Reaction of 2-Benzoyl-1,2-dihydroisoquinoline-1-carbonitrile Tetrafluoroborate with (*Z*)-2-Arylidene-3(2*H*)-benzofuranones – Access to Chromenopyrrole Derivatives

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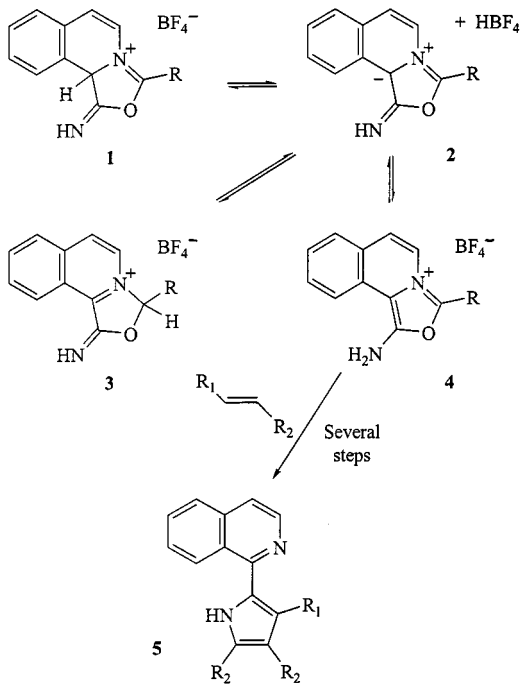
Spiro[pyrrole-3,2′-3(2*H*)-benzofuranones] **7** have been synthesized by [4+2] cycloaddition of 2-arylidene-3(2*H*)-benzofuranones with the 2-benzoyl-1,2-dihydroisoquinoline-1-carbonitrile tetrafluoroborates. In acidic medium or in refluxing DMF, the spiro compounds yield tetrasubstituted pyrroles or compounds derived from chromenopyrroles. The regio- and stereochemistry of the reaction was established by spectroscopic or X-ray analysis.

Introduction

Previous studies[1][2] have established that solutions of tetrafluoroborate salts of 2-acyl-1,2-dihydroisoquinoline-1-carbonitriles (Reissert compounds) exist as equilibrium mixtures of **1**−**4**, with **4** being the major component (Scheme 1). According to McEwen[3], the acid-catalysed condensation−rearrangement of these salts with an alkene afford substituted 2-(1-isoquinolinyl)pyrroles **5** (Scheme 1).

Scheme 1



The mechanism suggested by McEwen has been proved by other teams by means of the isolation of reaction intermediates[4][5][6].

Recently[7] we reported the preparation of 4-aryl-4,5-dihydro-5-hydroxy-2-(1-isoquinolinyl)-5-phenylspiro[3*H*-pyrrole-3,3′-(3′*H*)-benzofuran-2′-one] and 4-aryl-3-(*o*-hydroxyphenyl)-2-(1-isoquinolinyl)-5-phenylpyrrole by the reaction of the tetrafluoroborate salt **4** (Reissert compound) with (*Z*)- and (*E*)-3-arylidene-2(3*H*)benzofuranones. These cycloadducts are further converted in the reaction medium to yield a single stable product, regardless of the stereochemistry, (*Z*) or (*E*), of the dienophile used.

We report here the dipolarophilic activity of (*Z*)-2-arylidene-3(2*H*)-benzofuranones **6** with the same heterodiene **4** (Scheme 2), in order to determine the stereochemistry of the approach, and the conversion of the cycloadducts.

Results and Discussion

The dipolarophiles **6** react with tetrafluoroborate salts **1**−**4** (R = Ph) in DMF at 60°C for 12 h to yield mixtures of three compounds (Scheme 2) identified as: 4-aryl-4,5-dihydro-5-hydroxy-2-(1-isoquinolinyl)-5-phenylspiro[3*H*-pyrrole-3,2′-(2*H*)-benzofuran]-3′-one (**7**), 4-aryl-3-(*o*-carboxyphenoxy)-2-(1-isoquinolinyl)-5-phenylpyrrole (**8**) and 3-aryl-4-(1,2-dihydro-1-oxo-2-isoquinolinyl)2-phenylchromeno[2,3-*b*]pyrrole (**9**).

Analytical and spectroscopic data (IR, ^{1}H and ^{13}C NMR) are in agreement with the assigned structures **7** and **8**. Compounds **9**, which are minor products (3−5%), were unexpected as indicated by previous results. The radiocrystallographic study of **9b** enabled us to determine the structure of this compound.

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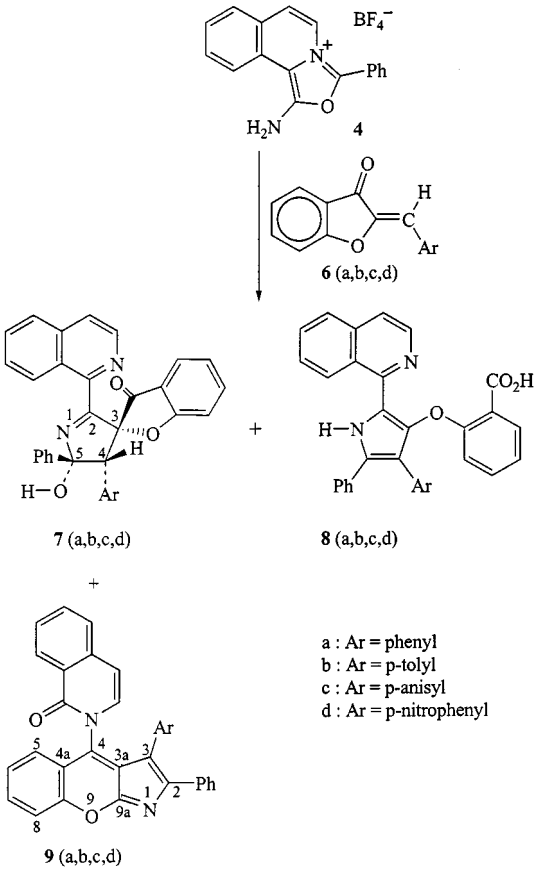
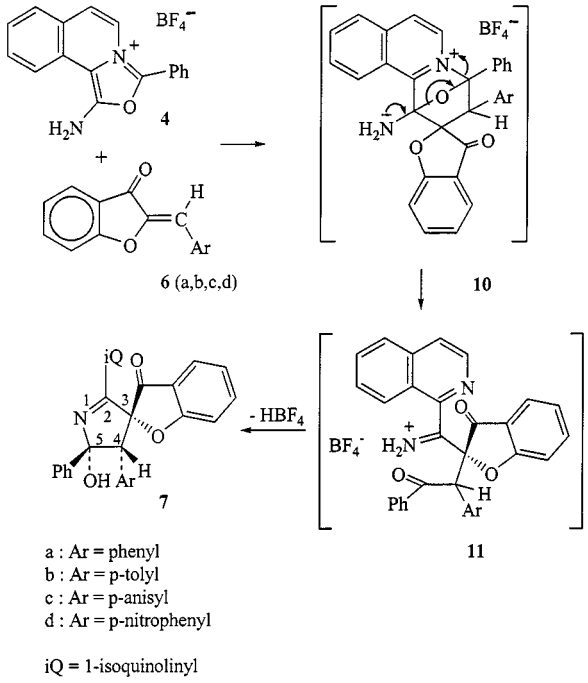
Scheme 2



Scheme 3



The reaction occurs according to the mechanism proposed by McEwen et al[3] (Scheme 3), starting with a Diels-Alder cycloaddition involving the heterodiene moiety of **4**, to give the cycloadduct **10**.

The latter is subsequently converted into compound **7**, through the intermediate **11**. The regiochemistry of the reaction is similar to that observed[3][7][8] for an olefin activated by an electron-withdrawing group, which is always situated at the 3-position of the resulting pyrrole.

The regiochemistry was confirmed by ^{1}H NMR data. The observation of long-range coupling (^4J = 0.8−1.4 Hz) between 4-H and the hydroxyl proton suggests a *trans*-pseudodiaxial conformation for OH and 4-H, in a ″W″ configuration[9].

In fact, for the opposite regiochemistry, the product of the conversion of the initial cycloadduct would be a 4,5-dihydropyrrole, whose NH group would have easily been detected in the IR and ^{1}H NMR spectra.

The stereochemistry of compounds **7** is in good agreement with the thermodynamically more stable *trans* arrangement for the two 4- and 5-aryl groups. Since the reaction sequence begins with a Diels−Alder cycloaddition, the relative stereochemistry about the C^3-C^4 bond is the same as that of the initial olefin **6**.

It is possible that compounds **8** and **9**, which are produced concomitantly with the spiro-derivatives **7**, are actually derived from the latter.

In order to verify this hypothesis, we first refluxed compounds **7** in an AcOH/HCl (10:1) mixture. To allow for comparison, the experiment was repeated, heating the same compounds in DMF.

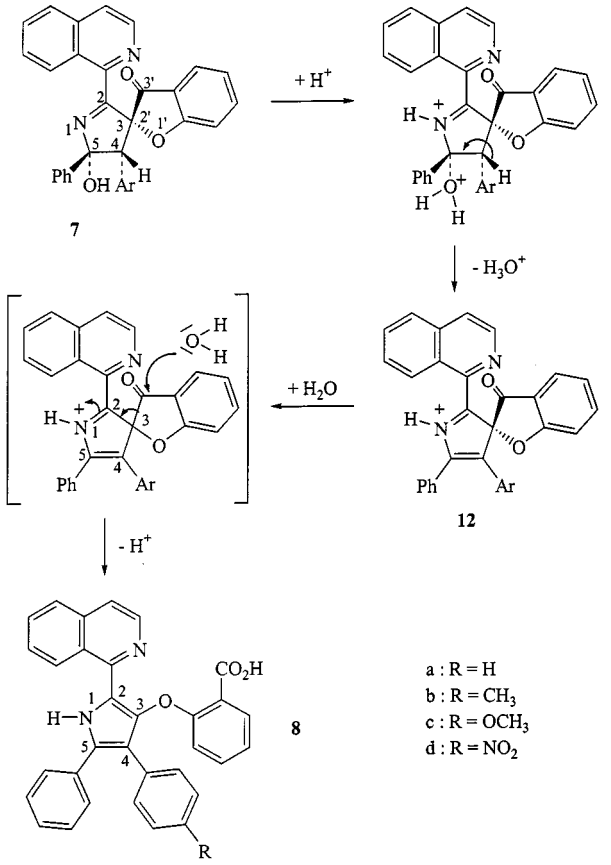
In both cases, physical characteristics, IR and NMR spectroscopic data of the products obtained were identical to those of compounds **8** and **9** obtained from the initial reaction medium.

Acid-promoted dehydration is followed by the introduction of a water molecule to the cycle of the remaining 3(2*H*)-benzofuranone (Scheme 4). This leads to a heteroaromatic pyrrole derivative **8**, which is sterically less hindered than the initial spiro-compound **7**. We have observed a similar conversion during the condensation of the same tetrafluoroborate salt with (*Z*)- and (*E*)-3-arylidene-3*H*-benzofuran-2-one[7]. We should mention here the studies of D. Adnani et al.[8], who observed a similar process with the products obtained in the condensation of an arylidenetetralone with tetrafluoroborate **4**.

The structure of minor products **9** required a radiocrystallographic study, which showed an original tricyclic chromenopyrrole skeleton (Figure 1).

The phenyl and *p*-tolyl substituents of the pyrrole nucleus are linked to two neighbouring carbon atoms, as in compound **7**. The oxygen atom of the initial benzofuran ring is now bonded to the carbon atom of the C=N bond; the latter originally bore the 1-isoquinolinyl group in product **7**. The structure of **9** suggests that the last step of the rearrangement (Scheme 5) can be related to the Chapman transposition[10][11], in which the imidate ″i″ is transformed into an amide upon heating.

Scheme 4



Scheme 5

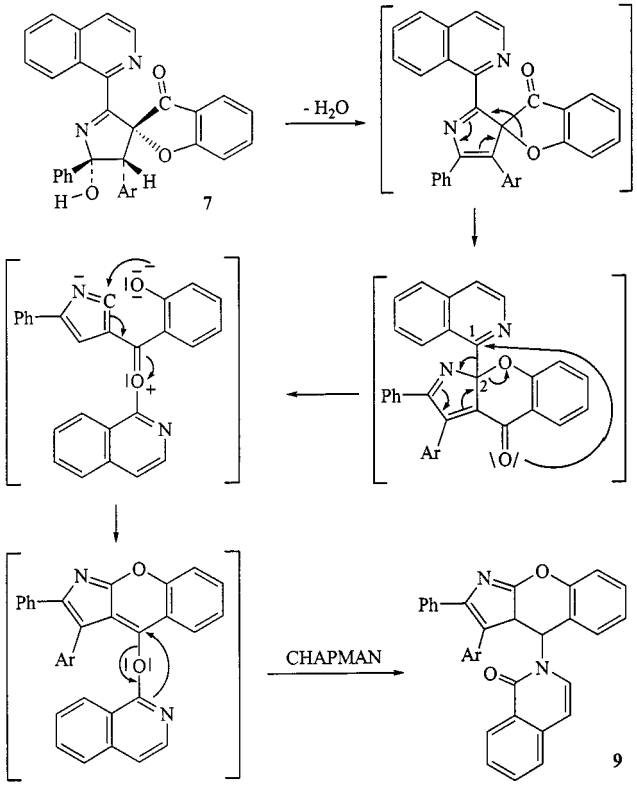
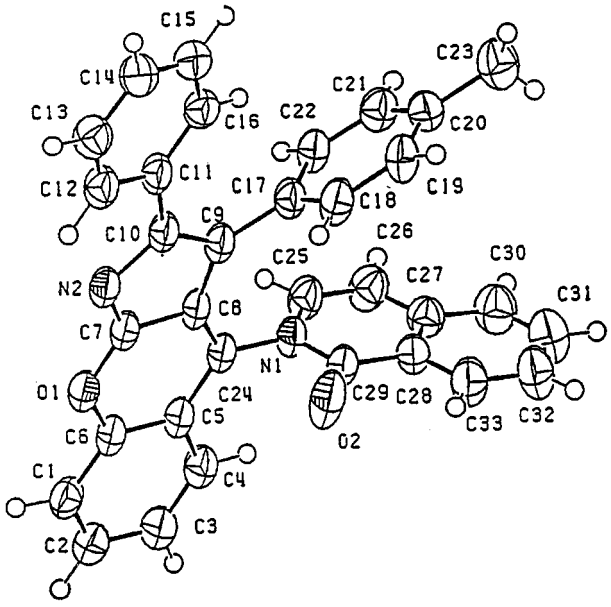


Figure 1. Molecular structure of compound **9b** in the crystal; the thermal ellipsoids are drawn at the 50% probability level[16]



Conclusion

The reaction of 2-benzoyl-1,2-dihydroisoquinoline-1-carbonitrile tetrafluoroborate with (*Z*)-2-arylidene-3(2*H*)-benzofuranones is regio- and diastereospecific. The spiro-compound obtained from the initially formed cycloadduct undergoes a further rearrangement in acidic medium to yield two stable aromatic heterocycles: a tetrasubstituted pyrrole as the major product and an unexpected chromeno-pyrrole derivative, as the minor product.

Experimental Section

Melting points were determined with a Kofler apparatus, and are uncorrected. IR spectra (KBr) were recorded on a Bio-Rad FTS-7 spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker-Spectrospin AC 200 spectrometer operating at 200 MHz for ^{1}H and at 50 MHz for ^{13}C NMR spectra. Chemical shifts were measured relative to TMS in $CDCl_3$ or [D_6]DMSO as solvent. Analytical data were obtained by the CNRS Vernaison (France) and were satisfactory (C, H, N ± 0.30% from theoretical).

2-Benzoyl-1,2-dihydroisoquinoline-1-carbonitrile tetrafluoroborate was prepared according to ref.[3]. The starting olefins **6** were synthesized by condensation of benzaldehyde[12] or its substituted derivatives with 3(2*H*)-benzofuranone in acidic solution according to Pelter et al[13]. Only (*Z*) stereoisomers were isolated[14][15].

(Z)-2-Benzylidene-3(2H)-benzofuranone (**6a**): IR (KBr): $\tilde{\nu}$ = 3060 cm^{-1}, 1710, 1600, 1510. – ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 6.85 (s, 1 H, ethylenic CH), 7.05–7.95 (m, 9 aromatic H). – ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 113.0 (C ethylenic), 121.7–166.3 (C aromatic), 184.9 (C=O).

(Z)-2-(4-Methylbenzylidene)-3(2H)-benzofuranone (**6b**): IR (KBr): $\tilde{\nu}$ = 3060 cm^{-1}, 2960, 1715, 1600, 1500. – ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.18 (s, 3 H, CH_3), 6.85 (s, 1 H, CH ethylenic), 7.05–7.85 (m, 8 aromatic H). – ^{13}C NMR ($CDCl_3$, 50

MHz): δ = 20.5 (CH_3), 113.8 (C ethylenic), 121.9−166.2 (C aromatic), 185.0 (C=O).

(Z)-2-(4-Methoxybenzylidene)-3(2H)-benzofuranone (**6c**): IR (KBr): $\tilde{\nu}$ = 3060 cm^{-1}, 2960, 1705, 1600, 1485. − ^1H NMR ($CDCl_3$, 200 MHz): δ = 3.52 (s, 3 H, OCH_3), 7.10−7.95 (m, 8 aromatic H). − ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 55.4 (OCH_3), 113.0 (C ethylenic), 122.2−166.0 (C aromatic), 184.7 (C=O).

(Z)-2-(4-Nitrobenzylidene)-3(2H)-benzofuranone (**6d**): IR (KBr): $\tilde{\nu}$ = 3060 cm^{-1}, 1720, 1600, 1520, 1490, 1350. − ^1H NMR ($CDCl_3$, 200 MHz): δ = 6.85 (s, 1 H, CH ethylenic), 7.10−8.10 (m, 8 aromatic H). − ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 108.2 (C ethylenic), 120.3−165.8 (C aromatic), 183.3 (C=O).

Reaction of 2-Benzoyl-1,2-dihydroisoquinoline-1-carbonitrile Tetrafluoroborate **4** *with Enone* **6**. − *General Procedure:* To a solution of enone **6** (1 g) in DMF (40 ml), was added slowly, with stirring, tetrafluoroborate **4**.(1.3 g, 3.74 mmol). The mixture was warmed to 60°C for 12 h. After cooling, the mixture was taken up in diethyl ether (50 ml) and washed with water (4 × 25 ml). The resulting brick red solid was filtered. The filtrate was dried (Na_2SO_4) and evaporated in vacuo. The crude product was purified by recrystallisation from EtOH/DMF (90:10) to give a colourless solid, identified as the product **7**. The brick red solid, which contained both **8** and **9**, was purified by chromatography on silica gel with EtOAc/hexane (70:30, R_f = 0.84) to give compound **9**, followed by elution with pure EtOAc (R_f = 0.65) to afford compound **8.**

7a: Yield 30%. − IR (KBr): $\tilde{\nu}$ = 3200 cm^{-1}, 3060, 1700, 1600, 1495. − ^1H NMR ([D_6]DMSO, 200 MHz): δ = 3.42 (d, J = 1.3 Hz, 1 H, 4-H), 4.16 (d, J = 1.3 Hz, 1 H, OH), 6.72−9.89 (m, 20 aromatic H). − ^{13}C NMR ([D_6]DMSO, 50 MHz): δ = 63.7 (C-4), 97.9 (C-3), 103.4 (C-5), 112.9−172.8 (C ethylenic), 197.7 (C=O). − An analytical sample was obtained by recrystallisation from EtOH/DMF (70:30), colourless solid, m. p. 192°C. − $C_{32}H_{22}N_2O_3$ (482.5): calcd. C 79.65, H 4.60, N 5.80; found C 79.77, H 4.52, N 5.99.

7b: Yield 25%. − IR (KBr): $\tilde{\nu}$ = 3200 cm^{-1}, 3065, 2960, 1690, 1600, 1490. − ^1H NMR ([D_6]DMSO, 200 MHz): δ = 2.25 (s, 3 H, CH_3), 3.40 (d, ^4J = 0.8 Hz, 1 H, 4-H), 3.78 (d, ^4J = 0.8 Hz, 1 H, OH), 6.98−9.97 (m, 19 aromatic H). − ^{13}C NMR ([D_6]DMSO, 50 MHz): δ = 20.8 (CH_3), 63.8 (C-4), 98.3 (C-3), 103.3 (C-5), 112.9−172.8 (C ethylenic), 198.0 (C=O). − An analytical sample was obtained by recrystallisation from EtOH/DMF (70:30), colourless solid, m. p. 210°C. − $C_{33}H_{24}N_2O_3$ (496.5): calcd. C 79.82, H 4.87, N 5.64; found C 79.93, H 4.83, N 5.69.

7c: Yield 25%. − IR (KBr): $\tilde{\nu}$ = 3380 cm^{-1}, 3060, 2960, 1700, 1600, 1495. − ^1H NMR ([D_6]DMSO, 200 MHz): δ = 3.72 (s, 3 H, OCH_3), 3.40 (d, ^4J = 0.8 Hz, 1 H, 4-H), 4.15 (d, ^4J = 0.8 Hz, 1 H, OH), 6.48−9.82 (m, 19 aromatic H). − ^{13}C NMR ([D_6]DMSO, 50 MHz): δ = 54.7 (OCH_3), 61.9 (C-4), 98.5 (C-3), 103.4 (C-5), 112.6−169.6 (C ethylenic), 198.3 (C=O). − An analytical sample was obtained by recrystallisation from EtOH/DMF (70:30), colourless solid, m. p. 190°C. − $C_{33}H_{24}N_2O_4$ (512.5): calcd. C 77.33, H 4.72, N 5.46; found C 77.56, H 4.69, N 5.52.

7d: Yield 30%. − IR (KBr): $\tilde{\nu}$ = 3280 cm^{-1}, 3065, 1695, 1600, 1490. − ^1H NMR ($CDCl_3$, 200 MHz): δ = 3.50 (d, ^4J = 1.2 Hz, 1 H, 4-H), 3.94 (d, ^4J = 1.2 Hz, 1 H, OH), 6.88−9.98 (m, 19 aromatic H). − ^{13}C NMR ([D_6]DMSO, 50 MHz): δ = 63.1 (C-4), 98.1 (C-3), 103.5 (C-5), 112.9−172.6 (C ethylenic), 197.5 (C=O). − An analytical sample was obtained by recrystallisation from EtOH/DMF (70:30), colourless solid, m. p. 220°C. − $C_{32}H_{21}N_2O_5$ (527.5): calcd. C 72.86, H 4.01, N 7.96; found C 73.07, H 3.92, N 7.98.

3-(2-Carboxyphenoxy)-2-(1-isoquinolinyl)-4,5-diphenylpyrrole (**8a**): Yield 25%. − IR (KBr): $\tilde{\nu}$ = 3270−2600 cm^{-1}, 3070, 1685, 1600, 1490. − ^1H NMR ([D_6]DMSO, 200 MHz): δ = 6.68−8.67 (m, 21 H, aromatic et OH), 11.97 (s, 1 H, N−H). − ^{13}C NMR ([D_6]DMSO, 50 MHz): δ = 114.3−157.3 (C aromatic), 166.6 (C=O). − An analytical sample was obtained by recrystallisation from EtOH/DMF (70:30), yellow solid, m. p. 265°C. − $C_{32}H_{22}N_2O_3$ (482.5): calcd. C 79.65, H 4.60, N 5.80; found C79.81, H 4.56, N 5.93.

3-(2-Carboxyphenoxy)-2-(1-isoquinolinyl)-5-phenyl-4-(p-tolyl)pyrrole (**8b**): Yield 25%. − IR (KBr): $\tilde{\nu}$ = 3300−2500 cm^{-1}, 3070, 2960, 2870, 1660, 1490. − ^1H NMR ([D_6]DMSO, 200 MHz): δ = 2.26 (s, 3 H, CH_3), 6.56−8.68 (m, 19 H, aromatic and OH), 11.93 (s, 1 H, N-H). − ^{13}C NMR ([D_6]DMSO, 50 MHz): δ = 114.0−157.4 (C aromatic), 166.6 (C=O). − An analytical sample was obtained by recrystallisation from EtOH/DMF (70:30), yellow solid, m. p. 250°C. − $C_{33}H_{24}N_2O_3$ (496.5): calcd. C 79.82, H 4.87, N 5.64; found C 79.89, H 4.87, N 5.63.

4-(p-Anisyl)-3-(2-carboxyphenoxy)-2-(1-isoquinolinyl)-5-phenylpyrrole (**8c**): Yield 17%. − IR (KBr): $\tilde{\nu}$ = 3200−2800 cm^{-1}, 3060, 2960, 2870, 1660, 1490. − ^1H NMR ([D_6]DMSO, 200 MHz): δ = 3.79 (s, 3 H, OCH_3), 6.58−8.78 (m, 19 H, aromatic and OH), 11.96 (s, 1 H, N−H). − ^{13}C NMR ([D_6]DMSO, 50 MHz): δ = 54.9 (OCH_3), 113.7−157.6 (C aromatic), 166.5 (C=O). − An analytical sample was obtained by recrystallisation from EtOH/DMF (70:30), yellow solid, m. p. 260°C. − $C_{33}H_{24}N_2O_4$ (512.5): calcd. C 77.33, H 4.72, N 5.46; found C 77.61, H 4.64, N 5.59.

3-(2-Carboxyphenoxy)-2-(1-isoquinolinyl)-4-(p-nitrophenyl)-5-phenylpyrrole (**8d**): Yield 30%. − IR (KBr): $\tilde{\nu}$ = 3300−2600 cm^{-1}, 3060, 1700, 1600, 1500. − ^1H NMR ([D_6]DMSO, 200 MHz): δ = 6.51−8.72 (m, 19 H, aromatic and OH), 12.48 (s, 1 H, N−H). − ^{13}C NMR ([D_6]DMSO, 50 MHz): δ = 113.0−156.4 (C aromatic), 166.4 (C=O). − An analytical sample was obtained by recrystallisation from EtOH/DMF (70:30), yellow solid, m. p. 242°C. − $C_{32}H_{22}N_3O_5$ (527.5): calcd. C 72.86, H 4.01, N 7.96; found C 73.05, H 4.04, N 7.94.

4-[2-(1,2-Dihydro-1-oxoisoquinolinyl)]-2,3-diphenylchromeno[2,3-b]pyrrole (**9a**): Yield 5%. − IR (KBr): $\tilde{\nu}$ = 3070 cm^{-1}, 1650, 1600, 1510. − ^1H NMR ($CDCl_3$, 200 MHz): δ = 6.11 (d, J = 7.3 Hz, 1 H), 6.62 (d, J = 7.3 Hz, 1 H), 6.81−8.36 (m, 18 aromatic H). − ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 106.3−161.6 (C aromatic), 163.2 (C=O). − An analytical sample was obtained by recrystallisation from EtOH, red solid, m. p. > 300°C. − $C_{32}H_{20}N_2O_2$ (464.5): calcd. C 82.74, H 4.34, N 6.03; found C 82.79, H 4.39, N 5.87.

4-[2-(1,2-Dihydro-1-oxoisoquinolinyl)]-2-phenyl-3-(p-tolyl)chromeno[2,3-b]pyrrole (**9b**): Yield 5%. − IR (KBr): $\tilde{\nu}$ = 3065 cm^{-1}, 1670, 1600, 1495. − ^1H NMR ($CDCl_3$, 200 MHz): δ = 1.98 (s, 3 H, CH_3), 6.15 (d, J = 7.4 Hz, 1 H), 6.65 (d, J = 7.4 Hz, 1 H), 7.05−8.35 (m, 17 aromatic H). − ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 20.8 (CH_3), 106.3−153.4 (C aromatic), 159.0 (C=O). − An analytical sample was obtained by recrystallisation from EtOH, red solid, m. p. > 300°C. − $C_{33}H_{22}N_2O_2$ (478.5): calcd. C 82.83, H 4.63, N 5.85; found C 82.91, H 4.72, N 5.77.

3-(p-Anisyl)-4-[2-(1,2-dihydro-1-oxoisoquinoleinyl)]-2-phenylchromeno[2,3-b]pyrrole (**9c**): Yield 3%. − IR (KBr): $\tilde{\nu}$ = 3060 cm^{-1}, 2960, 1670, 1600, 1505. − ^1H NMR ($CDCl_3$, 200 MHz): δ = 3.42 (s, 3 H, OCH_3), 6.15 (d, J = 7.3 Hz, 1H), 6.65 (d, J = 7.3 Hz, 1 H), 7.03−8.37 (m, 17 aromatic H). − ^{13}C NMR ($CDCl_3$, 50

MHz): δ = 54.6 (OCH_3), 106.3−162.5 (C aromatic), 165.4 (C=O). − An analytical sample was obtained by recrystallisation from EtOH, red solid, m. p. > 300°C. − $C_{33}H_{22}N_2O_3$ (494.5): calcd. C 80.16, H 4.48, N 5.66; found C 80.21, H 4.53, N 5.67.

4-[2-(1,2-Dihydro-1-oxoisoquinolinyl)]-3-(p-nitrophenyl)-2-phenylchromeno[2,3-b]pyrrole (**9d**): Yield 3%. − IR (KBr): $\tilde{\nu}$ = 3065 cm^{-1}, 1650, 1600, 1500. − ^1H NMR ($CDCl_3$, 200 MHz): δ = 6.38 (d, *J* = 7.1 Hz, 1 H), 6.79 (d, *J* = 7.1 Hz, 1 H), 7.89−8.38 (m, 17 aromatic H). − ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 106.5−162.5 (C aromatic), 165.6 (C=O). − An analytical sample was obtained by recrystallisation from EtOH, red solid, m. p. > 300°C. − $C_{33}H_{19}N_3O_4$ (509.5): calcd. C 75.43, H 3.75, N 8.25; found C 75.46, H 3.79, N 8.16.

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