvent afforded the crude product that was further purified by column chromatography (silica gel/ benzene-EtAc, 10:1) in 92–95% yield.

Compound **5c**: Light yellow solid, yield 92%, mp 255–257 °C (benzene/EtOAc). ^{1}H nmr (500 MHz, DMSO-d_6) δ: 6.82 (d, 1H, J = 9.8 Hz), 7.86 (dd, 2H, J = 3.8 and 6.0 Hz), 7.98 (d, 1H, J = 9.4 Hz), 8.01 (s, 2H), 8.09 (dd, 1H, J = 3.5 and 5.9 Hz), 8.53 (d, 1H, J = 9.4 Hz), 9.11 (d, 1H, J = 9.9 Hz), 9.39 (br dd, 1H, J ~ 3.0 and 9.0 Hz), 9.71 (s, 1H) ppm; IR (KBr) ν_{max}: 1736.58 (strong) cm^{-1}; hrms (ESI, 70 eV); *m/z*: 298.0859 (M^++H) [calculated mass for $C_{20}H_{12}NO_2$: 298.0868 (M^++H)]. Compound **5d**: Light yellow solid, yield 95%, mp 252–254 °C (benzene-EtOAc). ^{1}H nmr (500 MHz, DMSO-d_6) δ: 3.98 (s, 3H), 6.80 (d, 1H, J = 9.8 Hz), 7.44 (dd, 1H, J = 2.4 and 8.9 Hz), 7.57 (d, 1H, J = 2.5 Hz), 7.93 (d, 1H, J = 9.0 Hz), 7.94 (d, 1H, J = 9.4 Hz), 7.97 (d, 1H, J = 9.0 Hz), 8.46 (d, 1H, J = 9.4 Hz), 9.08 (d, 1H, J = 9.8 Hz), 9.25 (d, 1H, J = 8.9 Hz), 9.64 (s, 1H) ppm; IR (KBr) ν_{max}: 1730.8 (strong, sharp) cm^{-1} Hrms (ESI, 70 eV); *m/z*: 328.00938 (M^++H) [calculated mass for $C_{21}H_{14}NO_3$: 328.0974 (M^++H)]. Compound **5e**: Light yellow solid, yield 95%, mp 255–257 °C (benzene/EtOAc). ^{1}H nmr (400 MHz, DMSO) δ: 6.82 (d, 1H, J = 9.7 Hz), 7.98–8.08 (m, 5H), 8.55 (d, 1H, J = 9.3 Hz), 9.09 (d, 1H, J = 9.8 Hz), 9.46 (d, 1H, J = 9.8 Hz), 9.73 (s, 1H) ppm; IR (KBr) ν_{max}: 1718.26 (broad) cm^{-1}; hrms (ESI, 70 eV); *m/z*: 376.0053 (M^++H) 378.0078 (M^++2 + H) [calculated mass for $C_{20}H_{11}BrNO_2$: 375.9973 (M^++H), 377.9953 (M^++2 + H)].

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