A three-component reaction between azaarenes (phenanthridine, quinoline or isoquinoline), acetylenic esters and phenol derivatives

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Three component condensation reactions between azaarenes (phenanthridine, quinoline or isoquinoline) and acetylenic esters were undertaken in the presence of phenol derivatives (2,4-di-*tert*-butylphenol, 2,6-dimethylphenol, 4-methylphenol and 4-chloro-3,5-dimethylphenol) for generation of *C*-arylation in good yields. The reactions proceeded smoothly at room temperature without using any catalyst.

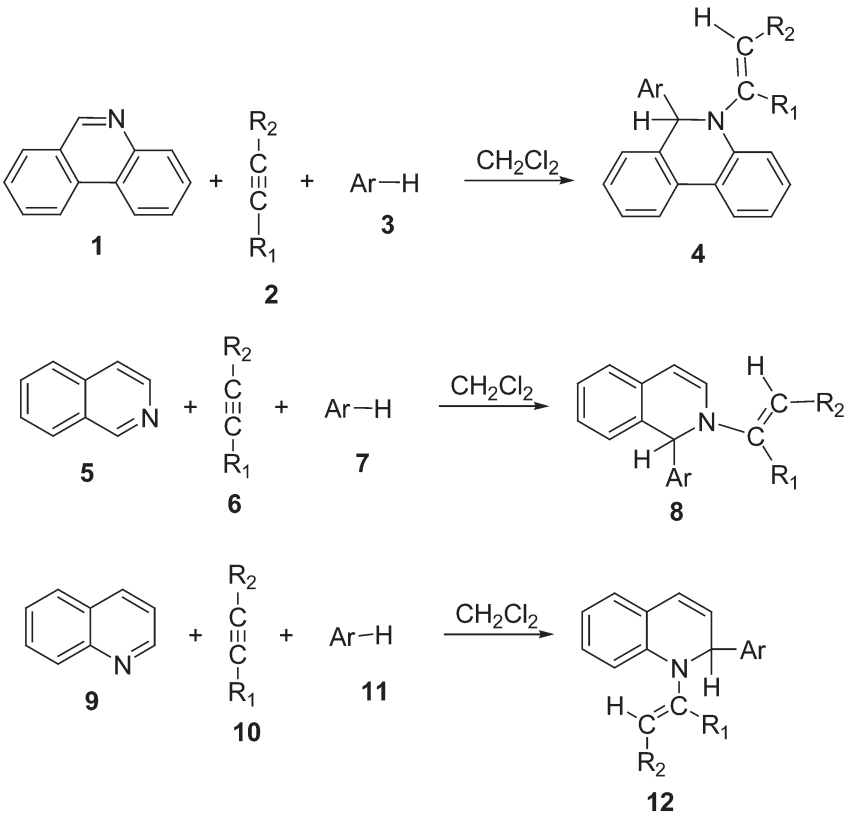
Keywords: phenanthridine, isoquinoline, quinoline, acetylenic esters, phenol derivatives, C-arylation

Introduction

Azaarenes are a group of substituted polycyclic aromatic hydrocarbons (PAHs) where one carbon atom of the aromatic ring is replaced by a nitrogen atom.[1] They are found in environmental matrices such as ambient air, lake and marine sediment, and seawater. They are also found in stack gases from coal combustion and automobile exhaust gases.[2–5] The isoquinoline skeleton is found in a large number of naturally occurring and synthetic biologically active heterocyclic compounds.[6,7] These compounds exhibit sedative,[8] antidepressant[9] and antimicrobial activities.[10]Quinoline is a representative nitrogen heterocyclic compounds that is commonly generated by coking plants, pharmaceutical factories, and related industries.[11]A number of biological activities have been associated with quinoline-containing compounds such as anti-flammatory, antimalarial, antibacterial, anticancer and antiparasitic.[12,13] Substituted phenanthridines are an important class of heterocyclic compounds in material science[14] and in medicinal chemistry due to their significant biological activities,[15–17] including antifungal, antimicrobial and anti-inflammatory.[18] I now describe the reaction between azaarenes (phenanthridine, quinoline and isoquinoline) and acetylenic esters in the presence of phenol derivatives (see Scheme 1 and Table 1).

Results and discussion

The reaction between azaarenes (phenanthridine **1**, isoquinoline **5** or quinoline **9**) and acetylenic esters **2**, **6** or **10** as a Michael acceptor[19–24] was undertaken in the presence of phenol derivatives (2,5-dimethylphenol, 2,4-di-*tert*-butylphenol, 4-methylphenol and 4-chloro-3,5-dimethylphenol) at ambient temperature (see Scheme 2 and Table 1). Reactions were carried out by first mixing the phenanthridine, isoquinoline or quinoline and phenol derivatives and then the acetylenic ester was added slowly. The reactions proceeded smoothly in CH_2Cl_2 and then the whole reaction mixture solidified into yellow or brown solid within a few hours. The ^1H and ^{13}C NMR spectra



Scheme 1

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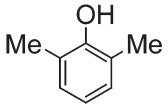
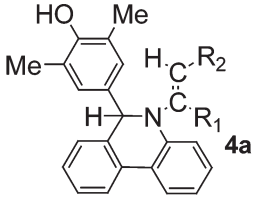
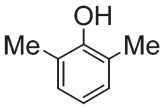
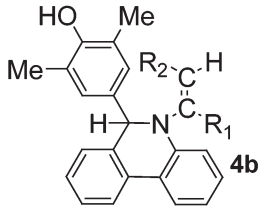
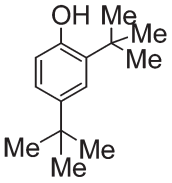
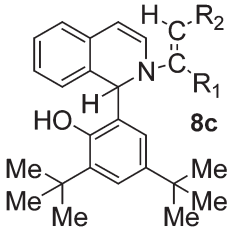
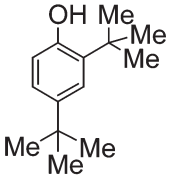
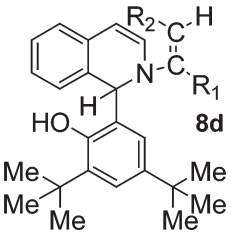
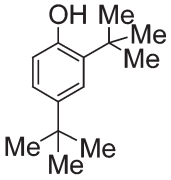
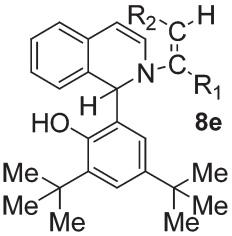
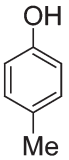
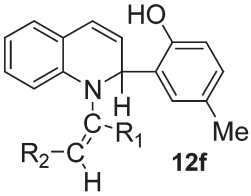
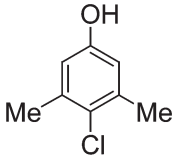
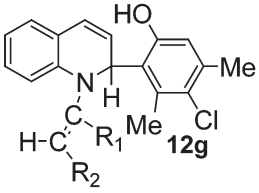
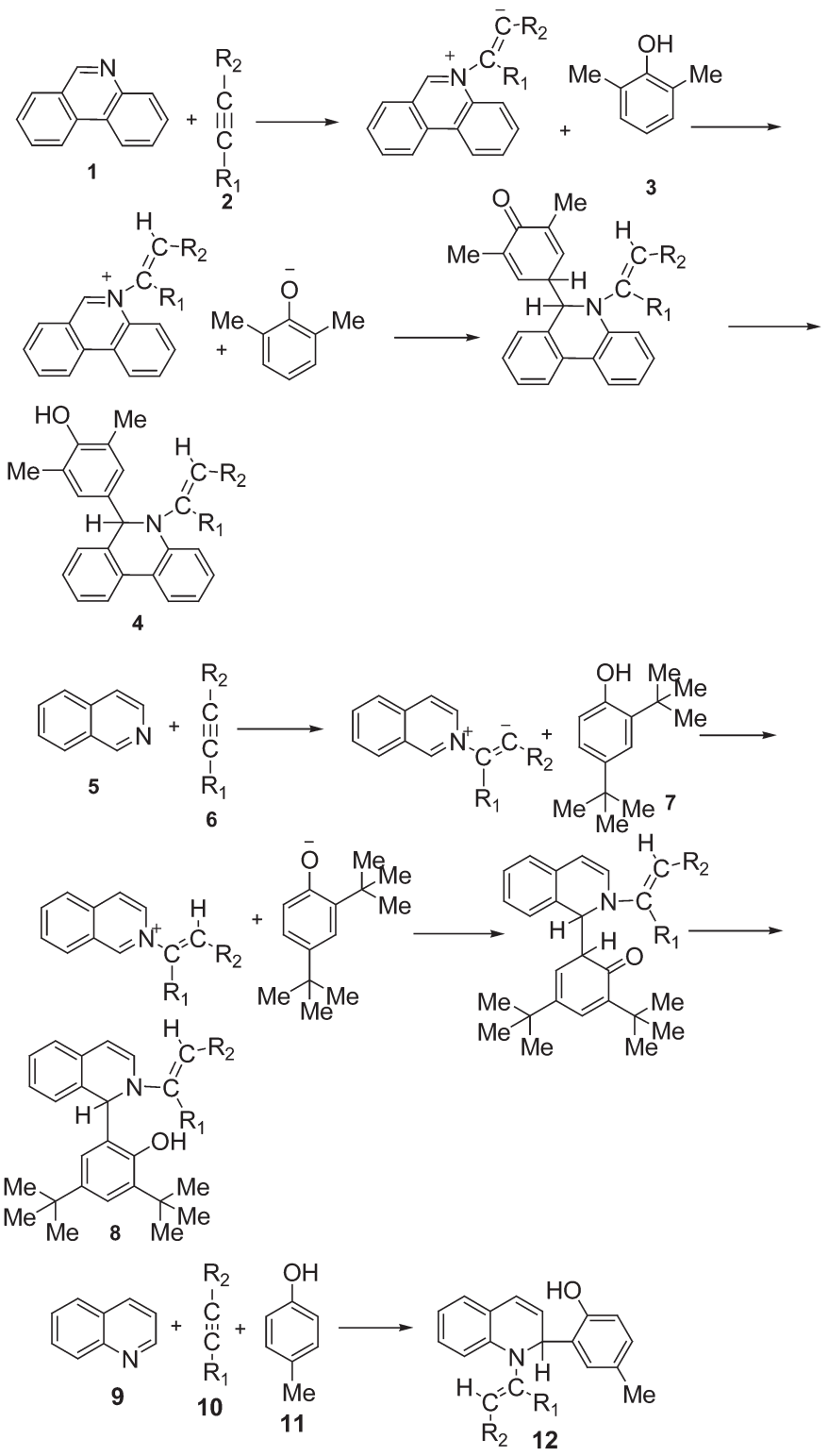
Table 1 Reaction between azaarenes with acetylenic esters in the presence of phenol derivatives

Entry	ArH	R_1	R_2	Z or E	Product	Yield/%
1		H	CO_2Et	E	 **4a**	88%
2		CO_2Et	CO_2Et	Z	 **4b**	91%
3		H	CO_2Me	E	 **8c**	90%
4		CO_2Me	CO_2Me	Z	 **8d**	94%
5		CO_2Et	CO_2Et	Z	 **8e**	91%
6		CO_2Me	CO_2Me	Z	 **12f**	92%
7		H	CO_2Me	E	 **12g**	89%

of the crude products clearly indicated the formation of compounds **4a–b**, **8c–e** and **12f–g**. No product other than **4a–b**, **8c–e** and **12f–g** could be detected by NMR spectroscopy. The structures of compounds **4a–b**, **8c–e** and **12f–g** were confirmed by elemental analyses, mass, IR, ^{1}H NMR and ^{13}C NMR spectra. The ^{1}H NMR 500 MHz spectrum of **4a** exhibited two signals identified as methyl (δ = 1.31, 3H, t, $^3J_{HH}$ = 7.00 Hz , OCH_2CH_3 and δ = 2.28, 6H, s, 2Me, phenol), methylene (δ = 4.21, 2H, q, $^3J_{HH}$ = 7.00 Hz, OCH_2CH_3), olefinic protons (δ = 5.51 and 8.22, 2d, $^3J_{HH}$ = 13.6 Hz, N-C*H*=C*H*-$CO_2CH_2CH_3$), a sharp line at δ = 6.69 ppm for the OH group, and also a sharp line for methine proton (δ = 9.30 ppm, 1H, s, NC*H*C). Aromatic protons, along with multiplets at δ = 6.74–8.15 ppm for the phenanthridine and phenol moiety. The ^{13}C NMR spectrum

of **4a** showed **26** distinct resonances in agreement with the proposed structure. In addition, product **4a** displayed ^{13}C NMR resonances at δ = 63.14, 109.35 and 123.90 ppm, respectively for the N*C*HC, N-CH=*C*H-$CO_2CH_2CH_3$, and N-*C*H=CH-$CO_2CH_2CH_3$ units.[22–24] The carbonyl group resonance in the ^{13}C NMR spectrum of **4a** appear at δ = 168.35 ppm. The ^{1}H and ^{13}C NMR spectra of compounds **4b**, **8c–e** and **12f–g** are similar to those of **4a**. The ^{1}H NMR of each of the isolated products **4b**, **8d–e** and **12f** exhibited a N–C=C*H* proton signal at about 5.67–6.65 ppm, which is in agreement with the (*Z*) configuration[25,26] for the vinyl moiety in **4b**, **8d–e** and **12f**. (see Scheme 2 and Table 1).

Briefly, I now describe a new method to access a novel class of heterocyclic derivatives. The present procedure has the advantage that not only is the reaction performed under neutral conditions, but also the reactants can be mixed without any prior activation or modification. It seems that, this procedure is very useful to functionalise azaarenes in a one-pot operation.



Scheme 2

Experimental

Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus and a Shimadzu IR-460 spectrometer, respectively. The ^{1}H and ^{13}C NMR spectra were obtained with a Bruker DRX-500 Avance instrument using $CDCl_3$ as applied solvent and TMS as internal standard at 500.1 and 125.8 MHz respectively. In addition, the mass spectra were recorded on a GCMS-QP5050A mass spectrometer operating at an ionisation potential of 70 eV. Elemental analyses for C, H and N were performed using a Heraeus CHN-O-Rapid analyser. All the chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General procedure

Ethyl-(2E)-3-[6-(4-hydroxy-3,5-dimethylphenyl)phenanthridin-5(6H)-yl] acrylate (**4a**): To a magnetically stirred solution of phenanthridine (0.18 g, 1 mmol) and 2,6-dimethylphenol (0.12 g, 1 mmol) in CH_2Cl_2 (10 mL) was added, dropwise, a mixture of ethyl propiolate (1 mmol) in CH_2Cl_2 (5 mL) at –10°C over 10 min. After 5 hours stirring at ambient temperature, the whole reaction mixture solidified into a brown solid, the solvent was then removed under reduced pressure and product washed with cold diethyl ether (2×5 mL). Then the product was recrystallised from a mixture of acetonitrile and acetone. Brown powder, yield 88%, 0.35g, m.p. 88–90 °C, IR (ν_{max}, cm^{-1}): 1740 (C=O), 3200 (OH). MS, *m/z* (%) = 399 (M, 9), 370 (M-Et, 83), 326 (M-CO_2Et, 37), 179 ($C_{13}H_9N$, 100). Anal. Calcd for $C_{26}H_{25}NO_3$ (399.49): C, 78.17; H, 6.31; N, 3.50. Found: C, 78.01; H, 6.39; N, 3.61%. ^{1}H NMR (500.1 MHz, $CDCl_3$), δ_H 1.31 (3H, t, $^3J_{HH}$ = 7.00 Hz, OCH$_2$*CH$_3$*), 2.28 (6H, s, 2CH$_3$, phenol), 4.21 (2H, q, $^3J_{HH}$ = 7.00 Hz, O*CH*$_2$CH$_3$), 5.51 (1H, d, $^3J_{HH}$ = 13.6 Hz, N–CH=C*H*–CO$_2$Et), 6.69 (1H, s, OH), 6.74–8.15 (11H$_{aro}$, phenanthridine and phenol), 8.22 (1H, d, $^3J_{HH}$ = 13.6 Hz, N–C*H*=CH–CO$_2$Et), 9.30 (1H, s, NC*H*C). ^{13}C NMR (125.8 MHz, $CDCl_3$), 13.30 (OCH$_2$*CH3*), 28.17 (2CH$_3$), 58.29 (O*CH*$_2$CH$_3$), 63.14 (N*C*HC), 109.35 (N–C=*C*H–CO$_2$CH$_2$CH$_3$), 123.90 (N–*C*=CH–CO$_2$CH$_2$CH$_3$), 124.80, 127.37, 127.45, 127.90, 128.13, 128.56, 129.00, 129.17, 131.29, 131.82, 132.02, 133.41, 135.31, 141.07 and 152.60 (18C, phenanthridine and phenol), 168.35 (C=O, ester).

Diethyl 2-[6-(4-hydroxy-3,5-dimethylphenyl)phenanthridin-5(6H)-yl] maleate (**4b**): Yellow powder, yield 91%, 0.43g, m.p. 92–94 °C, IR (ν_{max}, cm^{-1}): 1720 and 1654 (C=O), 3280 (OH). MS, *m/z* (%) = 471 (M, 12), 426 (M-OEt, 46), 398 (M-CO_2Et, 71), 350 (M-C_8H_9O, 100), 179 ($C_{13}H_9N$, 86), 121 (C_8H_9O, 25). Anal. Calcd for $C_{29}H_{29}NO_5$ (471.55): C, 73.87; H, 6.20; N, 2.97. Found: C, 73.98; H, 6.11; N, 3.09%. ^{1}H NMR (500.1 MHz, $CDCl_3$), δH 0.79 and 0.98 (6H, 2t, $^3J_{HH}$ = 7.1 Hz, 2OCH$_2$*CH$_3$*), 2.35 (6H, s, 2Me, phenol), 3.70 and 3.81 (4H, 2m, 2ABX3system, 2O*CH*$_2$CH$_3$), 5.29 (1H, s, OH), 6.65 (1H, s, N–C=C*H*–CO$_2$CH$_2$CH$_3$), 6.72–8.08 (10H$_{aro}$, m, phenanthridine and phenol).^{13}C NMR (125.8 MHz, $CDCl_3$), 14.13 and 14.73 (2OCH$_2$*CH$_3$*), 27.50 (2CH$_3$, phenol), 57.12 and 60.51(2O*CH*$_2$CH$_3$), 65.13 (N*C*HC), 120.20 (1C, N–C=*C*H–CO$_2$CH$_2$CH$_3$), 123.19 (1C, N–*C*=CH–CO$_2$C(CH$_3$)$_3$), 123.59, 123.91, 124.26, 124,36, 125.75, 127.11, 127.36, 127.94, 128.34, 128.53, 129.71, 130.25, 141.61, 144.31, 151.89 and 152. 80 (18C$_{aro}$, phenanthridine and phenol), 165.82 and 167.70 (2C=O, ester).

Methyl-(2E) 3-[1-(3,5-di-tert-butyl-2-hydroxyphenyl]isoquinolin-2(1H)-yl)acrylate (**8c**): Brown powder, yield 90%, 0.38g, m.p. 113–115 °C, IR (ν_{max}, cm^{-1}): 1646 (C=O), 3197 (OH). MS, *m/z* (%) = 419 (M, 6), 388 (M-OMe, 63), 360 (M-CO_2Me, 47), 214 (M-$C_{14}H_{21}O$, 17), 205 ($C_{14}H_{21}O$, 25), 129 (C_9H_7N, 73), 85 ($C_4H_5O_2$, 41). Anal. Calcd for $C_{27}H_{33}NO_3$ (419.56): C, 77.30; H, 7.93; N, 3.34. Found: C, 77.10; H, 8.06; N, 3.30%. ^{1}H NMR (500.1 MHz, $CDCl_3$), δH 1.12 and 1.35 (18H, 2s, 2CMe$_3$), 3.83 (3H, s, OMe), 5.63 (1H, d, $^3J_{HH}$ = 13.4 Hz, N–CH=C*H*–CO$_2$Me), 5.76 (1H, s, OH), 6.63 (1H, s, NC*H*C), 6.95–8.65 (9H$_{aro}$, m, isoquinoline and phenol). ^{13}C NMR (125.8 MHz, $CDCl_3$), δC 27.34 (2C*Me*$_3$), 33.91 and 34.18 (2*C*Me$_3$), 51.76 (2OCH$_3$), 57.14 (N*C*HC), 93.16 (N–CH=*C*H–CO$_2$Me), 123.14 (N–*C*H=CH–CO$_2$Me), 125.15, 125.55, 126.32, 126.83, 127.20, 127.91, 128.38, 128.45, 129.35 132.46, 133.11, 141.71, 142.45 and 152.51 (14C, isoquinoline and phenol), 166.51 (C=O, ester).

Dimethyl 2-[1-(3,5-di-tert-butyl-2-hydroxyphenyl)isoquinolin-2(1H)-yl] maleate (**8d**): Brown powder, yield 94%, 0.45g, m.p. 96–98 °C, IR (ν_{max}, cm^{-1}): 1783 and 1722 (C=O), 3230 (OH). MS, *m/z* (%) = 477 (M, 6), 446 (M-OMe, 40), 418 (M-CO_2Me, 50), 359 (M-2CO_2Me, 48), 272 (M-$C_{14}H_{21}O$, 15), 205 ($C_{14}H_{21}O$, 22), 129 (C_9H_7N, 100). Anal. Calcd for $C_{29}H_{35}NO_5$ (477.59): C, 72.93; H, 7.38; N, 2.93. Found: C, 72.98; H, 7.31; N, 2.85%. ^{1}H NMR (500.1 MHz, $CDCl_3$), δH 1.19 and 1.45 (18H, 2s, 2OC*Me*$_3$), 3.61 and 3.93 (6H, 2s, 2OMe), 5.35 (1H, s, OH), 5.86 (1H, s, N–C=CH–CO_2Me), 6.13 (1H, d, $^3J_{HH}$ = 7.8 Hz, N–CH=C*H*, isoquinoline), 6.47 (1H, s, NC*H*C), 6.57 (1H, d, $^3J_{HH}$ = 7.8 Hz, N–C*H*=CH, isoquinoline), 7.03–8.52 (7H$_{aro}$, m, isoquinoline and phenol). ^{13}C NMR (125.8 MHz, $CDCl_3$), 30.35 and 31.52 (2C*Me*$_3$), 34.23 and 34.35 (2*C*Me$_3$), 51.01 and 52.32 (2CO$_2$*Me*), 91.13 (N*C*HC), 105.84 (1C, N–C=*C*H–CO$_2$Me), 122.97 and 123.57 (2C, isoquinoline and phenol), 124.89 (1C, N–*C*=CH–CO$_2$Me), 126.85, 127.46, 127.63, 128.81, 129.75, 130.47, 131.83, 134.90, 142.99 and 148.05 (12C, isoquinoline and phenol), 166.38 and 169.45 (2C=O, ester).

Diethyl 2-[1-(3,5-di-tert-butyl-2-hydroxyphenyl)isoquinolin-2(1H)-yl] maleate (**8e**): Brown powder, yield 91%, 0.46g, m.p. 115–117 °C, IR (ν_{max}, cm^{-1}): 1632 and 1741 (C=O), 3240 (OH). MS, *m/z* (%) = 505 (M, 7), 460 (M-OEt, 41), 432 (M-CO_2Et, 24), 300 (M-$C_{14}H_{21}O$, 67), 205 ($C_{14}H_{21}O$, 19), 129 (C_9H_7N, 100) . Anal. Calcd for $C_{31}H_{39}NO_5$ (505.65): C, 73.64; H, 7.77; N, 2.77. Found: C, 73.50; H, 7.85; N, 2.86%. ^{1}H NMR (500.1 MHz, $CDCl_3$), δ_H 1.15 (3H, t, $^3J_{HH}$ = 7.0 Hz, OCH$_2$*CH$_3$*), 1.28 (9H, s, C*Me*$_3$), 1.33 (3H, t, $^3J_{HH}$ = 7.0 Hz, OCH$_2$*CH$_3$*), 1.40 (9H, s, C*Me*$_3$), 4.28 (4H, m, 2ABX3system, 2O*CH*$_2$CH$_3$), 5.39 (1H, s, OH), 5.74 (1H, s, N–C=C*H*–CO$_2$CH$_2$CH$_3$), 6.12 (1H, d, $^3J_{HH}$ = 7.2 Hz, N–CH=C*H*, isoquinoline), 6.24 (1H,s, NC*H*C), 6.41 (1H, d, $^3J_{HH}$ = 7.2 Hz, N–C*H*=CH, isoquinoline), 6.66–8.55 (7H$_{aro}$, m, isoquinoline and phenol). ^{13}C NMR (125.8 MHz, $CDCl_3$), 13.87 and 14.20 (2OCH$_2$*CH$_3$*), 27.68 and 29.03 (2C*Me*$_3$), 56.80 and 57.11 (2O*CH*$_2$CH$_3$), 63.17 (N*C*HC), 94.45 (1C, N–C=*C*H–CO$_2$CH$_2$CH$_3$), 122.35 and 122.49 (2C, isoquinoline and phenol), 123.18 (1C, N–*C*=CH–CO$_2$CH$_2$CH$_3$), 125.10, 126.31, 127.37, 128.21, 128.39, 129.53, 130.11, 131.34, 133.60, 143.19, 145.56 and 151.36 (12C, isoquinoline and phenol), 169.17 and 171.65 (2C=O, ester).

Dimethyl 2-[2-(2-hydroxy-5-methylphenyl)quinolin-1(2H)-yl] maleate (**12f**): Brown powder, yield 92%, 0.35g, m.p. 102–104 °C, IR (ν_{max}, cm^{-1}): 1634 and 1729 (C=O), 3180 (OH). MS, *m/z* (%) = 379 (M, 8), 320 (M-CO_2Me, 40), 272 (M-C_7H_7O, 54), 261 (M-2CO_2Me, 32), 143 ($C_6H_7O_4$, 26), 129 (C_9H_7N, 100), 107 (C_7H_7O, 18). Anal. Calcd for $C_{22}H_{21}NO_5$ (379.40): C, 69.65; H, 5.58; N, 3.69. Found: C, 69.69; H, 5.65; N, 3.81%. ^{1}H NMR (500.1 MHz, $CDCl_3$), δH 2.29 (3H, s, Me), 3.66 and 3.72 (6H, 2s, 2OMe), 5.39 (1H, s, OH), 5.67 (1H, s, N–C=C*H*–CO$_2$Me), 6.55 (1H, s, NC*H*C), 6.61–8.33 (10Haro, m, quinoline and phenol). ^{13}C NMR (125.8 MHz, $CDCl_3$), δC 14.15 (Me), 51.83 and 52.11 (2OMe), 67.31 (N*C*HC), 73.97 (N–C=*C*H–CO$_2$CH$_3$), 114.12., 115.15, 116.15, 118.43, 121.07 and 121.13 (6C, quinoline and phenol), 124.14 (N–*C*=CH–CO$_2$CH$_3$), 125.86, 126.48, 128.96, 130.30, 141.67 and 151.80 (8C, quinoline and phenol), 167.53 and 169.80 (2C=O, ester).

Methyl (2E)-3-[2-(3-chloro-6-hydroxy-2,4-dimethylphenyl)quinolin-1(2H)-yl]acrylate (**12g**): Brown powder, yield 89%, 0.33g, m.p. 140–142 °C, IR (ν_{max}, cm^{-1}): 1732 (C=O), 3385 (OH). MS, *m/z* (%) = 369 (M, 8), 354 (M-Me, 50), 338 (M-OMe, 12), 310 (M-CO_2Me, 14), 129 (C_9H_7N, 100), 120 (C_8H_8O, 39). Anal. Calcd for $C_{21}H_{20}ClNO_3$ (369.89): C, 68.19; H, 5.45; N, 3.78. Found: C, 68.19; H, 5.46; N, 3.74%. ^{1}H NMR (500.1 MHz, $CDCl_3$), δ_H 2.27 and 2.36 (6H, 2Me, phenol), 3.80 (3H, OMe), 5.30 (1H,d, $^3J_{HH}$ = 13. 3 Hz, N-CH=C*H*–CO$_2$Me), 6.59 (1H, s, OH), 6.83 (1H, s, NC*H*C), 6.98–8.24 (8Haro. quinoline and phenol), 8.28 (1H, d, $^3J_{HH}$ = 13.3 Hz, N–C*H*=CH–CO$_2$Me). ^{13}C NMR (125.8 MHz, $CDCl_3$), 15.11 (Me), 51.31 (OMe), 65.48 (N*C*HC), 79.31 (1C, N–CH=*C*H–CO$_2$Me), 114.95, 117.37, 117.60, 118.69, 120.57 and 122.85 (6C, quinoline and phenol), 123.28 (1C, N–*C*H=CH–CO$_2$Me), 125.16, 127.31, 128.42, 132.96, 141.38, 143.19 and 152.13 (8C, quinoline and phenol), 169.99 (C=O, ester).

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