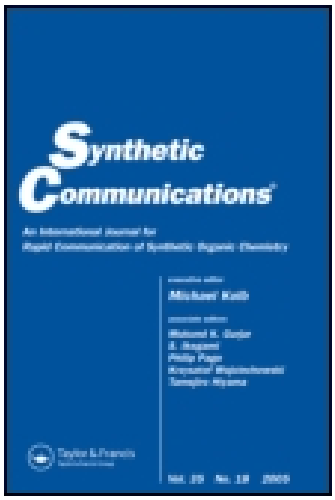

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Synthesis of Substituted Indolo[1,2- a]quinoxalines

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Synthesis of Substituted Indolo[1,2-*a*]quinoxalines

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ABSTRACT

1-(2-Nitrophenyl)indole-2-carboxylates **5,** obtained by the *N*-arylation of indole-2-carboxylates **4**, on catalytic reductive cyclization afford indolo[1,2-*a*]quinoxalino-6(5*H*)-ones **6**. These compounds on reduction with LAH in ether/THF yielded indolo[1,2-*a*]quinoxalines **7**.

Key Words: N-Arylation; LAH; Reduction; Ethyl indole-2-carboxylates; Indolo[1,2-a]quinoxalines.

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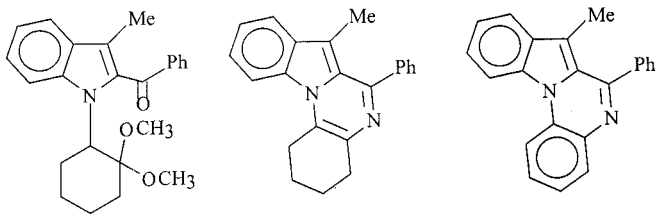
The tetracyclic ring system of indolo[1,2-*a*]quinoxalin-6(5*H*)-one is an interesting skeleton and has attracted the interests of many organic chemists. Synthesis of indolo[1,2-*a*]quinoxalin-6(5*H*)-ones and 5,6-dihydroindolo[1,2-*a*]quinoxalines with either methyl or phenyl group at 7-position were reported earlier from this laboratory.[1–4] A literature survey revealed only one report[5,6] on the synthesis of fully aromatic system which involved boiling sodio derivative of 2-aroylindole with ketal of α-bromocyclohexanone, cyclisation of resulting *N*-cyclohexyl derivative **1** by the action of ammonium acetate in boiling acetic acid to indolo[1,2-*a*]quinoxaline **2**, dehydrogenation of **2** over Raney nickel yielded the fully aromatic indolo[1,2-*a*]quinoxaline **3**.



1 2 3

The difficulty in obtaining substituted 2-chlorocyclohexanones restricts the generality of this procedure. Herein, we report the synthesis of fully aromatic 7-benzylquinoxalino[1,2-*a*]indoles **7** which makes use of easily available substituted orthonitrobenzenes.

DISCUSSION

Various ethyl 5-substituted indole-2-carboxylates **4** were prepared in 40–63% yields via Fischer indolization of appropriate phenylhydrazones using ethanolic HCl as the cyclising agent (Table 1). The phenylhydrazones were, in turn, obtained by Japp-Klingemann condensation between appropriate diazonium salt and α-phenethylacetoacetate. *N*-Arylation of carboxylates **4** was achieved via Ullmann chemistry[7,8] by refluxing appropriate indole-2-carboxylate with substituted 2-bromo/chloronitrobenzene in pyridine in presence of cupric oxide and potassium carbonate to obtain **5**. The occurrence of *N*-arylation was confirmed by the absence of stretching vibrations due to N–H in the IR spectra (Table 2). Russell et al.[9] used similar principles for the synthesis of 7-unsubstituted indolo[1,2-*a*]quinoxalin-6(5*H*)-one which takes the advantage of nucleophilic displacement of the fluorine atom in substituted

Table 1. Physical, IR, and ^{1}H-NMR, spectral data of ethyl 5-substituted 3-benzylindole-2-carboxylates.

Product	*R*	Yield[a] (%)	M.p.[b] (°C)	IR (KBr)[c] ν (cm^{-1})	^{1}H NMR[d,e] ($CDCl_3$, TMS) δ (ppm), *J* (Hz)
4a	H	46	148–149 (lit. m.p. 145–146)[14]	3320 1678	1.40 (t, 3H, $J=7.13$, $COOCH_2CH_3$), 4.44(q, 2H, $J=7.14$, $COOCH_2CH_3$), 4.55 (s, 2H, $CH_2C_6H_5$), 7.10–7.67 (m, 9H, ArH), 8.88 (br, 1H, NH).
4b	CH_3	49	178–179 (lit. m.p. 177–178)[14]	3314 1678	1.39 (t, 3H, $J=7.12$, $COOCH_2CH_3$), 2.44 (s, 3H, CH_3–Ph), 4.42 (q, 2H, $J=7.13$, $COOCH_2CH_3$), 4.51 (s, 2H, $CH_2C_6H_5$), 7.16–7.42 (m, 8H, ArH), 8.65 (br, 1H, NH).
4c	OCH_3	40	185–186	3308 1678	1.36 (t, 3H, $J=7.14$, CH_3), 3.78 (s, 3H, OCH_3), 4.38 (q, 2H, $J=7.14$, $COOCH_2CH_3$), 4.48 (s, 2H, $CH_2C_6H_5$), 6.95 (d, 1H, $J=2.35$, H-4), 6.98 (dd, 1H, $J=8.88$, $J=2.43$, H-6), 7.13–7.27 (m, 5H, ArH), 7.28 (d, 1H, $J=8.9$, H-7), 8.75 (br, 1H, NH).
4d	OC_2H_5	63	145–146	3314 1683	1.40 (t, 3H, $J=7.11$, $COOCH_2CH_3$), 1.42 (t, 3H, $J=6.95$, OCH_2CH_3), 4.02 (q, 2H, $J=6.94$, OCH_2CH_3), 4.41 (q, 2H, $J=7.11$ $COOCH_2CH_3$), 4.50 (s, 2H, $CH_2C_6H_5$), 6.99–7.31 (m, 8H, ArH), 8.81 (br, 1H, NH).

[a]The yields are not optimized.
[b]Melting points were taken in open capillaries and are uncorrected.
[c]The IR spectra were recorded on Nicolet Impact-410 FT-IR spectrophotometer.
[d]The NMR spectra were recorded at 300 MHz on Bruker Avance-300 MHz FT-NMR instrument.
[e]Satisfactory microanalyses were obtained: C, ±0.30; H, ±0.32; N, ±0.17.

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Table 2. Physical and IR spectral data of 5-substituted ethyl 1-(2-nitrophenyl)-3-benzylindole-2-carboxylates.

Product	*R*	*X*	Yield[a] (%)	M.p.[b] (°C)	IR (KBr)[c,d] ν (cm^{-1})
5a	H	H	65	125–126	1702, 1523, 1338, 1270, 1171
5b	CH_3	H	52	105–106	1709, 1524, 1345, 1283, 1171
5c	H	Cl	47	108–109	1696, 1535, 1350, 1279, 1232, 1181
5d	CH_3	Cl	48	118–119	1708, 1536, 1338, 1276, 1202, 1171
5e	OCH_3	Cl	52	130–131	1696, 1530, 1350, 1280, 1214, 1177
5f	OC_2H_5	Cl	50	92–93	1696, 1540, 1352, 1214, 1176
5g	H	OCH_3	55	102–103	1709, 1542, 1340,1276, 1227, 1172
5h	CH_3	OCH_3	66	140–141	1714, 1540, 1340, 1270, 1171
5i	OCH_3	OCH_3	58	135–136	1700, 1542, 1340, 1214, 1171
5j	OC_2H_5	OCH_3	63	98–99	1702, 1524, 1348, 1172, 1122

[a]The yields are not optimized.
[b]Melting points were taken in open capillaries and are uncorrected.
[c]The IR spectra were recorded on Nicolet Impact-410 FT-IR spectrophotometer.
[d]Satisfactory microanalyses were obtained: C, ±0.30; H, ±0.32; N, ±0.17.

2-fluoronitrobenzenes. The *N*-arylated products **5** were subjected to catalytic reductive cyclisation in DMF, instead of methanol and THF,[1–4] because of the poorer solubility of the formed quinoxalin-6-ones **6**. These were recrystallised from DMF and are described in Table 3.

The quinoxalin-6-ones when subjected to reduction with LAH in ether/THF did not give the expected reduction products **8,** instead fluorescent solids **7**, were obtained in 60–85% yields. The structures of compounds **7** were assigned by IR and ^{1}H-NMR data (Table 4). Confirmation for the structures assigned was obtained from the 2D-NMR techniques-HETCOR, ^{1}H–^{1}H-COSY, HSQC, and HMBC. The correlations thus obtained for **7d** (Table 5) are shown in Fig. 1.

Usually, reaction of lactams with LAH result in the reduction of amide (–CON<) group to amine (–CH_2–N<).[10,11] However, to our surprise, reduction of **6** with LAH in THF yielded the fully aromatic

Table 3. Physical and IR spectral data of 9-substituted 7-benzylindolo[1,2-*a*]-quinoxalin-6(5*H*)-ones.

Product	*R*	*X*	Yield[a] (%)	M.p.[b] (°C)	IR (KBr)[c,d] ν (cm^{-1})
6a	H	H	80	295–296	3181, 1665, 1617, 1502, 1451, 1409
6b	CH_3	H	82	> 300	3178, 1659, 1498, 1455, 1400
6c	H	Cl	65	> 300	3172, 1677, 1597, 1504, 1407
6d	CH_3	Cl	75	> 300	3179, 1678, 1600, 1561, 1504, 1409
6e	OCH_3	Cl	70	310–311	3168, 1657, 1602, 1493, 1450, 1403, 1377
6f	OC_2H_5	Cl	64	> 300	3184, 1666, 1616
6g	H	OCH_3	78	289–290	3165, 1665, 1528, 1505, 1453, 1413
6h	CH_3	OCH_3	75	> 300	3172, 1665, 1530, 1501, 1449, 1406
6i	OCH_3	OCH_3	65	290–291	3162, 1658,1604, 1526, 1498, 1452
6j	OC_2H_5	OCH_3	80	285–286	3166, 1677, 1535

[a]The yields are not optimized.
[b]Melting points were taken in open capillaries and are uncorrected.
[c]The IR spectra were recorded on Nicolet Impact-410 FT-IR spectrophotometer.
[d]Satisfactory microanalyses were obtained: C, ±0.30; H, ±0.32; N, ±0.17.

indolo[1,2-*a*]quinoxaline in contrast to our earlier observations.[1–4] Such an unusual reaction has been reported by de Mayo and Rigby[12] who obtained phenanthridenes by the LAH reduction of phenanthridones. Even when the lactams **6** were reduced with excess of LAH[13] (1:4), then also compounds with fully aromatic systems **7** were obtained but not the compounds **8** (Sch. 1).

The results of the studies on the fluorescent properties of the indolo[1,2-*a*]quinoxalines **7** will be published elsewhere.

EXPERIMENTAL

Preparation of Compounds 5a–j: General Procedure

A mixture of appropriate ethyl 3-benzylindole-2-carboxylate **4** (50 mmol), appropriate 2-bromo/chloronitrobenzene (50 mmol),

Table 4. Physical, IR, and ^{1}H-NMR spectral data of 9-substituted 7-benzylindolo[1,2-*a*]quinoxalines.

Product	*R*	*X*	Yield[a] (%)	M.p.[b] (°C)	IR (KBr)[c] ν (cm^{-1})	^{1}H NMR[d,e] ($CDCl_3$, TMS) δ (ppm), *J* (Hz)
7a	H	H	75	168–169	1622, 1560, 1492, 1449, 1372, 1285	4.50 (s, 2H, $CH_2C_6H_5$), 7.16–7.27 (m, 5H, C-7 ArH), 7.39 (t, 2H, H-9 and H-10), 7.50–7.61 (m, 2H, H-2 and H-3), 7.85 (d, 1H, $J=7.90$, H-8), 7.95 (dd, 1H, $J=7.95$, $J=1.55$, H-4), 8.40 (d, 2H, $J=8.5$, H-1 and H-11), 8.94 (s, 1H, HC=N).
7b	CH_3	H	73	209–210	1628, 1559, 1491, 1455, 1406, 1361, 1284	2.52 (s, 3H, CH_3), 4.47 (s, 2H, $CH_2C_6H_5$), 7.15–7.27 (m, 5H, C-7 ArH), 7.33–7.36 (m, 1H, H-4), 7.40 (dd, 1H, $J=7.82$, $J=1.27$, H-10), 7.56 (dd, 1H, $J=8.38$, $J=1.67$, H-2), 7.61 (d, 1H, $J=1.75$, H-8), 7.95 (dd, 1H, $J=7.88$, $J=1.61$, H-4), 8.28 (d, 1H, $J=8.80$, H-11), 8.38 (dd, 1H, $J=8.32$, $J=1.13$, H-1), 8.92 (s, 1H, CH=N).
7c	H	Cl	60	173–174	1616, 1603, 1473, 1430	4.52 (s, 2H, $CH_2C_6H_5$), 7.18–7.28 (m, 5H, C-7 ArH), 7.38–7.61 (m, 3H, H-2, H-9 and H-10), 7.88 (d, 1H, $J=8.07$, H-8), 7.94 (d, 1H, $J=2.51$, H-4), 8.33 (d, 2H, $J=8.89$, H-1 and H-11), 8.96 (s, 1H, CH=N).
7d	CH_3	Cl	70	198–199	1622, 1551, 1473, 1427, 1374, 1286	2.52 (s, 3H, CH_3), 4.45 (s, 2H, $CH_2C_6H_5$), 7.18–7.26 (m, 5H, C-7 ArH), 7.35 (dd, 1H, $J=8.83$, $J=1.57$, H-10), 7.48 (dd, 1H, $J=8.89$, $J=2.5$, H-2), 7.62 (s, 1H, H-8), 7.90 (d, 1H, $J=2.48$, H-4), 8.15 (d, 1H, $J=8.83$, H-11), 8.23 (d, 1H, $J=8.91$, H-1), 8.88 (s, 1H, –CH=N).
7e	OCH_3	Cl	75	195–196	1610, 1597, 1493, 1431, 1326, 1258, 1241	3.85 (s, 3H, OCH_3), 4.46 (s, 2H, $CH_2C_6H_5$), 7.20–7.21 (m, 2H, H-8 and H-10), 7.23–7.27 (m, 5H, C-7 ArH), 7.49 (dd, 1H, $J=8.90$, $J=2.51$, H-2), 7.91 (d, 1H, $J=2.47$, H-4), 8.21 (d, 1H, $J=9.15$, H-11), 8.22 (d, 1H, $J=8.91$, H-1), 8.88 (s, 1H, –CH=N).

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7f	OC_2H_5	Cl	65	144–145	1622, 1455, 1390, 1228, 1204	1.46 (t, 3H, $J=6.96$, CH_3), 4.08 (q, 2H, $J=6.96$, OCH_2CH_3), 4.48 (s, 2H, $CH_2C_6H_5$), 7.16–7.28 (m, 7H, H-8, H-10, C-7 ArH), 7.61 (dd, 1H, $J=8.57$, $J=2.47$, H-2), 7.98 (d, 1H, $J=2.51$, H-4), 8.32 (d, 1H, $J=9.07$, H-11), 8.38 (d, 1H, $J=8.61$, H-1), 8.93 (s, 1H, CH=N).
7g	H	OCH_3	78	145–146	1598, 1560, 1490, 1437, 1334, 1261	3.93 (s, 3H, OCH_3), 4.52 (s, 2H, $CH_2C_6H_5$), 7.19 (dd, 1H, $J=9.1$, $J=2.96$, H-2), 7.25–7.27 (m, 5H, C-7 ArH), 7.38 (m, 1H, H-9), 7.49 (d, 1H, $J=2.74$, H-4), 7.54 (dd, 1H, $J=8.6$, $J=1.28$, H-10), 7.86 (d, 1H, $J=8.06$, H-8), 8.35 (d, 1H, $J=9.13$, H-1), 8.37 (d, 1H, $J=8.64$, H-11), 8.96 (s, 1H, CH=N).
7h	CH_3	OCH_3	85	186–187	1610, 1592, 1560, 1495, 1434, 1328, 1265, 1240	2.52 (s, 3H, CH_3), 3.93 (s, 3H, OCH_3), 4.49 (s, 2H, $CH_2C_6H_5$), 7.19 (dd, 1H, $J=8.94$, $J=3.04$, H-2), 7.25–7.27 (m, 5H, C-7 ArH), 7.34 (dd, 1H, $J=8.83$, $J=1.58$, H-10), 7.46 (d, 1H, $J=2.96$, H-4), 7.62 (s, 1H, H-8), 8.25 (d, 1H, $J=8.87$, H-11), 8.31 (d, 1H, $J=9.1$, H-1), 8.92 (s, 1H, CH=N).
7i	OCH_3	OCH_3	75	195–196	1610, 1555, 1477, 1452, 1427, 1330, 1261	3.85 (s, 3H, C-9 OCH_3), 3.93 (s, 3H, C-3 OCH_3), 4.48 (s, 2H, $CH_2C_6H_5$), 7.14–7.22 (m, 3H, H-2, H-8, H-10), 7.26–7.28 (m, 5H, C-7 ArH), 7.48 (d, 1H, $J=2.96$, H-4), 8.27 (d, 1H, $J=9.76$, H-11), 8.30 (d, 1H, $J=9.14$, H-1), 8.91 (s, 1H, CH=N).
7j	OC_2H_5	OCH_3	80	165–166	1609, 1553, 1452, 1431, 1391, 1328, 1262	1.45 (t, 3H, $J=7.00$, OCH_2CH_3), 3.93 (s, 3H, OCH_3), 4.07 (q, 2H, $J=6.98$, OCH_2CH_3), 4.48 (s, 2H, $CH_2C_6H_5$), 7.15 (d, 1H, $J=2.94$, H-8), 7.17–7.22 (m, 2H, H-2 and H-10), 7.25–7.28 (m, 5H, C-7 ArH), 7.48 (d, 1H, $J=2.98$, H-4), 8.26 (d, 1H, $J=9.73$, H-11), 8.29 (d, 1H, $J=9.14$, H-1), 8.91 (s, 1H, CH=N).

[a]The yields are not optimized.
[b]Melting points were taken in open capillaries and are uncorrected.
[c]The IR spectra were recorded on Nicolet Impact-410 FT-IR spectrophotometer.
[d]The NMR spectra were recorded on Bruker Avance-200 MHz FT-NMR Instrument.
[e]Satisfactory microanalyses were obtained: C, ±0.30; H, ±0.32; N, ±0.17.

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Table 5. ^{1}H-NMR (500 MHz), ^{13}C-NMR (125 MHz), ^{1}H–^{1}H COSY, and HMBC spectral data of 3-chloro-9-methyl-7-benzylindolo[1,2-a]quinoxaline.[a]

Position	δ (ppm), mult, intgr., J (Hz)	δC (HSQC)	^{1}H-^{1}H COSY	HMBC (C. No.)
1	8.17, d, 1H, 8.5	115.30	H-2	4a, 3
12a	—	129.12	—	—
2	7.47, dd, 1H, 9,2.5	128.10	H-1	4, 12a
3	—	128.44	—	—
4	7.91, d, 1H, 2.5	129.48	H-2	2, 12a
4a	—	136.83	—	—
6	8.88, s, 1H	147.18	H-7′	6a, 4a
6a	—	126.75	—	6, 7′
7	—	112.60	—	8
7a	—	129.42	—	11, 7′
8	7.63, s, 1H	120.33	H-10	7, 10, 11a, 9′
9	—	132.23	—	—
10	7.35, d, 1H, 9	126.87	H-8, H-11	11a, 8
11	8.11, d, 1H, 8.5	113.78	H-10	9, 7a
11a	—	130.41	—	—
1′	—	140.04	—	—
2′, 6′	7.35, m, 2H	128.56	—	—
3′, 5′	7.35, m, 2H	128.10	—	—
4′	7.25, m, 1H	126.30	—	—
7′	4.45, s, 1H	29.31	—	7, 6a, 2′, 6′, 7a, 1′
9′	2.55, s, 1H	21.50	–	8, 9, 10

[a]The NMR spectra were recorded at 500 MHz on Bruker DRX-500 Instrument.

anhydrous potassium carbonate (7 g) and cupric oxide (0.25 g) in dry pyridine (20 mL) was heated under reflux for 30–35 h. The mixture was cooled and filtered. The residue was washed thoroughly with hot pyridine. The combined filtrate was poured into ice-cold dilute hydrochloric acid. The brownish solid that separated was collected by filtration and dried. The solid was crystallized from ethanol to get the corresponding ethyl 1-(2-nitrophenyl)-3-benzylindole-2-carboxylates **5a–j** as yellow shining needles.

Preparation of Compounds 6a–j: General Procedure

Appropriate nitro compound **5** (5 g) was subjected to reductive cyclization in dimethylformamide (80 mL) with freshly prepared Raney nickel

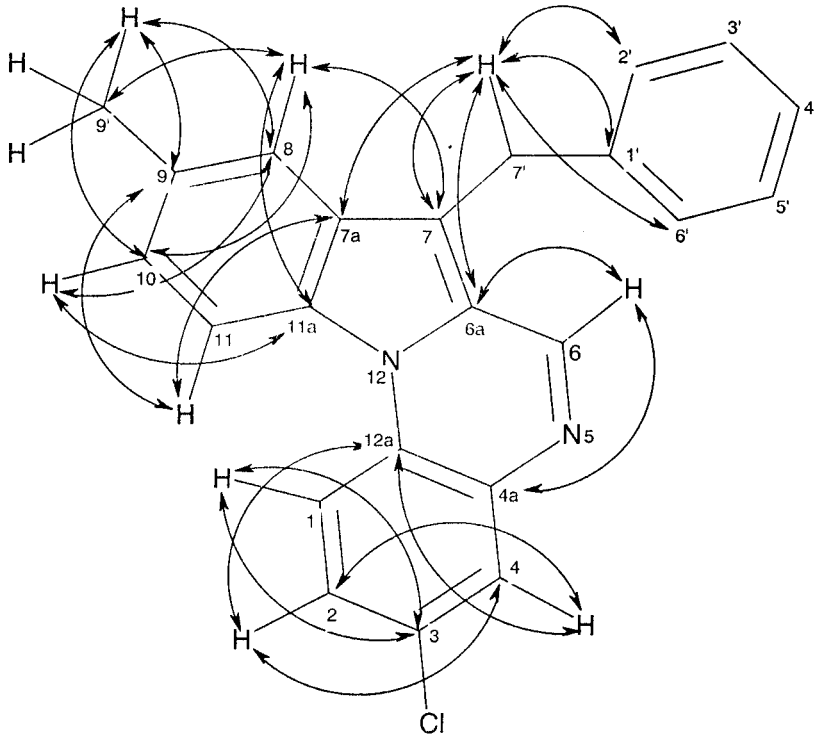
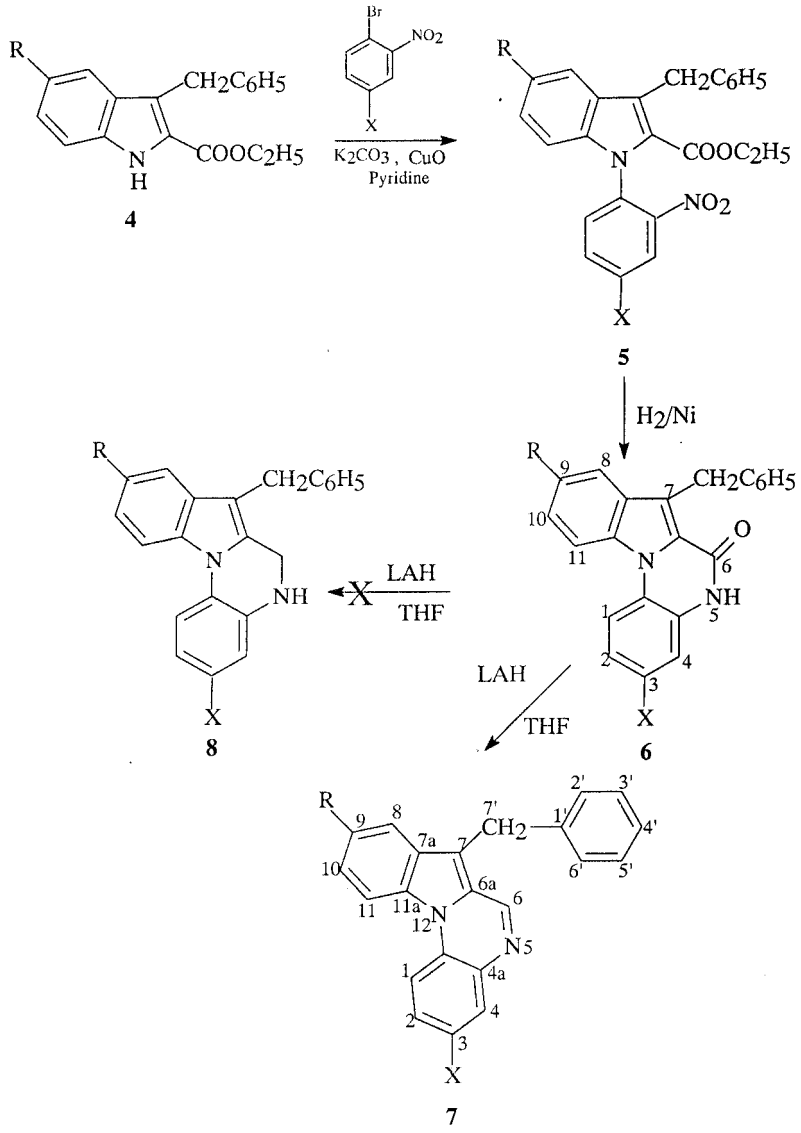


Figure 1. 2D-NMR correlations.

(2.5 g) and hydrogen (60 lb/in^2) in Paar low-pressure hydrogenator for 8 h. The catalyst was removed by filtration and repeatedly washed with dimethylformamide. The solvent was removed under reduced pressure when quinoxaline amides were obtained as very light yellow solids. All these quinoxaline amides were crystallized from dimethylformamide-ethanol mixture to get corresponding 7-benzylindolo[1,2-*a*]quinoxalin-6(5*H*)-ones **6a–j** as pale yellow crystals.

Reduction of 6a–j: General Procedure

A suspension of appropriate **6** (10 mmol) in dry tetrahydrofuran (80 mL) was added to a well stirred slurry of LAH (20 mmol) in dry ether (50 mL). The mixture was heated under reflux for 10 h and cooled. The excess of lithium aluminium hydride was decomposed by drop wise addition of water (1 mL), sodium hydroxide (15%, 1.5 mL)



Scheme 1.

and water (2 mL). The white precipitate formed was filtered off and the precipitate was washed several times with boiling benzene. The combined filtrate, after washing with water was dried over anhydrous sodium sulfate. Removal of the solvent by distillation under reduced pressure furnished yellow solid, which was crystallized from ethanol to get the corresponding 7-benzylindolo[1,2-*a*]quinoxalines **7a–j** as yellow shining needles.

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