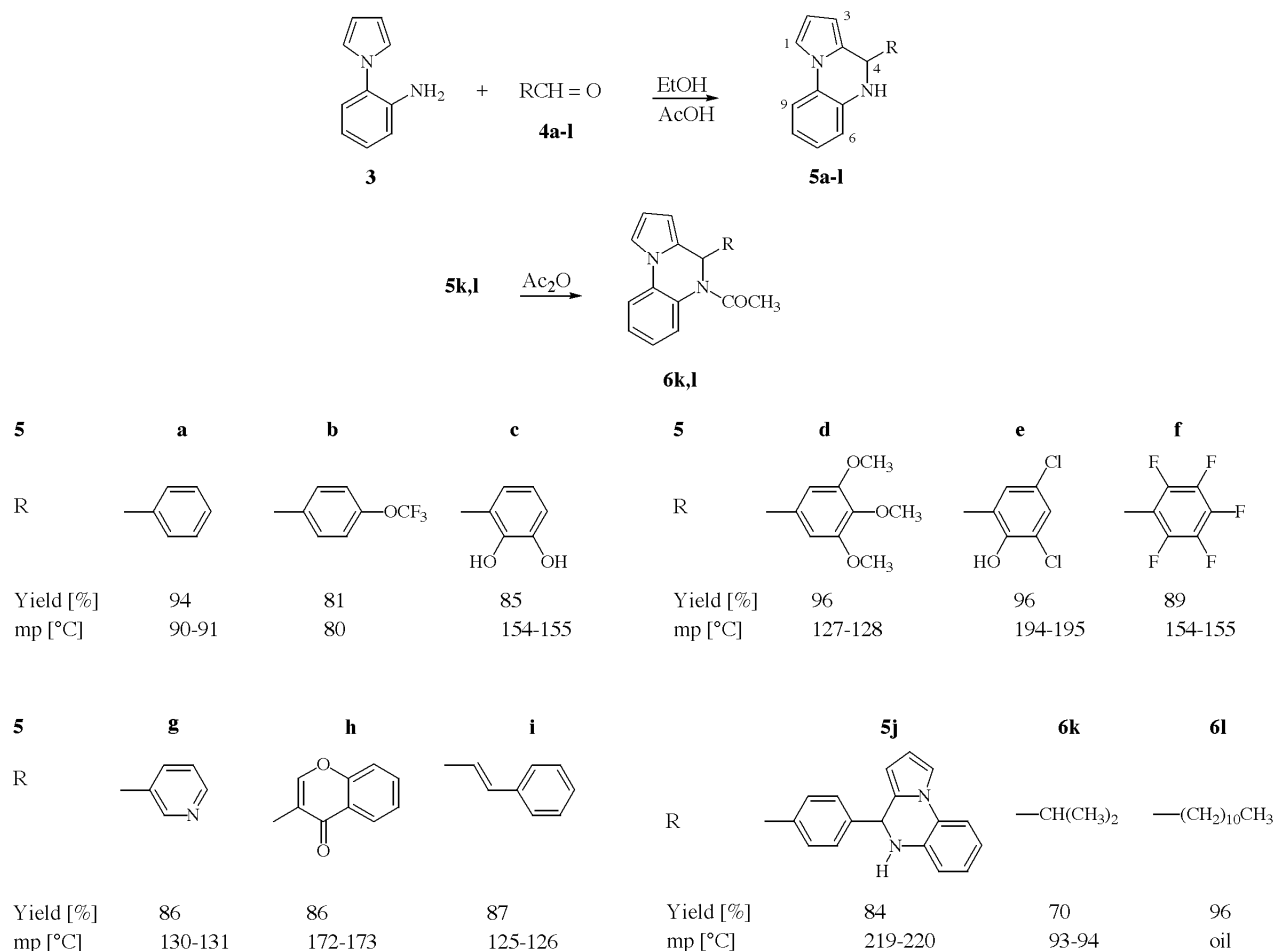




Scheme 2



product precipitated immediately. It turned out that the primarily formed Schiff base **7** does not undergo cyclization, even when subjected to higher temperatures and longer reaction times.

The mass spectra of compounds **5** showed that the molecular ions  $M^{+\bullet}$  are preferentially cleaved to  $[M - R]^+$  where R represents the substituent on C-4; additionally the acetyl groups in **6k,l** are split off, so that the ion  $[C_{11}H_9N_2]^+$  with  $m/z = 169$  is formed in all cases **5a-j** and **6k,l**. The compounds were extensively characterized by their  $^1H$  and  $^{13}C$  nmr spectra including 2D-techniques like COSY, HMBC, NOESY [14]. The assignments of the  $^1H$

and  $^{13}C$  signals for **5d** are shown in Figure 1. The various substituents on C-4 have only a small influence on the chemical shifts of the tricyclic scaffold.

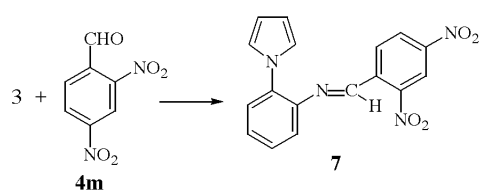
## EXPERIMENTAL

The melting points were determined on a Kofler apparatus and are uncorrected. The  $^1H$  and  $^{13}C$  nmr spectra were recorded with Varian Gemini 200 and Bruker AM 400 instruments; chemical shifts ( $\delta$ ) are reported in ppm relative to tetramethylsilane as internal standard. Silica gel plates (Merck F<sub>254</sub>) were used for analytical tlc. The mass spectra were run on a Varian Model MAT MS-311 spectrometer at 70 eV. Microanalyses were performed with a Perkin Elmer Model 240 C Elemental Analyser.

**General Procedure for the Preparation of the 4,5-Dihydropyrrolo[1,2-a]quinoxalines **5a-l**.**

A solution of 1-(2-aminophenyl)pyrrole **3** (0.20 g, 1.26 mmol) and the corresponding aldehyde **4** (1.26 mmol) and 0.63 mmol of **4j**), in ethanol (2 ml) and acetic acid (5 drops) was heated to 50 °C during 5 to 10 minutes, following the reaction by tlc on silica gel plates with chloroform as the

Scheme 3



eluent. After cooling, the resulting precipitate of **5a-i** was filtered off and recrystallized from ethanol. As soon as the starting compounds for **5k,l** were consumed, 194 mg (1.9 mmoles) of acetic anhydride was added, and the reaction mixture was heated at 50 °C for 2 minutes more. The solvent was removed under reduced pressure ( $1.5 \times 10^3$  Pa) and the residue was purified by column chromatography (3 x 20 cm silica gel, chloroform).

#### 4,5-Dihydro-4-phenylpyrrolo[1,2-*a*]quinoxaline (**5a**).

The compound was obtained from benzaldehyde (**4a**) as colorless crystals in 94% yield, mp 90-91 °C. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  4.13 (br s, 1H, N-H), 5.53 (s, 1H, 4-H), 5.60 (dd,  $^3J = 2.9$  Hz,  $^4J = 1.5$  Hz, 1H, 3-H), 6.26 (t, 1H, 2-H,  $^3J = 3.0$  Hz), 6.73 (dd, 1H, 6-H), 6.86 (td, 1H, 8-H), 6.98 (td, 1H, 7-H), 7.20 (dd,  $^3J = 3.0$  Hz,  $^4J = 1.5$  Hz, 1H, 1-H), 7.32-7.46 ppm (m, 6H, 9-H and phenyl protons);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta = 56.2$  (C-4), 106.0 (C-3), 110.2 (C-2), 114.4 (C-1), 114.8 (C-9), 115.5 (C-6), 119.5 (C-8), 124.7 (C-7), 125.6 (C-9a), 127.8, 128.3, 128.7 (CH, Phenyl), 129.8 (C-3a), 136.0 (C-5a), 141.3 (C<sub>q</sub>, Phenyl); ms:  $m/z$  (%) 246 (44) [ $\text{M}^{+\bullet}$ ], 169 (100).

*Anal.* Calcd. for  $\text{C}_{17}\text{H}_{14}\text{N}_2$ : C, 82.90; H, 5.73; N, 11.37. Found: C, 82.86; H, 5.57; N, 11.36.

#### 4,5-Dihydro-4-(4-trifluoromethoxyphenyl)pyrrolo[1,2-*a*]quinoxaline (**5b**).

This compound was obtained from *p*-trifluoromethoxybenzaldehyde (**4b**) as colorless crystals in 81% yield, mp 80°. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  4.18 (br s, 1H, N-H), 5.54 (s, 1H, 4-H), 5.57 (dd,  $^3J = 3.4$ ,  $^3J = 1.5$  Hz, 1H, 3-H), 6.26 (t,  $^3J = 3.4$  Hz, 1H, 2-H), 6.74 (dd, 1H, 6-H), 6.87 (td, 1H, 8-H), 6.99 (td, 1H, 7-H), 7.22 (m, 3H, 1-H and two arom. protons), 7.34 (dd, 1H, 9-H), 7.52 (br d, 2 H, arom. protons);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  55.5 (C-4), 106.0 (C-3), 110.3 (C-2), 114.6/ 114.8 (C-1, C-9), 115.4 (C-6), 119.7 (C-8), 120.5 (OCF<sub>3</sub>,  $^1J = 384.2$  Hz), 121.1/ 129.4 (CH, Aryl), 124.8 (C-7), 125.4 (C-9a), 129.4 (C-3a), 135.8 (C-5a), 140.2/ 149.1 (C<sub>q</sub>, Aryl); ms:  $m/z$  (%) 330 (36) [ $\text{M}^{+\bullet}$ ], 169 (100).

*Anal.* Calcd. for  $\text{C}_{18}\text{H}_{13}\text{N}_2\text{OF}_3$ : C, 65.45; H, 3.97; N, 8.48. Found: C, 65.38; H, 3.79; N, 8.45.

#### 4-(2,3-Dihydroxyphenyl)-4,5-dihydropyrrolo[1,2-*a*]quinoxaline (**5c**).

This compound was obtained from 2,3-dihydroxybenzaldehyde (**4c**) as colorless crystals in 85% yield, mp 154-155 °C. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  5.52 (s, 1H, 4-H), 5.56 (dd,  $^3J = 3.4$ ,  $^4J = 1.5$  Hz, 1H, 3-H), 6.03 (br s, 1H, N-H), 6.24 (t,  $^3J = 3.4$  Hz, 1H, 2-H), 6.69 (dd, 1H, 6-H), 6.77-7.06 (m, 5H, 7-H, 8-H and aryl protons), 7.19 (m, 1H, 1-H), 7.35 (dd, 1H, 9-H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  56.3 (C-4), 106.7 (C-3), 110.7 (C-2), 115.2/ 115.3/ 115.4 (C-1, C-6, C-9), 117.3, 120.7, 122.3 (CH, aryl), 120.1 (C-8), 122.9/ 143.4/ 145.6 (C<sub>q</sub>, aryl), 124.7 (C-7), 127.1 (C-9a), 128.1 (C-3a), 134.5 (C-5); ms:  $m/z$  (%) 278 (46) [ $\text{M}^{+\bullet}$ ], 169 (100).

*Anal.* Calcd. for  $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_2$ : C, 73.37; H, 5.07; N, 10.07. Found: C, 73.38; H, 5.13; N, 10.06.

#### 4,5-Dihydro-4-(3,4,5-trimethoxyphenyl)pyrrolo[1,2-*a*]quinoxaline (**5d**).

This compound was obtained from 3,4,5-trimethoxybenzaldehyde (**4d**) as colorless crystals in 96% yield, mp 127-128 °C. The  $^1\text{H}$  nmr and the  $^{13}\text{C}$  nmr data are shown in Figure 1; ms:  $m/z$  (%) 336 (31) [ $\text{M}^{+\bullet}$ ], 169 (100).

*Anal.* Calcd. for  $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_2$ : C, 71.41; H, 5.99; N, 8.33. Found: C, 71.37; H, 5.99; N, 8.26.

#### 4-(3,5-Dichloro-2-hydroxyphenyl)-4,5-dihydropyrrolo[1,2-*a*]quinoxaline (**5e**).

This compound was obtained from 3,5-dichloro-2-hydroxybenzaldehyde (**4e**) as pale yellow crystals in 96% yield, mp 194-195 °C. The  $^1\text{H}$  nmr spectrum in dimethyl sulfoxide-*d*<sub>6</sub> showed signals at  $\delta$  5.72 (d,  $^3J = 2.9$  Hz, 1H, 3-H), 5.91 (s, 1H, 4-H), 6.19 (t, 1H,  $^3J = 2.9$  Hz), 6.51 (br s, 1H, N-H), 6.74 (td, 1H, 8-H), 6.86-6.94 (m, 2H, 6-H and 7-H), 7.01 (d, 1H, aryl), 7.42-7.46 (m, 2H, 1-H and aryl), 7.52 (d, 1H, 9-H);  $^{13}\text{C}$  nmr (hexadeuteriodimethyl sulfoxide):  $\delta$  48.5 (C-4), 105.2 (C-3), 110.0 (C-2), 114.6/ 114.8 (C-1, C-9), 115.2 (C-6), 118.2 (C-8), 121.9/ 123.2/ 124.1/ 124.8/ 126.0/ 126.9/ 127.7/ 135.9/ 149.1 (C-3a, C-7, C-9a and aryl-C), 133.8 (C-5a); ms:  $m/z$  (%) 330 (7) [ $\text{M}^{+\bullet}$ , Cl<sub>2</sub> pattern], 169 (100).

*Anal.* Calcd. for  $\text{C}_{17}\text{H}_{12}\text{N}_2\text{OCl}_2$ : C, 61.65; H, 3.65; N, 8.46. Found: C, 61.71; H, 3.71; N, 8.31.

#### 4,5-Dihydro-4-(pentafluorophenyl)pyrrolo[1,2-*a*]quinoxalines (**5f**).

This compound was obtained from pentafluorobenzaldehyde (**4f**) as colorless crystals in 89% yield, mp 154-155 °C. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  4.06 (br s, 1H, N-H), 5.86 (br d,  $^3J = 3.4$  Hz, 1H, 3-H), 6.26 (s, 1H, 4-H), 6.30 (t,  $^3J = 3.4$  Hz, 1H, 2-H), 6.68 (dd, 1H, 6-H), 6.85 (td, 1H, 8-H), 6.97 (td, 1H, 7-H), 7.22 (dd,  $^3J = 3.0$  Hz,  $^4J = 1.5$  Hz, 1H, 1-H), 7.34 (dd, 1H, 9-H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  45.8 (C-4), 105.6 (C-3), 110.1 (C-2), 114.4/ 114.6 (C-1, C-9), 115.1 (C-6), 119.6 (C-8), 124.8 (C-7), 126.4 (C-9a), 129.7 (C-3a), 133.8 (C-5a), 134.2-147.8 (aryl-C, superimposed); ms:  $m/z$  (%) 336 (35) [ $\text{M}^{+\bullet}$ ], 169 (100).

*Anal.* Calcd. for  $\text{C}_{17}\text{H}_9\text{N}_2\text{F}_5$ : C, 60.72; H, 2.70; N, 8.33. Found: C, 60.75; H, 2.69; N, 8.33.

#### 4,5-Dihydro-4-(3-pyridyl)pyrrolo[1,2-*a*]quinoxaline (**5g**).

This compound was obtained from 3-pyridinecarboxaldehyde (**4g**) as colorless crystals in 86% yield, mp 130-131 °C. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  4.41 (br s, 1H, N-H), 5.56 (m, 2H, 3-H and 4-H), 6.24 (t,  $^3J = 2.9$  Hz, 1H, 2-H), 6.76 (dd, 1H, 6-H), 6.85 (td, 1H, 8-H), 6.97 (td, 1H, 7-H), 7.19 (dd,  $^3J = 2.9$  Hz,  $^4J = 1.5$  Hz, 1H, 1-H), 7.24-7.34 (m, 2H, 9-H and pyridine-H), 7.81 (dt, 1H, pyridine H), 8.56 (dd, 1H, pyridine H), 8.66 (d, 1H, pyridine-H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  53.9 (C-4), 106.1 (C-3), 110.3 (C-2), 114.6/ 114.8 (C-1, C-9), 115.5 (C-6), 119.8 (C-8), 123.8/ 135.7/ 137.1/ 149.4/ 149.8/ (pyridine C), 124.8 (C-7), 125.4 (C-9a), 128.8 (C-3a), 135.6 (C-5a); ms:  $m/z$  (%) 247 (34) [ $\text{M}^{+\bullet}$ ], 169 (100).

*Anal.* Calcd. for  $\text{C}_{16}\text{H}_{13}\text{N}_3$ : C, 77.71; H, 5.30; N, 16.99. Found: 77.68; H, 5.34; N, 17.04.

#### 4-(3-Chromonyl)-4,5-dihydropyrrolo[1,2-*a*]quinoxaline (**5h**).

This compound was obtained from 3-chromonaldehyde (**4h**) as yellow crystals in 86% yield, mp 172-173 °C. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  5.28 (br s, 1H, N-H), 5.90 (s, 1H, 4-H), 6.10 (dd,  $^3J = 3.4$  Hz,  $^4J = 1.5$  Hz, 1H, 3-H), 6.37 (t,  $^3J = 3.4$  Hz, 1H, 2-H), 6.67 (dd, 1H, 6-H), 6.77 (td, 1H, 8-H), 6.88 (td, 1H, 7-H), 7.12 (s, 1H, CH-O), 7.25 (dd,  $^3J = 3.2$  Hz,  $^4J = 1.5$  Hz, 1H, 1-H), 7.28-7.42 (m, 3H, 9-H and two aromatic H), 7.62 (td, 1H, aromatic H), 8.21 (dd, 1H,

aromatic H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  47.7 (C-4), 106.1 (C-3), 110.3 (C-2), 113.7/ 118.2/ 123.8/ 124.1/ 124.6/ 125.0/ 125.2/ 125.7/ 133.9, 154.2, 156.4 (C-3a, C-7, C-9a and chrom. C), 114.6/ 114.9 (C-1, C-9), 116.4 (C-6), 119.3 (C-8), 134.5 (C-5a), 177.7 (C=O); ms:  $m/z$  (%) 314 (100) [ $\text{M}^{+\bullet}$ ], 169 (50).

Anal. Calcd for  $\text{C}_{20}\text{H}_{14}\text{N}_2\text{O}_2$ : C, 76.42; H, 4.49; N, 8.91. Found: C, 76.21; H, 4.55; N, 8.77.

#### 4,5-Dihydro-4-styrylpyrrolo[1,2-*a*]quinoxaline (**5i**).

This compound was obtained from cinnamaldehyde (**4i**) as yellow crystals in 87% yield, mp 125–126 °C. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  4.07 (br s, 1H, N-H), 5.13 (d,  $^3\text{J} = 7.8$  Hz, 1H, 4-H), 5.99 (dd,  $^3\text{J} = 2.9$  Hz,  $^4\text{J} = 1.4$  Hz, 1H, 3-H), 6.31 (t,  $^3\text{J} = 3.0$  Hz, 1H, 2-H), 6.41 (dd,  $^3\text{J} = 7.8$  Hz,  $^3\text{J}_{\text{trans}} = 15.6$  Hz, 1H, olefin. H), 6.64 (d,  $^3\text{J}_{\text{trans}} = 15.6$  Hz, 1H, olefin. H), 6.76 (dd, 1H, 6-H), 6.83 (td, 1H, 8-H), 6.97 (td, 1H, 7-H), 7.19 (dd,  $^3\text{J} = 2.9$  Hz,  $^4\text{J} = 1.5$  Hz, 1H, 1-H), 7.23–7.48 ppm (m, 6H, 9-H and other aromatic H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  56.8 (C-4), 106.5 (C-3), 110.3 (C-2), 113.7, 127.9, 128.5, 128.9, 137.4, 141.6 (aromatic and olefinic C), 114.3/ 114.5 (C-1, C-9), 115.8 (C-6), 119.2 (C-8), 124.6 (C-7), 125.5 (C-9a), 129.6 (C-3a), 136.1 (C-5a); ms:  $m/z$  (%) 272 (100) [ $\text{M}^{+\bullet}$ ], 169 (55).

Anal. Calcd for  $\text{C}_{19}\text{H}_{16}\text{N}_2$ : C, 83.79; H, 5.92; N, 10.29. Found: C, 83.72; H, 5.97; N, 10.02.

#### 1,4-Bis(4,5-dihydropyrrolo[1,2-*a*]quinoxalin-4-yl)benzene (**5j**).

This compound was obtained from terephthaldehyde (**4j**) as pale yellow crystals in 84% yield, mp 219–220 °C. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  4.17 (br s, 2H, N-H) 5.54 (s, 2H, 4-H), 5.59 (d,  $^3\text{J} = 3.4$  Hz, 2H, 3-H), 6.25 (t,  $^3\text{J} = 3.4$  Hz, 2H, 2-H), 6.71 (d, 2H, 6-H), 6.84 (td, 2H, 8-H), 6.97 (t, 2H, 7-H), 7.19 (dd,  $^3\text{J} = 2.9$  Hz,  $^4\text{J} = 1.5$  Hz, 2H, 1-H), 7.32 (dd, 2H, 9-H), 7.45 (s, 4H, aromatic H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  55.9 (C-4), 105.9 (C-3), 110.2 (C-2), 114.4/ 114.7 (C-1, C-9), 115.4 (C-6), 119.4 (C-8), 124.7 (C-7), 125.5 (C-9a), 128.4 (aromatic CH), 129.7 (C-3a), 136.0 (C-5a), 141.6 (aromatic  $\text{C}_q$ ); further small signals at 106.2, 110.3, 115.5, 119.5, 127.6, 129.0 and 134.0 are due to a diastereomer; ms:  $m/z$  (%) 414 (37) [ $\text{M}^{+\bullet}$ ], 169 (91).

Anal. Calcd for  $\text{C}_{28}\text{H}_{22}\text{N}_4$ : C, 81.13; H, 5.35; N, 13.52. Found: C, 80.93; H, 5.42; N, 13.26.

#### 5-Acetyl-4,5-dihydro-4-isopropylpyrrolo[1,2-*a*]quinoxaline (**6k**).

This compound was obtained from isobutyraldehyde (**4k**) as colorless crystals in 70% yield, mp 93–94 °C. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  0.87 (m, 6H,  $\text{CH}_3$ ), 1.46 (m, 1H, CH, isopropyl), 2.17 (s, 3H,  $\text{COCH}_3$ ), 5.56 (d,  $^3\text{J} = 10.3$  Hz, 1H, 4-H), 6.04 (dd,  $^3\text{J} = 3.4$  Hz,  $^4\text{J} = 1.0$  Hz, 1H, 3-H), 6.27 (t,  $^3\text{J} = 3.4$  Hz, 1H, 2-H), 7.11 (dd,  $^3\text{J} = 3.4$  Hz,  $^4\text{J} = 1.5$  Hz, 1H, 1-H), 7.14–7.30 (m, 3H, 6-H, 7-H, 8-H), 7.39 (d, 1H, 9-H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  19.0/ 19.3 ( $\text{CH}_3$ ), 22.5 ( $\text{COCH}_3$ ), 30.7 (CH), 55.6 (C-4), 107.2 (C-3), 110.3 (C-2), 114.0/ 116.0 (C-1, C-9), 123.9 (C-7), 126.1 (C-6), 126.6 (C-8), 128.3 (C-3a), 129.7 (C-9a), 131.0 (C-5a), 169.4 (C=O); ms:  $m/z$  (%) 254 (16) [ $\text{M}^{+\bullet}$ ], 169 (100).

Anal. Calcd for  $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}$ : C, 75.56; H, 7.13; N, 11.01. Found: C, 75.37; H, 7.17; N, 10.98.

#### 5-Acetyl-4,5-dihydro-4-undecylpyrrolo[1,2-*a*]quinoxaline (**6l**).

This compound was obtained from dodecanal (**4l**) as colorless oil in 96% yield. The  $^1\text{H}$  nmr spectrum in deuteriochloroform showed signals at  $\delta$  0.85 (t, 3H,  $\text{CH}_3$ ), 1.18–1.24 (br m, 20H,  $\text{CH}_2$ ), 2.18

(s, 3H,  $\text{COCH}_3$ ), 5.95 (d,  $^3\text{J} = 7.3$  Hz, 1H, 4-H), 6.03 (dd,  $^3\text{J} = 3.4$  Hz,  $^4\text{J} = 1.0$  Hz, 1H, 3-H), 6.27 (t,  $^3\text{J} = 3.4$  Hz, 1H, 2-H), 7.11 (dd,  $^3\text{J} = 2.9$  Hz,  $^4\text{J} = 1.5$  Hz, 1H, 1-H), 7.13–7.30 (m, 3H, 6-H, 7-H, 8-H), 7.39 (d, 1H, 9-H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  14.1 ( $\text{CH}_3$ ), 22.6/ 22.7/ 25.9/ 29.1/ 29.3/ 29.4/ 29.5/ 29.6/ 31.9/ 33.7 ( $\text{CH}_2$ ), 49.5 (C-4), 105.6 (C-3), 110.4 (C-2), 114.0/ 115.9 (C-1, C-9), 123.9 (C-7), 126.2 (C-6), 126.6 (C-8), 127.7 (C-9a), 129.7 (C-3a), 129.7 (C-5a), 169.2 (C=O); ms:  $m/z$  (%) 366 (23) [ $\text{M}^{+\bullet}$ ], 169 (100).

Anal. Calcd for  $\text{C}_{24}\text{H}_{34}\text{N}_2\text{O}$ : C, 78.64; H, 9.35; N, 7.64. Found: C, 78.39; H, 9.54; N, 7.74.

#### *N*-[(*E*)-2,4-Dinitrophenylmethylidene]-2-(1*H*-pyrrol-1-yl)aniline (**7**).

The compound was obtained in a quantitative yield in the reaction of **3** and 2,4-dinitrobenzaldehyde (**4m**); it is a yellow-brown solid with mp 151–152 °C. The  $^1\text{H}$  nmr spectrum in hexadeuteriodimethyl sulfoxide contains the following signals:  $\delta$  6.12 (m, 2H, pyrrole), 7.06 (m, 2H, pyrrol), 7.28 (dd, 1H, aromatic H), 7.33–7.53 (m, 3H, aromatic H), 8.24 (d, 1H, aromatic H), 8.64 (dd, 1H, aromatic H), 8.82 (d, 1H, aromatic H), 8.99 (s, 1H, CH=N);  $^{13}\text{C}$  nmr (hexadeuteriodimethyl sulfoxide):  $\delta$  109.5, 119.9, 120.2, 122.7, 124.9, 127.4, 128.0, 131.0, 134.3, 135.0, 143.5, 148.3, 148.9 (aromatic C), 158.6 (HC=N).

Anal. Calcd for  $\text{C}_{17}\text{H}_{12}\text{N}_4\text{O}_4$ : C, 60.67; H, 3.59; N, 16.72. Found: C, 60.67; H, 3.59; N, 16.72.

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