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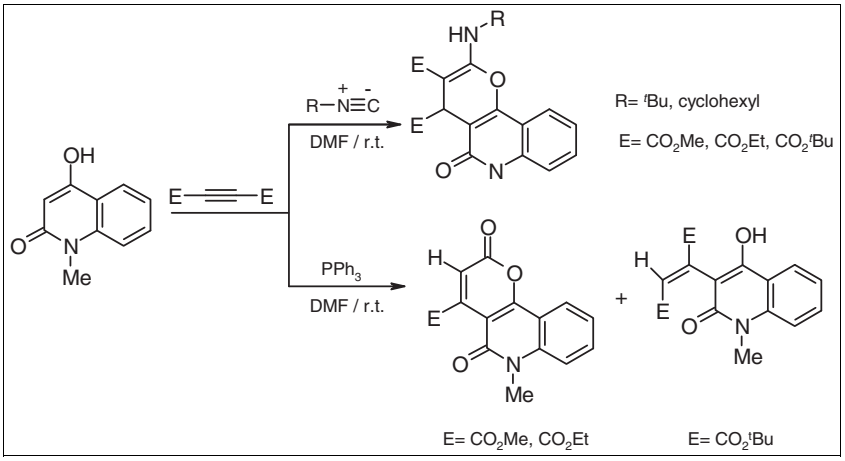

Pyranoquinoline derivatives have been synthesized via three-component reaction of 4-hydroxy-1-methyl-2(1*H*)-quinolinone and dialkyl acetylenedicarboxylates in the presence of nucleophiles such as alkyl isocyanides and triphenylphosphine.

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INTRODUCTION

Pyranoquinoline moiety is a structural feature of many natural bioactive alkaloids such as simulenoline [1], huajiaosimuline [2], and flindersine [3]. In recent years, pyranoquinoline derivatives have gained significant importance because of their diverse biological activities such as anti-inflammatory [4], sterogenic [5], and anti-allergic [6] and their pharmacological activities such as anti-coagulant [7], coronary constricting [8], anti-fungal [9], and cancer cell growth inhibitory activity [10]. In view of the highly pronounced biological and pharmacological activities of pyranoquinolines, several methods have been reported for the synthesis of these tricyclic compounds [11–14]. Herein, as part of our efforts to the synthesis of novel heterocyclic systems [15–17], we describe an efficient approach for the synthesis of pyranoquinoline derivatives via a three-component reaction using 4-hydroxy-1-methyl-2(1*H*)-quinolinone **1** and dialkyl acetylenedicarboxylates **2** in the presence of nucleophiles such as alkyl isocyanides **3** and triphenylphosphine **4**.

The reaction of 4-hydroxy-1-methyl-2(1*H*)-quinolinone **1** and dialkyl acetylenedicarboxylates **2** in the presence of alkyl isocyanides **3** proceeds in DMF at room temperature to produce compounds **5a–e** in good yields (Scheme 1).

Under similar conditions, the reactions of **1** with dialkyl acetylenedicarboxylates **2** in the presence of triphenylphosphine **4** lead to products **6a–b** and **7** in moderate yields (Scheme 2). In the case of using *tert*-butyl derivative of diester, the cyclization reaction did not occur may be because of the hindrance of the *tert*-butyl group. Therefore, the chain product **7** was obtained.

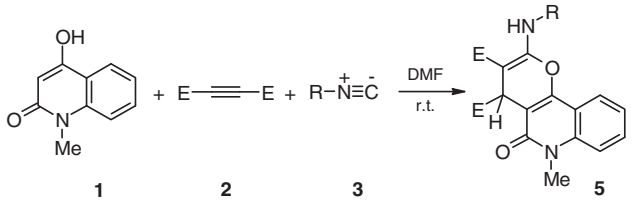
The reaction was carried out in the presence of alkyl acetylenecarboxylates instead of dialkyl acetylenedicarboxylates, but no result was found with acetylenic monoesters probably because of the less electrophilicity of β carbon in monoesters than diesters.

RESULTS AND DISCUSSION

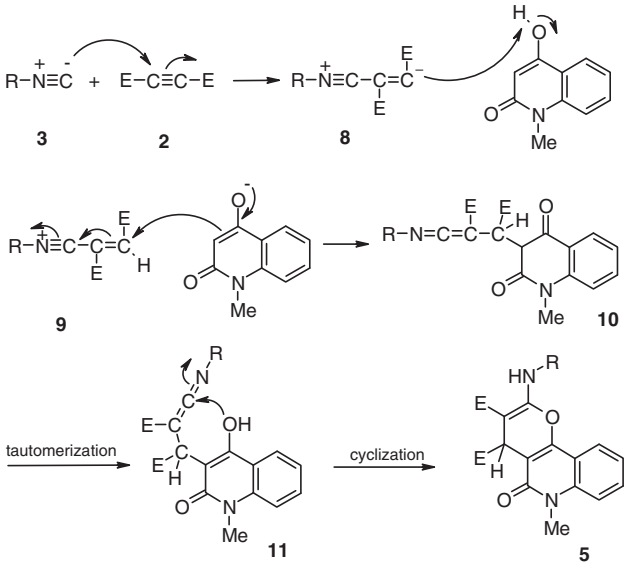
On the basis of the chemistry of isocyanides [18–20], it is conceivable that an initial addition of alkyl isocyanides **3** to dialkyl acetylenedicarboxylates **2** and subsequently the protonation of 1:1 adduct **8** by the *OH*-acid **1** leads to **9**, which can be attacked by the conjugated base of 4-hydroxy-1-methyl-2(1*H*)-quinolinone to generate intermediate **10**. This intermediate is converted to **11** by tautomerization, and then a cyclization reaction occurs to produce **5** (Scheme 3).

A plausible explanation for the formation of **6** is shown in Scheme 4. According to the chemistry of trivalent phosphorus nucleophiles [21], the initial event is the nucleophilic addition of PPh_3 **4** to the acetylenic diester **2** to form the zwitterion **12**. Subsequent protonation of 1:1

Scheme 1



Scheme 3



adduct **12** by the *OH*-acid **1** leads to **13**. Then, the positively charged ion can be attacked by the conjugated base of **1** to produce ylide **14**, which is converted to **15** via tautomerization and then [1,2]-proton transfer. Finally, compound **15** with elimination of PPh_3 leads to **7**, which undergoes a cyclization reaction to produce **6**.

The structures of the products were fully consistent with their ^{1}H NMR, ^{13}C NMR, IR and mass spectra, and elemental analysis.

The ^{1}H NMR spectrum of **5a** exhibited a sharp singlet for *tert*-butyl at 1.58 ppm, a singlet for NCH_3 at 3.70 ppm, two singlets for two methoxy groups at 3.74 and 3.76 ppm, a singlet for CH at 4.90 ppm, a multiplet for aromatic protons at 7.33–8.04 ppm, and a singlet for NH proton at 9.06 ppm. The ^{13}C NMR spectrum of **5a** displayed 19 resonances in agreement with the proposed structure. IR spectral data of **5a** displayed four absorption bands at 3380, 1731, 1683, and 1630 cm^{-1} indicating the presence of NH and carbonyl groups, respectively. The mass spectrum of **5a** displayed a molecular ion peak at *m/z* 400. The fragmentations involved the loss of side chains such as CO_2Me and C_4H_8.

The ^{1}H and ^{13}C NMR spectra of **5b–e** are similar to those of **5a**, except for the signals of alkoxy and cyclohexyl groups.

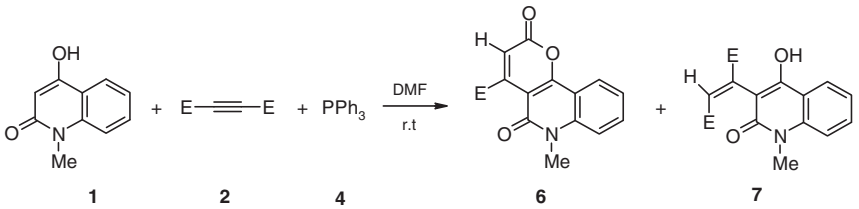
The ^{1}H NMR spectrum of **6a** exhibited three singlets for NCH_3, OCH_3, and CH groups at 3.75, 4.05, and 6.37 ppm along with a multiplet for aromatic protons at 7.40–8.30 ppm. The ^{13}C NMR spectrum of **6a** displayed 15 resonances in agreement with the proposed structure. The IR spectrum of **6a** displayed three absorption bands at 1740, 1658, and 1598 cm^{-1}, indicating the presence of carbonyl groups. The mass spectrum of **6a** displayed a molecular ion peak at *m/z* 285. The ^{1}H and ^{13}C NMR spectra of **6b** are similar to those of **6a**, except for the signals of the alkoxy groups.

The ^{1}H NMR spectrum of **7** exhibited three singlets for 2 CMe_3 and NCH_3 groups at 1.44, 1.50, and 3.69 ppm; a singlet for olefinic proton at 6.93 ppm; and a multiplet for aromatic protons at 7.26–8.20 ppm. The ^{13}C NMR spectrum of **7** displayed 18 resonances in agreement with the proposed structure. The IR spectrum of **7** displayed four absorption bands at 3316, 1713, 1690, and 1614 cm^{-1}, indicating the presence of OH and carbonyl groups, respectively. The mass spectrum of **7** displayed a molecular ion peak at *m/z* 401.

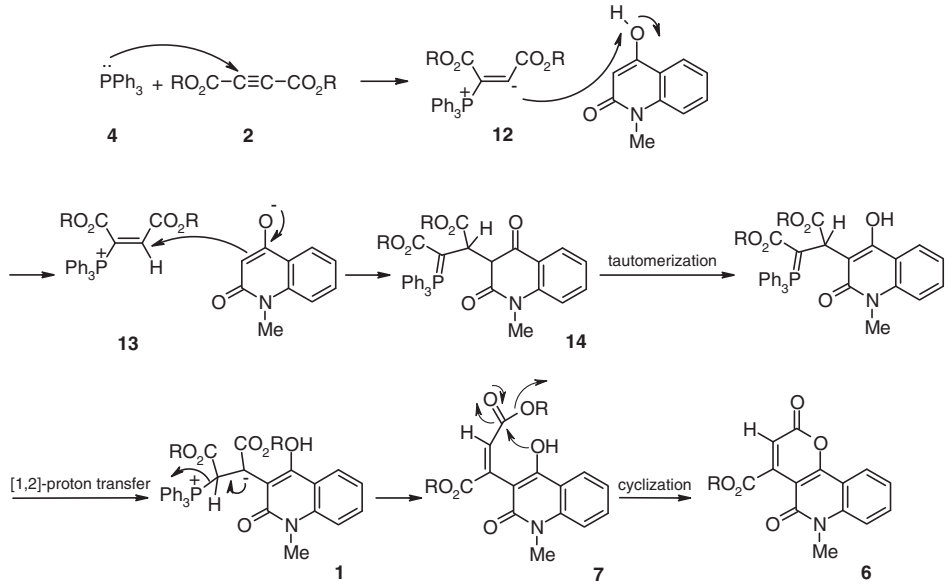
CONCLUSIONS

In conclusion, we have described a mild and efficient method for the synthesis of pyranoquinoline derivatives via a one-pot three-component reaction of 4-hydroxy-1-methyl-2(1*H*)-quinolinone and dialkyl acetylenedicarboxylates with alkyl isocyanides and PPh_3 under neutral conditions.

Scheme 2



Scheme 4



EXPERIMENTAL

Elemental analyses were performed using a F002 Heraeus CHN-O-Rapid analyzer (Elementar Analysesysteme GmbH, Hanau, Germany) using acetanilide as a standard. NMR spectra were recorded with BRUCKER DRX-400 AVANCE spectrometer (at 400.1 MHz for ^{1}H and 100.6 MHz for ^{13}C) with $CDCl_3$ as solvent. Mass spectra were recorded on a Finnigan-Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a FTIR, Brucker, VECTOR 22 spectrometer. TLC was carried out on Fluka Silica gel TLC-cards. All other reagents and solvents were used as received from commercial suppliers. All of the coupling constants are given in hertz.

General procedure for the preparation of compound 5. To a magnetically stirred solution of 4-hydroxy-1-methyl-2(1*H*)-quinolinone (2 mmol) and dialkyl acetylenedicarboxylate (2 mmol) in DMF (5 ml), 2 mmol of *tert*-butyl isocyanide was added dropwise at room temperature over 10 min. Then, the reaction mixture was stirred for 24 h. The solvent was removed under reduced pressure, and the residue was purified by column chromatography with the use of hexane: ethyl acetate (60:40) as eluent to take pure product **5a** as a white powder.

Dimethyl 2-(*tert*-butylamino)-6-methyl-5-oxo-5,6-dihydro-4*H*-pyrano[3,2-*c*]quinoline-3,4-dicarboxylate (5a, $C_{21}H_{24}N_2O_6$). White powder, yield: 80%. mp 189–191 °C. IR (KBr) (υ_{max}/cm^{-1}): 3380 (NH), 1731, 1683, and 1630 (3C═O). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ = 1.58 (s, 9H, CMe_3), 3.70 (s, 3H, NCH_3), 3.74 and 3.76 (2s, 6H, $2OCH_3$), 4.90 (s, 1H, CH), 7.33–8.04 (m, 4H, aromatic protons), 9.06 (s, 1H, NH). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = 29.81 (NCH_3), 30.55 (NC*Me*$_3$), 36.59 (CH), 51.11 (N*C*Me$_3$), 52.47 and 52.58 ($2OCH_3$), 73.03 (NH–C═*C*), 108.16 (N–CO–*C*═C), 113.90, 114.47, 122.33, 122.68, 131.37 and 139.18 (aromatic carbons), 151.88 (N–CO–C═*C*), 160.37 and 161.31 (2CO), 169.93 (NH–C═C), 173.87 (NC═O). MS: *m*/*z* (%): 400 (M^+, 3), 341 [$M^+ - CO_2Me$, 53], 292 [$M^+ - (2CH_3OH + CH_3 + NCH_3)$, 100], 261 [$M^+ - (2CH_3OH + 3CH_3 + NHCH_3)$, 62], 225 [$M^+ - (CO_2Me + C_4H_9)$, 18], 77 ($Ph^+$, 23). *Anal*. Calcd for $C_{21}H_{24}N_2O_6$ (400.40): C, 62.99; H, 6.03; N, 6.99. Found: C, 62.92; H, 6.07; N, 6.97.

Diethyl 2-(*tert*-butylamino)-6-methyl-5-oxo-5,6-dihydro-4*H*-pyrano[3,2-*c*]quinoline-3,4-dicarboxylate (5b, $C_{23}H_{28}N_2O_6$). White powder, yield: 85%. mp 150–152 °C. IR (KBr) (υ_{max}/cm^{-1}): 3238 (NH), 1726, 1689, and 1630 (3C═O). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ = 1.27 and 1.33 (t, 6H, $^3J_{HH}$ = 7.2 Hz, $2CH_3$), 1.57 (s, 9H, CMe_3), 3.73 (s, 3H, NCH_3), 4.15 (m, 2H, OCH_2), 4.17–4.25 (m, 2H, OCH_2), 4.89 (s, 1H, CH), 7.32–8.04 (m, 4H, aromatic protons), 9.08 (s, 1H, NH). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = 14.20 and 14.55 ($2CH_3$), 29.77 (NCH_3), 30.56 (NC*Me*$_3$), 36.74 (CH), 52.49 (N*C*Me$_3$), 59.69 and 61.05 ($2OCH_2$), 73.11 (NH–C═*C*), 108.35 (N–CO–*C*═C), 113.95, 114.41, 122.27, 122.70, 131.28 and 139.17 (aromatic carbons), 151.87 (N–CO–C═*C*), 160.28 and 161.38 (2CO), 169.63 (NH–C═C), 173.73 (NC═O). MS: *m*/*z* (%): 428 (M^+, 2), 383 [$M^+ - EtO$, 45], 355 [$M^+ - CO_2Et$, 40], 327 [$M^+ - (EtO + C_4H_8)$, 100], 271 [$M^+ - (CO_2Et + C_4H_8 + C_2H_4)$, 63], 225 [$M^+ - (2CO_2Et + C_4H_9)$, 22], 57 ($C_4H_9$, 21). *Anal*. Calcd for $C_{23}H_{28}N_2O_6$ (428.45): C, 64.48; H, 6.58; N, 6.54. Found: C, 64.51; H, 6.56; N, 6.57.

Di(*tert*-butyl) 2-(*tert*-butylamino)-6-methyl-5-oxo-5,6-dihydro-4*H*-pyrano[3,2-*c*]quinoline-3,4-dicarboxylate (5c, $C_{27}H_{36}N_2O_6$). White powder, yield: 76%. mp 188–190 °C. IR (KBr) (υ_{max}/cm^{-1}): 3249 (NH), 1726, 1667, and 1651 (3C═O). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ = 1.46 (s, 9H, CMe_3), 1.54 and 1.56 (2s, 18H, $2OCMe_3$), 3.73 (s, 3H, NCH_3), 4.75 (s, 1H, CH), 7.30–8.04 (m, 4H, aromatic protons), 9.00 (s, 1H, NH). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = 28.12 and 28.62 ($2OCMe_3$), 29.71 (NCH_3), 30.65 (NC*Me*$_3$), 38.12 (CH), 52.27 (N*C*Me$_3$), 74.79 (NH–C═*C*), 79.57 and 80.76 ($2OCMe_3$), 109.03 (N–CO–*C*═C), 114.11, 114.30, 122.09, 122.76, 131.03 and 139.14 (aromatic carbons), 151.97 (N–CO–C═*C*), 160.15 and 161.36 (2CO), 169.22 (NH–C═C), 173.16 (NC═O). MS: *m*/*z* (%): 484 (M^+, 4), 383 [$M^+ - (3CH_3 + C_4H_8)$, 48], 327 [$M^+ - (CO_2{}^tBu + C_4H_8)$, 100], 271 [$M^+ - (CO_2{}^tBu + 2C_4H_8)$, 70], 253 [$M^+ - (CO_2{}^tBu + C_4H_9O + C_4H_9)$, 50], 225 [$M^+ - (2CO_2{}^tBu + C_4H_8)$, 20], 57 ($C_4H_9$, 80). *Anal*. Calcd for $C_{27}H_{36}N_2O_6$ (484.55): C, 66.93; H, 7.48; N, 5.78. Found: C, 66.97; H, 7.50; N, 5.73.

Dimethyl 2-(cyclohexylamino)-6-methyl-5-oxo-,5,6-dihydro-4*H*-pyrano[3,2-*c*]quinoline-3,4-dicarboxylate (5d, $C_{23}H_{26}N_2O_6$). White powder, yield: 88%. mp 190–192 °C; IR (KBr) (υ_{max}/cm^{-1}): 3254 (NH), 1726, 1683, and 1651 (3C=O). ^1H NMR (400.1 MHz, $CDCl_3$): $\delta = 1.40–2.13$ (m, 10H, $5CH_{2cy}$), 3.70 (s, 3H, NCH_3), 3.74 and 3.76 (2s, 6H, $2OCH_3$), 3.92–3.95 (m, 1H, CH_{cy}), 4.88 (s, 1H, CH), 7.32–7.88 (m, 4H, aromatic protons), 8.74–8.75 (bd, 1H, $^3J_{HH} = 7.6$ Hz, NH). ^{13}C NMR (100.6 MHz, $CDCl_3$): $\delta = 24.50$, 24.56, 25.49, 33.49 and 33.79 ($5CH_{2cy}$), 29.76 (NCH_3), 36.78 (CH), 50.55 (CH_{cy}), 51.04 and 52.48 ($2OCH_3$), 72.30 (NH–C=*C*), 108.08 (N–CO–*C*=C), 113.96, 114.38, 122.24, 122.35, 131.44 and 139.18 (aromatic carbons), 151.66 (N–CO–C=*C*), 159.08 and 161.30 (2CO), 169.75 (NH–C=C), 173.95 (NC=O). MS: *m/z* (%): 426 (M^+, 2), 367 ($M^+ - CO_2Me$, 100), 285 [$M^+ - (C_6H_{10} + CO_2Me)$, 17], 253 [$M^+ - (C_6H_{10} + CO_2Me + CH_3OH)$, 51], 225 [$M^+ - (C_6H_{11} + 2CO_2Me)$, 17], 83 ($C_6H_{11}$, 16). *Anal.* Calcd for $C_{23}H_{26}N_2O_6$ (426.44): C, 64.78; H, 6.14; N, 6.57. Found: C, 64.81; H, 6.17; N, 6.60.

Diethyl 2-(cyclohexylamino)-6-methyl-5-oxo-5,6-dihydro-4*H*-pyrano[3,2-*c*]quinoline-3,4-dicarboxylate (5e, $C_{25}H_{30}N_2O_6$). White powder, yield: 85%. mp 177–178 °C. IR (KBr) (υ_{max}/cm^{-1}): 3249 (NH), 1715, 1688, and 1646 (3C=O). ^1H NMR (400.1 MHz, $CDCl_3$): $\delta = 1.27$ and 1.33 (2t, 6H, $^3J_{HH} = 7.2$ Hz, $2CH_3$), 1.39–1.51 (m, 10H, $5CH_{2cy}$), 3.73 (s, 3H, NCH_3), 3.92–3.96 (m, 1H, CH_{cy}), 4.10–4.28 (2m, 4H, $2OCH_2$), 4.87 (s, 1H, CH), 7.30–7.87 (m, 4H, aromatic protons), 8.31 (d, 1H, $^3J_{HH} = 7.6$ Hz, NH). ^{13}C NMR (100.6 MHz, $CDCl_3$): $\delta = 14.20$ and 14.55 ($2CH_3$), 24.51, 24.58, 25.51, 33.51 and 33.80 ($5CH_{2cy}$), 29.76 (NCH_3), 36.92 (CH), 50.51 (CH_{cy}), 59.58 and 61.05 ($2OCH_2$), 72.41 (NH–C=*C*), 108.25 (N–CO–*C*=C), 114.01, 114.31, 122.25, 122.27, 131.33 and 139.17 (aromatic carbons), 151.65 (N–CO–C=*C*), 158.99 and 161.37 (2CO), 169.45 (NH–C=C), 173.80 (NC=O). MS: *m/z* (%): 454 (M^+, 2), 381 ($M^+ - CO_2Et$, 100), 299 [$M^+ - (CO_2Et + C_6H_{10})$, 11], 253 [$M^+ - (CO_2Et + C_6H_{10} + EtOH)$, 46], 225 [$M^+ - (2CO_2Et + C_6H_{11})$, 16], 77 (Ph, 4). *Anal.* Calcd for $C_{25}H_{30}N_2O_6$ (454.49): C, 66.07; H, 6.65; N, 6.16. Found: C, 66.11; H, 6.68; N, 6.12.

Methyl 6-methyl-2,5-dioxo-5,6-dihydro-2*H*-pyrano[3,2-*c*] quinoline-4-carboxylate (6a, $C_{15}H_{11}NO_5$). Yellow powder, yield: 60%. mp 160–162 °C. IR (KBr) (υ_{max}/cm^{-1}): 1740, 1658, and 1598 (3C=O). ^1H NMR (400.1 MHz, $CDCl_3$): $\delta = 3.75$ (s, 3H, NCH_3), 4.05 (s, 3H, OCH_3), 6.37 (s, 1H, CH), 7.40–8.30 (m, 4H, aromatic protons). ^{13}C NMR (100.6 MHz, $CDCl_3$): $\delta = 29.34$ (NCH_3), 53.39 (OCH_3), 105.05 (N–CO–*C*=C), 112.66, 113.15, 114.77, 123.32, 124.37 and 140.09 (aromatic carbons), 134.10 (=CH), 147.41 (*C*–CO_2Et), 158.40, 159.45 and 159.50 (3CO), 166.05 (C=*C*–O). MS: *m/z* (%): 285 (M^+, 100), 254 ($M^+ - CH_3O$, 39), 226 ($M^+ - CO_2Me$, 42), 170 [$M^+ - (CO_2Me + C_4H_8)$, 50], 104 (NC_7H_6, 32). *Anal.* Calcd for $C_{15}H_{11}NO_5$ (285.24): C, 63.16; H, 3.88; N, 4.91. Found: C, 63.18; H, 3.85; N, 4.94.

Ethyl 6-methyl-2,5-dioxo-5,6-dihydro-2*H*-pyrano[3,2-*c*] quinoline-4-carboxylate (6b, $C_{16}H_{13}NO_5$). Yellow powder, yield: 58%. mp 184–186 °C. IR (KBr) (υ_{max}/cm^{-1}): 1740, 1656, and 1598 (3C=O). ^1H NMR (400.1 MHz, $CDCl_3$): $\delta = 1.44$ (t, 3H, $^3J_{HH} = 7.2$ Hz, CH_3), 3.75 (s, 3H, NCH_3), 4.52 (q, 2H, $^3J_{HH} = 7.2$ Hz, OCH_2), 6.36 (s, 1H, CH), 7.28–8.29 (m, 4H, aromatic protons). ^{13}C NMR (100.6 MHz, $CDCl_3$): $\delta = 14.04$ (CH_3), 29.83 (NCH_3), 62.73 (OCH_2), 105.05 (N–CO–*C*=C), 112.58, 113.15, 114.73, 123.24, 124.34 and 140.25 (aromatic carbons), 134.05 (=CH), 147.80 (*C*–CO_2Et), 158.26, 158.51 and 159.43 (3CO), 165.53 (C=*C*–O). MS: *m/z* (%): 299 (M^+, 67), 254 ($M^+ - EtOH$, 37), 227 [$M^+ - (CO_2Et + CO_2H)$, 100], 170 [$M^+ - (CO_2Et + C_2H_2 + NHCH_3)$, 57], 104 ($NC_7H_6$, 24). *Anal.* Calcd for $C_{16}H_{13}NO_5$ (299.26): C, 64.22; H, 4.37; N, 4.68. Found: C, 64.25; H, 4.39; N, 4.65.

Di(*tert*-butyl) (*E*)-2-(4-hydroxy-1-methyl-2-oxo-1,2-dihydro-3-quinolinyl)-2-butenedioate (7, $C_{22}H_{27}NO_6$). Yellow powder, yield: 50%. mp 192–194 °C. IR (KBr) (υ_{max}/cm^{-1}): 3316 (OH), 1713, 1690, and 1614 (3C=O). ^1H NMR (400.1 MHz, $CDCl_3$): $\delta = 1.43$ and 1.50 (2s, 18H, $2CMe_3$), 3.68 (s, 3H, NCH_3), 6.93 (s, 1H, CH), 7.26–8.20 (m, 4H, aromatic protons), 9.16 (s, 1H, OH). ^{13}C NMR (100.6 MHz, $CDCl_3$): $\delta = 27.82$ and 27.89 ($2CMe_3$), 29.38 (NCH_3), 82.96 and 83.37 ($2OCMe_3$), 108.54 (N–CO–*C*=C), 113.67, 117.07, 121.76, 125.11, 131.59 and 139.70 (aromatic carbons), 129.98 (=CH), 138.96 (*C*=CH), 158.38 (NC=O), 162.31 (C=*C*–OH), 166.78 and 167.50 (2CO). MS: *m/z* (%): 401 (M^+, 2), 299 ($M^+ - CO_2{}^tBu + 1$, 12), 225 [$M^+ - (CO_2{}^tBu + C_4H_9O)$, 17], 195 [$M^+ - (CO_2{}^tBu + C_4H_{10}O + NCH_3)$, 8], 167 [$M^+ - (2CO_2{}^tBu + NHCH_3 + 2H)$]. *Anal.* Calcd for $C_{22}H_{27}NO_6$ (401.43): C, 65.82; H, 6.77; N, 3.49. Found: C, 65.79; H, 6.72; N, 3.46.

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