PALLADIUM CATALYSED COUPLING OF IODOQUINOLINES AND ACETYLENES - A NOVEL ENTRY TO THE PYRROLO[3,2,1-*ij*]-QUINOLINE NUCLEUS

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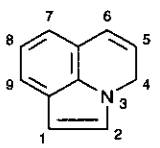
Abstract - Treatment of 4-arylamino-8-iodoquinoline derivatives with propargyl alcohol and triethylamine in the presence of iodo(phenyl)bis(triphenylphosphine) palladium and copper(I)iodide gives 6-imino-substituted pyrrolo[3,2,1-*ij*]quinolines under mild conditions and in moderate yields. Catalytic hydrogenation over Lindlar's catalyst affords functionalised *4H*-pyrrolo[3,2,1-*ij*]quinolines in good yield.

Diverse biological activities including antiinflammatory, antifungal, antihyperlipidemic, antihypertensive and serotonin receptor antagonism have been ascribed to compounds containing the pyrrolo[3,2,1-*ij*]quinoline nucleus (**1**).[1-5]



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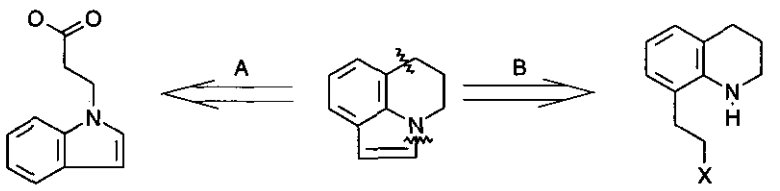
Black *et al.* have described[6] the synthesis of various 4-oxo-4*H*-pyrroloquinolines as well as the related dihydro-pyrroloquinolines starting from an appropriately substituted indole derivative. Similarly, the preparation of the corresponding 6-oxopyrroloquinolines has generally been based on the cyclodehydration of a suitably functionalised indole[7] (Scheme 1, Route A). More recently, Majumar[8] and independently, Rodighiero *et al.*[9] have developed a conceptually different approach to the pyrrolo[3,2,1-*ij*]quinoline system involving the construction of the five membered heterocyclic ring using a preformed quinoline as a template (Scheme 1, Route B). However, the overall yields using either method are low and the conditions used are often not compatible with the presence of sensitive functionality elsewhere in the molecule.



X = Br, COR

Scheme 1

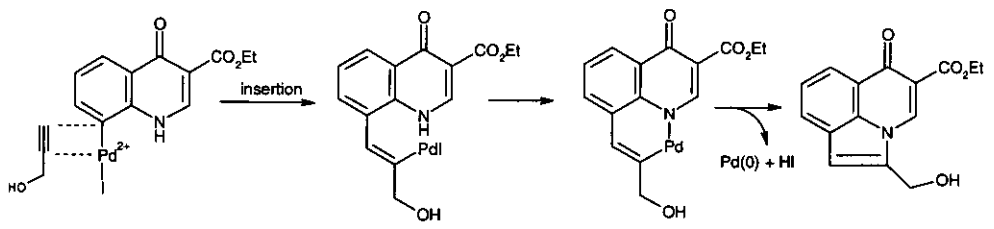
The palladium catalysed coupling of iodoarenes with terminal and internal acetylenes to give arylated acetylenes is a well known process which Larock *et al.*[10] have developed into an elegant synthesis of indoles. Thus, reaction of substituted *o*-iodoanilides with internal acetylenes gives good yields of functionalised indoles under mild conditions. We report herein the extension of this methodology to the synthesis of the pyrrolo[3,2,1-*ij*]quinoline system and demonstrate that a quinoline nitrogen is able to function effectively as a nucleophile in the formation of the indole ring. Formally, the reaction constitutes the synthesis of the pyrrolo[3,2,1-*ij*]quinoline system by pathway B above.

We chose to investigate the reaction of various 4-substituted 8-iodoquinoline derivatives since these, in principle, provided scope for further synthetic manipulation following ring formation. The substituted 8-iodoquinolines (**2**-**6**) were prepared in good overall yields from *o*-iodoaniline by appropriate modification of the literature procedure[11] and were subjected to palladium catalysed coupling with propargyl alcohol as a prototypical acetylene. Upon treatment with propargyl alcohol, iodo(phenyl)bis(triphenylphosphine) palladium,[12] copper(I)iodide, and triethylamine in toluene solution at room temperature, the iodoquinolines were smoothly converted into the corresponding 6-substituted pyrrolo[3,2,1-*ij*]quinolines (Table 1). The structure of the products was assigned on the basis of microanalytical and spectroscopic data and the regiochemistry of cyclisation confirmed by a positive ^{1}H-^{1}H nOe difference between H4 and the methylene protons at C2 in compound (**11**). At rt, the ^{1}H NMR spectra (in DMSO-d_6) of the 6-arylimino derivatives (**8**) and (**11**) showed evidence of the presence of *E* and *Z* imino isomers although these were not separable by chromatography. Additionally, the ratio of *E*:*Z* isomers varied in each case but upon warming the solutions to 363 K, only single isomers were observed suggesting that interconversion between the isomers was relatively rapid at this temperature. Interestingly the NMR spectrum of the 3-unsubstituted derivative (**9**) did not show evidence of isomers strongly implying that the steric bulk of the 3-substituent is necessary for imino geometrical isomers to be observed.

A noteworthy feature of the reaction is the facile conversion of the 4-quinolone derivative (**2**) and the 4-chloroquinoline analog (**5**), both of which gave derivatives of the same 6-oxopyrroloquinoline

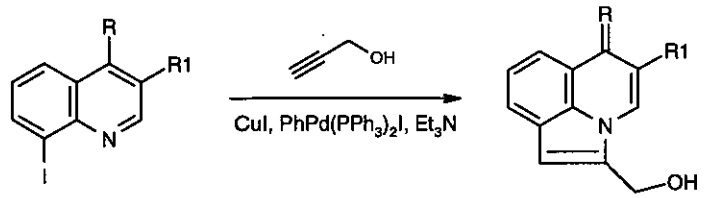
system. The possibility that prior hydrolysis of **5** occurred to generate the corresponding quinolone which subsequently underwent reaction cannot be precluded although precautions to exclude water were followed. This may also, in part, account for the lower yield observed in the reaction of **5**. The quinoline 3-substituent appeared to have little effect on the course of the reaction although clearly, only a limited range of compounds have been examined. However, the yields of cyclised product were generally higher for 3-keto and ester substituents compared with the 3-unsubstituted system (eg. **3**→**8** and **4**→**9**). Although the 4-anilino derivatives readily underwent cyclisation, the corresponding 4-methylamino analog failed to react cleanly and only starting quinoline was recovered in low yield from an intractable mixture of polar products. Presumably, the basicity of the quinoline nitrogen is important for the cyclisation to proceed smoothly with weakly basic quinolines appearing to undergo cyclisation more easily. It is well known that electron withdrawing substituents on the quinoline nucleus reduce quinoline basicity; whereas, electron donating substituents, particularly at the quinoline 4-position, can dramatically increase basicity.[13] Alternatively, it is conceivable that a 3-carbonyl substituent could facilitate proton removal from a 4-NH or 4-OH quinoline tautomer. In an attempt to further define the scope of the reaction and with a view towards formulating a general synthesis of indoles, the behaviour of *o*-iodoaniline was examined. Under the standard reaction conditions, however, cyclisation failed to occur and the major product isolated was unreacted aniline together with small amounts of the corresponding *o*-iodoacetylene. As expected,[12] *o*-iodonitrobenzene reacted smoothly to give 3-(2-iodophenyl)-2-propyn-1-ol.

Although the exact mechanism of the reaction has not been investigated, a plausible pathway is shown in Scheme 2. Oxidative addition of the aryl iodide to Pd(0) generates an arylpalladium species which undergoes acetylene insertion followed by intramolecular nucleophilic attack by quinoline nitrogen and finally reductive elimination to regenerate the catalytically active palladium species.



Scheme 2

Catalytic hydrogenation of the 6-arylimino derivative (**11**) over Lindlar's catalyst led to reduction of the imine and subsequent double bond migration gave the substituted 4*H*-pyrrolo[3,2,1-*ij*]quinoline (**12**) as the sole product in 72% yield; reduction of the indolic system was not observed (Scheme 3).



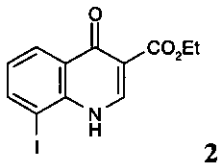
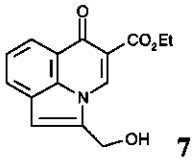
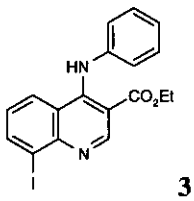
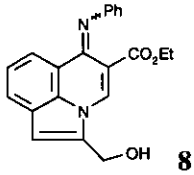
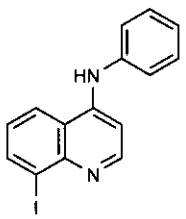
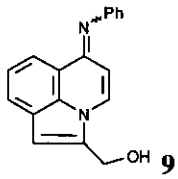
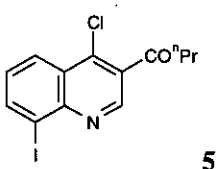
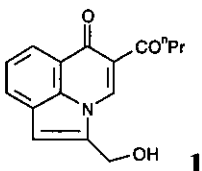
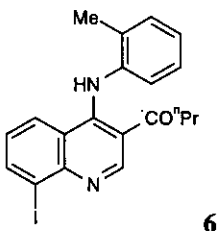
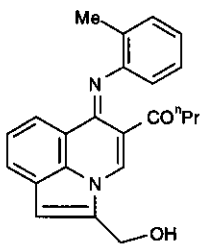
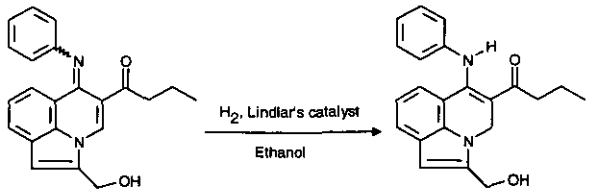
Compound	Product	% Isolated yield
 **2**	 **7**	54
 **3**	 **8**	50
 **4**	 **9**	17
 **5**	 **10**	8
 **6**	 **11**	68

Table 1. Reaction of 8-iodoquinolines to give substituted pyrrolo[3,2,1-*ij*]quinolines



11 **12**

Scheme 3

Conclusion

The pyrrolo[3,2,1-*ij*]quinoline nucleus can be generated readily, in one step, *via* palladium catalysed intramolecular coupling of various 8-iodoquinoline derivatives. Although the yields of product obtained are modest, the reaction conditions are mild and give easy access to a highly functionalised tricyclic heterocycle. The reaction pathway is proposed to involve nucleophilic attack of a quinoline nitrogen on a Pd(II) species followed by reductive elimination to regenerate catalytically active Pd(0). The catalytic hydrogenation of 2-hydroxymethyl-6-phenyliminopyrrolo[3,2,1-*ij*]quinoline has been shown to give 2-hydroxymethyl-6-phenylamino-*4H*-pyrrolo[3,2,1-*ij*]quinoline, presumably by reduction of the imine and subsequent double bond migration.

EXPERIMENTAL SECTION

Melting points were determined on a Buchi 510 melting point apparatus and are uncorrected. All analytical data were obtained by the Analytical Sciences Department at SmithKline Beecham. NMR spectra were obtained for all compounds as $CDCl_3$ or DMSO-d_6 solutions on a Bruker AM250 spectrometer and chemical shifts are quoted in parts per million (δ) relative to tetramethylsilane. MS spectral determinations were carried out on a VG analytical 7070F mass spectrometer. Analytical and preparative chromatography was carried out on Merck Kieselgel 60 grade silica gel. All starting materials were obtained from commercial sources and were used as received unless otherwise stated.

Ethyl 4-hydroxy-8-iodoquinoline-3-carboxylate (2)

A solution of *o*-iodoaniline (14.4 g, 66 mmol) in chloroform (70 mL) was treated with diethylethoxymethylene malonate (14.2 g, 67 mmol) and the resulting mixture heated under reflux for 16 h. The reaction mixture was evaporated to dryness and the residue recrystallised from ethanol to a white solid (20.0 g, 78%), mp 110-111°C. The solid was added, in portions, to boiling diphenyl ether (200 mL) and refluxing continued for a further 10 min. After cooling to rt, addition of excess petroleum ether precipitated the product (16 g, 100%), mp 230-231°C. Anal. Calcd for $C_{12}H_{10}NO_3I$: C, 42.01; H, 2.94; N, 4.08. Found: C, 42.19; H, 3.01; N, 4.06. ^{1}H NMR (DMSO-d_6): δ 1.28 (t, J = 7.1 Hz, 3H, $CO_2CH_2\mathit{CH_3}$), 4.23 (q, J = 7.1 Hz, 2H, $CO_2\mathit{CH_2}CH_3$), 7.21 (m, 1H, *H-6*), 8.17, 8.20 (each

dd, *J* = 1.4, 8.0 Hz, 2H, *5,H-7*), 8.50 (1H, s, *H-2*); 11.22 (br s, 1H, *NH*). MS m/z 343 (M+), 297 (100), 170, 142, 114. IR: ν 1710, 1528 cm^{-1}.

Ethyl 4-chloro-8-iodo-3-quinolinecarboxylate

Ethyl 4-hydroxy-8-iodoquinoline-3-carboxylate (10 g, 29 mmol) was refluxed in $POCl_3$ (40 mL, 0.42 mol) for 25 min and the cooled solution poured into ice/water. The resulting mixture was neutralised with concentrated NH_4OH, the resulting solid isolated, washed with water and dried (10 g, 95%). Recrystallisation from ethanol/water gave analytically pure material, mp 81-82°C. Anal. Calcd for $C_{12}H_9NO_2ClI$: C, 39.86; H, 2.51; N, 3.87. Found: C, 39.54; H, 2.58; N, 3.87. ^1H NMR (DMSO-d_6): δ 1.39 (t, *J* = 7.1 Hz, 3H, $CO_2CH_2CH_3$), 4.45 (q, *J* = 7.1 Hz, 2H, $CO_2CH_2CH_3$), 7.60 (*m*, 1H, *H-6*), 8.41, 8.60 (each dd, *J* = 1.2, 8.5 Hz, 2H, *5,H-7*), 9.22 (s, 1H, *H-2*). MS m/z 363, 361 (M^+, 100), 333, 316, 288. IR: ν 1728 cm^{-1}.

Ethyl 4-(phenylamino)-8-iodo-3-quinolinecarboxylate (3)

Ethyl 4-chloro-8-iodo-3-quinolinecarboxylate (4.0 g, 11 mmol) and freshly distilled aniline (2 g, 22 mmol) were heated at 140°C for 2 h and cooled to rt. The resulting solid was partitioned between CH_2Cl_2 and dilute HCl. The aqueous layer was separated, washed with CH_2Cl_2, basicified with Na_2CO_3 and extracted with CH_2Cl_2. The extracts were dried ($MgSO_4$) and evaporated to a yellow solid (2.3 g, 49%). Recrystallisation from MeOH/EtOAc gave a colourless solid, mp 137-139°C. Anal. Calcd for $C_{18}H_{15}N_2O_2I$: C, 51.69; H, 3.62; N, 6.60. Found: C, 51.50; H, 3.68; N, 6.69. ^1H NMR (DMSO-d_6): δ 1.16 (t, *J* = 7.1 Hz, 3H, $CO_2CH_2CH_3$); 4.00 (q, *J* = 7.1 Hz, 2H, $CO_2CH_2CH_3$); 7.07 (m, 3H, *Ph-H*); 7.27 (m, 1H, *H-6*); 7.30 (m, 2H, *Ph-H*); 8.08 (d, *J* = 7.9 Hz, 1H, *H-7*); 8.40 (d, *J* = 7.9 Hz, 1H, *H-5*); 8.97 (s, 1H, *H-2*); 9.28 (s, 1H, *NH*). MS m/z 418 (M^+), 372 (100), 245. IR: ν 3244, 1670 cm^{-1}.

4-Phenylamino-8-iodoquinoline (4)

Prepared from 4-chloro-8-iodoquinoline[14] using the above procedure (61%), mp 231-232°C (from EtOAc). Anal. Calcd for $C_{15}H_{11}N_2I.0.5H_2O$: C, 50.72; H, 3.40; N, 7.89. Found: C, 50.92; H, 3.01; N, 8.08. ^1H NMR (DMSO-d_6): δ 6.97 (d, *J* = 5.3 Hz, 1H, *H-3*); 7.18 (m, 1H, *Ph-4-H*); 7.29 (m, 1H, *H-6*); 7.46 - 7.36 (m, 4H, *Ph-2,3,5,H-6*); 8.33 (d, *J* = 7.3 Hz, 1H, *H-5*); 8.44 (d, *J* = 8.5 Hz, 1H, *H-7*); 8.50 (d, *J* = 5.3 Hz, 1H, *H-2*); 9.09 (s, 1H, *NH*). MS m/z 346 (M^+, 100), 218. IR: ν 3228, 3177 cm^{-1}.

3-(2-Oxobutyl)-4-chloro-8-iodoquinoline (5)

Prepared from *o*-iodoaniline and ethyl 2-(1-oxobutyl)-3-ethoxyacrylate using the literature[11] procedure (64%) and obtained as an oil which was used without purification. ^1H NMR ($CDCl_3$) 1.03 (t, *J* = 8.0

Hz, 3H, CH_3CH_2); 1.79 (m, 2H, $CH_3CH_2CH_2$); 3.04 (t, J = 7.5 Hz, 2H, $COCH_2CH_2$); 7.46 (t, J = 7.5 Hz, 1H, *H-6*); 8.43 (d, J = 7.6 Hz, 1H, *H-7*); 8.47 (d, J = 7.4 Hz, *H-5*); 9.05 (s, 1H, *H-2*).

3-(2-Oxobutyl)-4-[(2-methylphenyl)amino]-8-iodoquinoline (6)

Prepared from 3-(2-oxobutyl)-4-chloro-8-iodoquinoline and *o*-anisidine using the procedure described above (84%), mp 142-144°C. Anal. Calcd for $C_{20}H_{19}N_2OI$: C, 55.83; H, 4.45; N, 6.51. Found: C, 55.62; H, 4.51; N, 6.31. ^{1}H NMR ($CDCl_3$): δ 1.06 (t, J = 7.4 Hz, 3H, CH_3CH_2); 1.82 (m, 2H, $CH_3CH_2CH_2$); 2.35 (s, 3H, *Ph-2-CH3*); 3.04 (t, J = 7.5 Hz, 2H, $COCH_2CH_2$); 6.77 (t, J = 7.5 Hz, 1H, *H-6*); 6.89 and 7.29 (each d, J = 7.7 Hz, 2H, *Ph-3,H-6*); 7.07-7.18 (m, 2H, *Ph-4,H-5*); 7.52 (d, J = 7.3 Hz, 1H, *H-7*); 8.23 (d, J = 7.3 Hz, *H-5*); 9.28 (s, 1H, *H-2*). MS m/z 431 (M^++H, 20), 277, 185. IR: ν 1630, 1598, 1580 cm^{-1}.

Ethyl 2-hydroxymethyl-6-phenyliminopyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (8)

A solution of ethyl 4-phenylamino-8-iodo-3-quinolinecarboxylate (1.0 g, 2 mmol) in toluene (10 mL) was treated at rt with triethylamine (0.5 g, 5 mmol) and propargyl alcohol (0.2 g, 3 mmol). The resulting mixture was placed under an argon atmosphere and copper(I) iodide (5 mg) added together with iodo(phenyl)bis(triphenylphosphine)palladium (20 mg). After 48 h, dichloromethane (90 mL) was added and the solution washed with water, dried ($MgSO_4$) and evaporated. Purification by flash chromatography using 9:1 EtOAc:petroleum ether gave an oil which was recrystallised from ethanol (0.39 g, 50%), mp 91°C. Anal. Calcd for $C_{21}H_{18}N_2O_3.0.5H_2O$: C, 70.97; H, 5.39; N, 7.88. Found: C, 71.36; H, 5.09; N, 8.08. ^{1}H NMR (DMSO-d_6): δ 0.95 (t, J = 6.80 Hz, 3H, $CO_2CH_2CH_3$); 3.38 (q, J = 7.20 Hz, 2H, $CO_2CH_2CH_3$); 4.82 (d, J = 4.80 Hz, 2H, CH_2OH); 5.64 (t, J = 5.20 Hz, 1H, *OH*); 6.80 (s, 1H, *H-1*); 6.81 (d, J = 7.20 Hz, 2H, *Ph-2,H-6*); 7.00 (t, J = 7.2 Hz, 1H, *Ph-4-H*); 7.28 (m, 2H, *Ph-3,H-5*); 7.45 (t, J = 8.0 Hz, 1H, *H-8*); 7.77 (d, J = 7.2 Hz, 1H, *H-9*); 8.03 (d, J = 7.60 Hz, 1H, *H-7*); 8.34 (s, 1H, *H-4*). MS m/z 346 (M^+, 50), 301, 273 (100), 256. IR: ν 1712, 1644 cm^{-1}.

The following compounds were prepared in an analogous manner:

2-Hydroxymethyl-6-phenyliminopyrrolo[3,2,1-*ij*]quinoline (9)

4-Phenylamino-8-iodoquinoline and propargyl alcohol were converted into the title compound in 17% yield. mp 58°C. Anal. Calcd for $C_{18}H_{14}N_2O.0.5H_2O$: C, 76.30; H, 5.37; N, 9.89. Found: C, 76.06; H, 5.59; N, 9.11. ^{1}H NMR ($CDCl_3$): δ 4.88 (s, 2H, CH_2OH); 6.16 (d, J = 8.4 Hz, 1H, *H-5*); 6.56 (s, 1H, *H-1*); 6.96 (d, J = 9.6 Hz, 2H, *Ph-2,H-6*); 7.10 (m, 1H, *Ph-4-H*), 7.38 (m, 3H, *Ph-3,H-5, H-8*); 7.42 (d, J = 8.3 Hz, 1H, *H-4*); 7.59 (d, J = 8.0 Hz, 1H, *H-9*); 7.68 (d, J = 8.0 Hz, 1H, *H-7*). MS m/z 274 (M^+, 100), 257, 243. IR: ν 1644 cm^{-1}.

Ethyl 2-hydroxymethyl-6-oxopyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (7)

Ethyl 4-hydroxy-8-iodo-3-quinolinecarboxylate and propargyl alcohol were converted into the title compound in 54% yield, mp 167-168°C. Anal. Calcd for $C_{15}H_{13}NO_4$: C, 66.41; H, 4.83; N, 5.16. Found: C, 66.97; H, 4.94; N, 5.18. ^{1}H NMR ($CDCl_3$): δ 1.42 (t, *J* = 6.80 Hz, 3H, $CO_2CH_2CH_3$); 2.87 (t, *J* = 6.0 Hz, 1H, *OH*); 4.40 (q, *J* = 6.80 Hz, 2H, $CO_2CH_2CH_3$); 5.01 (d, *J* = 5.60 Hz, 2H, CH_2OH); 6.79 (s, 1H, *H-1*); 7.43 (t, *J* = 7.60 Hz, 1H, *H-8*); 7.77 (d, *J* = 7.20 Hz, 1H, *H-9*); 8.06 (d, *J* = 8 Hz, 1H, *H-7*); 8.95 (s, 1H, *H-4*). MS m/z 271 (M^+, 30), 226 (30), 199 (100), 182. IR: ν 3353, 1707, 1647 cm^{-1}.

2-Hydroxymethyl-5-(2-oxobutyl)-6-oxopyrrolo[3,2,1-*ij*]quinoline (10)

3-(2-Oxobutyl)-4-chloro-8-iodoquinoline and propargyl alcohol were converted into the title compound in 8% yield, mp 141°C. Anal. Calcd for $C_{16}H_{15}NO_3.0.25H_2O$: C,70.19; H,5.71; N,5.12. Found: C,70.41; H,5.71; N,5.18. ^{1}H NMR (DMSO-d_6): δ 0.94 (t, *J* = 7.39 Hz, 3H, CH_3CH_2); 1.61 (m, 2H, $CH_3CH_2CH_2$); 3.10 (t, *J* = 7.20 Hz, 2H, $COCH_2CH_2$); 4.89 (s, 2H, CH_2OH); 5.77 (br, 1H, *OH*); 6.94 (s, 1H, *H-1*); 7.60 (t, *J* = 7.65 Hz, 1H, *H-9*); 8.04 (m, 2H, *H-7,8*); 8.86 (s, 1H, *H-4*); MS m/z 270 (M^++H, 100), 254, 235, 185, 93. IR: ν 3298, 1677, 1640 cm^{-1}.

2-Hydroxymethyl-5-(2-oxobutyl)-6-(2-methylphenylimino)pyrrolo[3,2,1-*ij*]quinoline (11)

3-(2-Oxobutyl)-4-[2-methylphenylamino-8-iodoquinoline and propargyl alcohol were converted into the title compound in 68% yield, mp 85°C. Anal. Calcd for $C_{23}H_{22}N_2O_2$: C, 77.07; H, 6.19; N, 7.82. Found: C, 76.74; H, 6.21; N, 7.81. ^{1}H NMR (DMSO-d_6, 363 K): δ 0.88 (t, *J* = 7.3 Hz, 3H, $CO_2CH_2CH_3$); 1.57 (br, 2H, $CH_2CH_2CH_3$); 2.08 (s, 3H, *Ph-2-CH*$_3$); 2.93 (br, 2H, $COCH_2CH_2$); 4.82 (d, *J* = 5.40 Hz, 2H, CH_2OH); 5.35 (t, *J* = 5.50 Hz, 1H, CH_2OH); 6.65 (d, *J* = 7.79 Hz, 1H, *H-8*); 6.73 (s, 1H, *H-1*); 7.25 - 6.96 (m, 5H, *Ph-3,4,5,H-6, H-7*); 7.69 (d, *J* = 7.85 Hz, 1H, *H-9*); 8.23 (s, 1H, *H-4*). MS m/z 359 (M^+), 185, 93. IR: ν 1672, 1629, 1557 cm^{-1}. Irradiation of the methylene resonance at δ 4.82 gave an enhancement of the singlet resonanes at δ 6.73 (*H-1*) and δ 8.23 (*H-4*). Irradiation of the *H-1* resonance gave enhancement of the resonances at δ 4.82 (CH_2OH) and δ 7.85 (*H-9*). Irradiation of the *H-4* resonance at δ 8.23 gave enhancement of the resonance at δ 4.82 (CH_2OH).

2-Hydroxymethyl-5-(2-oxobutyl)-6-(2-methylphenylamino)-4*H*-pyrrolo[3,2,1-*ij*]quinoline (12)

A solution of 2-hydroxymethyl-5-(2-oxobutyl)-6-(2-methylphenylimino)pyrrolo[3,2,1-*ij*]quinoline (1.0 g, 2.8 mmol) in ethanol (20 mL) was treated with Lindlar's catalyst (50 mg) under an inert atmosphere. The mixture was hydrogenated at 50 psi until hydrogen uptake ceased. The catalyst was

removed by filtration and the filtrate evaporated to dryness to give the product as an orange solid (0.73 g, 72%) which was recrystallised from ethanol, mp 161-162°C. Anal. Calcd for $C_{23}H_{24}N_2O_2$: C, 76.64; H, 6.71; N, 7.77. Found: C, 76.61; H, 6.74; N, 7.62. ^{1}H NMR ($CDCl_3$): δ 1.04 (t, *J* = 7.38 Hz, 3H, $CO_2CH_2CH_3$); 1.81- 1.69 (m, 3H, $CH_2CH_2CH_3$, CH_2OH); 2.35 (s, 3H, *Ph-2-CH3*); 2.53 (t, *J* = 7.29 Hz, 2H, $COCH_2CH_2$); 4.85 (d, *J* = 4.77 Hz, 2H, CH_2OH); 5.32 (br, 2H, *CH2-4*); 6.44 (s, 1H, *H-1*); 6.57 - 6.66 (m, 2H, *H-7,8*); 7.00 - 7.15 (m, 3H, *Ph-4,5,H-6*); 7.24 (d, *J* = 7.43 Hz, *Ph-3-H*); 7.43 (d, *J* = 7.59 Hz, 1H, *H-9*). MS m/z 361 (M^+), 185, 93. IR: ν 3413, 1628, 1544 cm^{-1}.

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