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Design of Schiff Base-Like Postmetallocene Catalytic Systems for Polymerization of Olefins: IV.* Synthesis of 2-(Aryliminomethyl)-pyrrole and 7-(Aryliminomethyl)indole Derivatives Containing Cycloalkyl Substituents

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Abstract—Reactions of 4,6-substituted 2-cycloalkylanilines with 1*H*-pyrrole-2-carbaldehyde and 1*H*-indole-7-carbaldehyde in methanol in the presence of formic acid gave the corresponding Schiff bases which can be used as ligands for titanium and zirconium complexes.

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We previously demonstrated prospects in structural modification of known catalysts for olefin polymerization based on bis(aryliminopyridine)iron complexes and bis(aryliminoacenaphthenyl)nickel complexes via introduction of cyclic substituents into the *ortho* position of the imino fragment; the modified structures exhibited a high activity at 70–90°C [2].

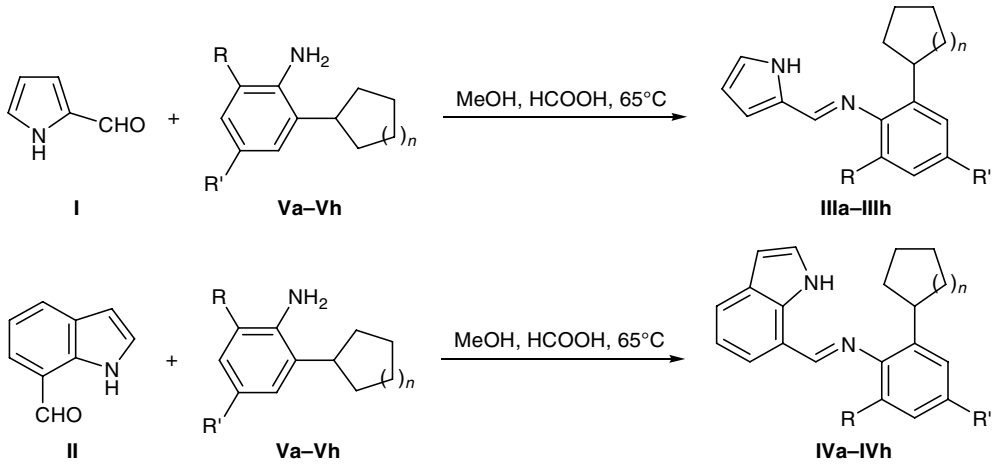
High efficiency of titanium and zirconium complexes with 2-(alkylphenyliminomethyl)pyrrole and 7-(fluorophenyliminomethyl)indole in polymerization and copolymerization of olefins was shown in [3–6]. The pyrrole-based ligands ensured preparation of polyethylene with a very high molecular weight, while the indole derivatives gave rise to living polymerization. Insofar as the high catalytic activity was observed only at moderate temperature, it was interesting and important to elucidate whether analogous complexes with ligands modified by cycloalkyl substituents will be active at elevated temperature. For this purpose we synthesized new ligands, derivatives of 2-(aryliminomethyl)pyrrole and 7-(aryliminomethyl)indole containing cyclopentyl and cyclohexyl substituents.

2-(Aryliminomethyl)pyrrole and 7-(aryliminomethyl)indole ligands were synthesized by reactions of 1*H*-pyrrole-2-carbaldehyde (**I**) and 1*H*-indole-7-carbaldehyde (**II**) with anilines; analogous reactions with alkylanilines and fluoroanilines were described in [4, 6]. We used anilines having cyclopentyl and cyclohexyl substituents in the *ortho* position to obtain new Schiff bases **III** and **IV** and optimized the reaction conditions. According to published data, Schiff bases of the pyrrole series are synthesized in anhydrous ethanol at room temperature [4], while the corresponding indole derivatives are obtained in boiling toluene in the presence of *p*-toluenesulfonic acid [6]. However, the conditions proposed in [4, 6] did not ensure formation of compounds **III** and **IV** in good yields; the conversion of the initial reactants was not complete even on prolonged reaction time, while the condensations in boiling toluene with simultaneous removal of water as azeotrope or in benzene in the presence of molecular sieves were accompanied by appreciable tarring.

We succeeded in attaining almost complete conversion of the reactants (according to the TLC data) by heating aldehydes **I** and **II** with cycloalkylanilines **Va–Vh** in boiling methanol for 6–12 h in the presence

* For communication III, see [1].

Scheme 1.



I Va–Vh IIIa–IIIh

II Va–Vh IVa–IVh

R = R' = H (**a**, **e**), Me (**c**, **g**); R = Me, R' = H (**b**, **f**); R = cyclopentyl, R' = H (**d**); R = cyclohexyl, R' = H (**h**); *n* = 1 (**a**–**d**), 2 (**e**–**h**).

of a catalytic amount of formic acid. In most cases, the target products, Schiff bases **III** and **IV**, separated from the reaction mixture on cooling. Readily soluble compounds **IVd** and **IVe** were isolated by removal of the solvent from the reaction mixture, followed by flash chromatography on silica gel. The yields of iminomethylpyrroles **III** were 48–86%, and iminomethylindoles **IV** were isolated in 50–87% yield.

1*H*-Indole-7-carbaldehyde **II** was synthesized according to the procedure reported in [7] by reaction of excess vinylmagnesium bromide with *o*-nitrobenzaldehyde dibutyl acetal (**VI**). Compound **VI** was not described previously. It was prepared in turn by heating *o*-nitrobenzaldehyde with 1-butanol in boiling toluene with simultaneous removal of water as azeotrope. In the synthesis of aldehyde **II**, the rate of addition of the Grignard compound to a solution of acetal **VI** was important. If the addition was complete in 30 min, the product was isolated in a poor yield (25–30%). The optimal time of addition was 7 to 10 min; in this case, aldehyde **II** was isolated in 50% yield.

The structure of Schiff bases **IIIa**–**IIIh** and **IVa**–**IVh** was confirmed by the analytical and spectral data. Compounds **III** and **IV** displayed in the ^{1}H NMR spectra singlets from the CH=N proton at δ 7.81–8.08 and 8.33–8.53 ppm, respectively. Methylene protons in the cycloalkyl groups resonated as multiplets at δ 1.05–2.14 ppm, and their CH proton signals appeared at δ 3.00–3.50 ppm. The spectra of Schiff bases **IIIb**, **IIIc**, **IIIf**, **IIIg**, **IVb**, **IVc**, **IVf**, and **IVg** contained singlets from the methyl groups at δ 2.08–2.63 ppm, and signals from aromatic protons were located in the region δ 6.10–7.71 ppm. Downfield broadened singlets at δ 10.69–10.71 (**IV**) and 9.47–10.90 ppm (**III**) were assigned to the NH protons in the indole and pyrrole fragments, respectively. In the IR spectra of **IIIa**–**IIIh** and **IVa**–**IVh** we observed a strong absorption band in the region 1619–1633 cm^{-1}, which belongs to stretching vibrations of the azomethine CH=N bond. The mass spectra of all Schiff bases **III** and **IV** were characterized by the presence of strong molecular ion peak.

Compounds **IIIa**–**IIIh** and **IVa**–**IVh** in $CDCl_3$ are likely to exist as a single *E* isomer, as follows from the ^{1}H NMR spectrum of 6-cyclohexyl-2,4-dimethyl-*N*-(1*H*-pyrrol-2-ylmethylidene)aniline (**IIIg**).

EXPERIMENTAL

The ^{1}H NMR spectra were recorded from solutions in carbon tetrachloride on a Bruker WP-200SY spectrometer (200 MHz) using hexamethyldisiloxane as internal reference. The IR spectra were measured in KBr or from neat compounds on a Vector 22 spectrometer. The progress of reactions and the purity of products were monitored by TLC on Silufol UV-254 plates using chloroform as eluent. Flash chromatography [8] was performed on silica gel, 5–40 μm; eluent chloroform–hexane, 2:1. The elemental compositions were determined on a Carlo Erba 1106 CHN analyzer, as well as from the high-resolution mass spectra which were obtained on a Finnigan MAT-8200 mass spectrometer. The melting points were determined by heating samples placed between glass plates at a rate of 1 deg/min.

Substituted cycloalkylanilines **Va**–**Vh** [9], 1*H*-pyrrole-2-carbaldehyde (**I**) [10], vinyl bromide [11],

vinylmagnesium bromide [12], and *o*-nitrobenzaldehyde [13] were synthesized by known methods.

1*H*-Indole-7-carbaldehyde (II) (cf. [7]). A solution of 15.0 g (53 mmol) of acetal **VI** in 150 ml of anhydrous tetrahydrofuran was cooled to –70°C, and a solution of 24.5 g (188 mmol) of vinylmagnesium bromide in 150 ml of anhydrous THF was added dropwise with stirring under argon so that the temperature did not exceed –40°C. The mixture was stirred for 25 min at room temperature, poured into 200 ml of a saturated aqueous solution of ammonium chloride, and treated with diethyl ether (2×50 ml). The extract was dried over $MgSO_4$, the solvent was distilled off under reduced pressure, a mixture of 10 ml of 0.5 M hydrochloric acid and 80 ml of THF was added to the residue, and the mixture was stirred for 40 min under argon, treated with 40 ml of a saturated aqueous solution of $NaHCO_3$, and extracted with diethyl ether (5×40 ml). The extract was dried over $MgSO_4$, the solvent was distilled off, and the oily residue was crystallized from hexane. Yield 3.85 g (50%), mp 87–88°C; published data [7]: mp 87–89°C. ^{1}H NMR spectrum, δ, ppm: 6.52 m (1H, 3-H), 7.17 t (1H, 5-H, *J* = 7.5 Hz), 7.28 m (1H, 2-H), 7.55 d (1H, 6-H, *J* = 7.5 Hz), 7.83 d (1H, 4-H, *J* = 7.5 Hz), 10.01 s (1H, CHO), 10.20 br.s (1H, NH).

Schiff bases IIIa–IIIh and IVa–IVh (*general procedure*). Two drops (10 mg) of 99% formic acid were added to a mixture of 2 mmol of aldehyde **I** or **II**, 10 ml of methanol, and 2 mmol of cycloalkylaniline **Va**–**Vh**. The mixture was heated with stirring under reflux for 6–12 h until the initial compounds disappeared (according to the TLC data), cooled, and the precipitate of Schiff base **IIIa**–**IIIh**, **IVa**–**IVc**, or **IVf**–**IVh** was filtered off and washed with 2 ml of cold methanol. Oily Schiff bases **IVd** and **IVe** were isolated by removal of the solvent, followed by flash chromatography.

2-Cyclopentyl-*N*-(1*H*-pyrrol-2-ylmethylidene)-aniline (IIIa). Yield 79%, mp 90–91°C. IR spectrum: ν 1619 cm^{-1} (N=C). ^{1}N NMR spectrum, δ, ppm: 1.38–1.80 m (6H, CH_2), 1.83–2.04 m (2H, CH_2), 3.48 m (1H, CH), 6.10 t (1H, 4'-H, *J* = 3 Hz), 6.51–7.10 m (6H, 3'-H, 5'-H, 3-H, 4-H, 5-H, 6-H), 8.08 s (1H, N=CH), 10.00 br.s (1H, NH). Found, %: C 80.60; H 7.69; N 11.70. $[M]^+$ 238.1448. $C_{16}H_{18}N_2$. Calculated, %: C 80.63; H 7.61; N 11.75. *M* 238.1470.

2-Cyclopentyl-6-methyl-*N*-(1*H*-pyrrol-2-ylmethylidene)aniline (IIIb). Yield 79%, mp 118–120°C. IR spectrum: ν 1632 cm^{-1} (N=C). ^{1}N NMR spectrum, δ, ppm: 1.38–1.75 m (6H, CH_2), 1.80–1.95 m (2H, CH_2), 2.22 s (3H, CH_3), 3.00 m (1H, CH), 6.08 t (1H, 4'-H, *J* = 3 Hz), 6.38 s (1H, 5-H), 6.45 d (1H, 3'-H, *J* = 3 Hz), 6.85–7.10 m (3H, 5'-H, 3-H, 4-H), 7.88 s (1H, N=CH), 10.35 br.s (1H, NH). Found, %: C 79.77; H 8.04; N 11.01. $[M]^+$ 252.1639. $C_{17}H_{20}N_2$. Calculated, %: C 80.91; H 7.99; N 11.10. *M* 252.1626.

2-Cyclopentyl-4,6-dimethyl-*N*-(1*H*-pyrrol-2-ylmethylidene)aniline (IIIc). Yield 79%, mp 140–142°C. IR spectrum: ν 1629 cm^{-1} (N=C). ^{1}H NMR spectrum, δ, ppm: 1.40–1.75 m (6H, CH_2), 1.80–1.95 m (2H, CH_2), 2.08 s (3H, CH_3), 2.28 s (3H, CH_3), 3.00 m (1H, CH), 6.12 t (1H, 4'-H, *J* = 3 Hz), 6.45 d (1H, 3'-H, *J* = 3 Hz), 6.56 s (1H, 3-H), 6.75 s (1H, 5-H), 6.83 s (1H, 5'-H), 7.86 s (1H, N=CH), 10.20 br.s (1H, NH). Found, %: C 80.98; H 8.46; N 10.57. $[M]^+$ 266.1789. $C_{18}H_{22}N_2$. Calculated, %: C 81.16; H 8.32; N 10.52. *M* 266.1783.

2,6-Dicyclopentyl-*N*-(1*H*-pyrrol-2-ylmethylidene)aniline (IIId). Yield 83%, mp 180°C. IR spectrum: ν 1629 cm^{-1} (N=C). ^{1}H NMR spectrum, δ, ppm: 1.40–1.75 m (12H, CH_2), 1.80–1.95 m (4H, CH_2), 3.01 m (2H, CH), 6.13 t (1H, 4'-H, *J* = 3 Hz), 6.45–6.55 m (2H, 3'-H, 5-H), 6.85–7.15 m (3H, 5'-H, 3-H, 4-H), 7.85 s (1H, N=CH), 10.00 br.s (1H, NH). Found, %: C 82.76; H 8.84; N 9.28. $[M]^+$ 306.2091. $C_{21}H_{26}N_2$. Calculated, %: C 82.31; H 8.55; N 9.14. *M* 306.2096.

2-Cyclohexyl-*N*-(1*H*-pyrrol-2-ylmethylidene)-aniline (IIIe). Yield 48%, mp 71–73°C. IR spectrum: ν 1623 cm^{-1} (N=C). ^{1}H NMR spectrum, δ, ppm: 1.10–1.60 m (6H, CH_2), 1.83–1.95 m (4H, CH_2), 3.03 m (1H, CH), 6.16 t (1H, 4'-H, *J* = 3 Hz), 6.51 d (1H, 3'-H, *J* = 3 Hz), 6.79 m (2H, 5-H, 6-H), 7.02–7.20 m (3H, 5'-H, 3-H, 4-H), 8.08 s (1H, N=CH), 9.47 br.s (1H, NH). Found, %: C 81.77; H 8.52; N 10.40. $[M]^+$ 252.1626. $C_{17}H_{20}N_2$. Calculated, %: C 80.91; H 7.99; N 11.10. *M* 252.1626.

2-Cyclohexyl-6-methyl-*N*-(1*H*-pyrrol-2-ylmethylidene)aniline (IIIf). Yield 86%, mp 150–153°C. IR spectrum: ν 1633 cm^{-1} (N=C). ^{1}H NMR spectrum, δ, ppm: 1.38–1.75 m (6H, CH_2), 1.80–1.95 m (4H, CH_2), 2.30 s (3H, CH_3), 2.80 m (1H, CH), 6.21 t (1H, 4'-H, *J* = 3 Hz), 6.32 s (1H, 5-H), 6.65 d (1H, 3'-H, *J* = 3 Hz), 7.05–7.25 m (3H, 5'-H, 3-H, 4-H), 8.05 s (1H, N=CH), 10.90 br.s (1H, NH). Found, %: C 81.78; H 8.49; N 10.47. $[M]^+$ 266.1786. $C_{18}H_{22}N_2$. Calculated, %: C 81.16; H 8.32; N 10.52. *M* 266.1783.

2-Cyclohexyl-4,6-dimethyl-*N*-(1*H*-pyrrol-2-ylmethylidene)aniline (IIIg). Yield 86%, mp 158–160°C. IR spectrum: ν 1627 cm^{-1} (N=C). ^{1}H NMR

spectrum, δ, ppm: 1.20–1.50 m (6H, CH_2), 1.55–1.85 m (4H, CH_2), 2.03 s (3H, CH_3), 2.24 s (3H, CH_3), 2.31 m (1H, CH), 6.15 t (1H, 4'-H, *J* = 3 Hz), 6.48 d (1H, 3'-H, *J* = 3 Hz), 6.73 s (1H, 5-H), 6.78 s (1H, 3-H), 6.83 m (1H, 5'-H), 7.84 s (1H, N=CH), 10.60 br.s (1H, NH). Found, %: C 81.26; H 8.77; N 10.09. $[M]^+$ 280.1944. $C_{19}H_{24}N_2$. Calculated, %: C 81.38; H 8.63; N 9.99. *M* 280.1939.

2,6-Dicyclohexyl-*N*-(1*H*-pyrrol-2-ylmethylidene)-aniline (IIIh). Yield 77%, mp 219–220°C. IR spectrum: ν 1629 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.40–1.75 m (12H, CH_2), 1.80–1.95 m (8H, CH_2), 3.01 m (2H, CH), 6.13 t (1H, 4'-H, *J* = 3 Hz), 6.51 d (1H, 3'-H, *J* = 3 Hz), 6.85–7.00 m (4H, 5'-H, 3-H, 4-H, 5-H), 8.31 s (1H, N=CH), 10.67 br.s (1H, NH). Found, %: C 82.84; H 9.25; N 8.58. $[M]^+$ 334.2370. $C_{23}H_{30}N_2$. Calculated, %: C 82.59; H 9.04; N 8.37. *M* 334.2409.

2-Cyclopentyl-*N*-(1*H*-indol-7-ylmethylidene)-aniline (IVa). Yield 70%, mp 40–45°C. IR spectrum: ν 1622 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.50–1.85 m (6H, CH_2), 1.90–2.14 m (2H, CH_2), 3.51 m (1H, CH), 6.51 m (1H, 3'-H), 6.80–7.36 m (7H, 2'-H, 3-H, 4-H, 5-H, 5'-H, 6-H, 6'-H), 7.69 d (1H, 4'-H, *J* = 7.5 Hz), 8.53 s (1H, N=CH), 10.71 br.s (1H, NH). Found, %: C 83.67; H 7.02; N 9.76. $[M]^+$ 288.1623. $C_{20}H_{20}N_2$. Calculated, %: C 83.30; H 6.99; N 9.71. *M* 288.1626.

2-Cyclopentyl-*N*-(1*H*-indol-7-ylmethylidene)-6-methylaniline (IVb). Yield 70%, mp 98–103°C. IR spectrum: ν 1625 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.47–1.79 m (6H, CH_2), 1.82–2.00 m (2H, CH_2), 2.14 s (3H, CH_3), 3.05 m (1H, CH), 6.53 m (1H, 3'-H), 6.86–7.28 m (6H, 2'-H, 3-H, 4-H, 5-H, 5'-H, 6'-H), 7.71 d (1H, 4'-H, *J* = 7.5 Hz), 8.35 s (1H, N=CH), 10.70 br.s (1H, NH). Found, %: C 83.50; H 7.39; N 9.23. $[M]^+$ 302.1782. $C_{21}H_{22}N_2$. Calculated, %: C 83.40; H 7.34; N 9.26. *M* 302.1783.

2-Cyclopentyl-*N*-(1*H*-indol-7-ylmethylidene)-4,6-dimethylaniline (IVc). Yield 60%, mp 74–76°C. IR spectrum: ν 1626 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.50–1.73 m (6H, $3CH_2$), 1.80–1.90 m (2H, CH_2), 2.10 s (3H, CH_3), 2.28 s (3H, CH_3), 3.02 m (1H, CH), 6.53 m (1H, 3'-H), 6.76 s (1H, 5-H), 6.85 s (1H, 3-H), 7.10–7.35 m (3H, 2'-H, 5'-H, 6'-H), 7.70 d (1H, 4'-H, *J* = 7.5 Hz), 8.33 s (1H, N=CH), 10.72 br.s (1H, NH). Found, %: C 83.95; H 7.83; N 8.97. $[M]^+$ 316.1941. $C_{22}H_{24}N_2$. Calculated, %: C 83.50; H 7.65; N 8.85. *M* 316.1939.

2,6-Dicyclopentyl-*N*-(1*H*-indol-7-ylmethylidene)-aniline (IVd). Yield 50%, oily substance. IR spectrum: ν 1627 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.50–1.75 m (12H, CH_2), 1.85–2.04 m (4H, CH_2), 3.00 m (2H, CH), 6.55 m (1H, 3'-H), 6.90–7.35 m (6H, 2'-H, 3-H, 4-H, 5-H, 5'-H, 6'-H), 7.72 d (1H, 4'-H, *J* = 8 Hz), 8.33 s (1H, N=CH), 10.69 br.s (1H, NH). Found, %: C 84.50; H 7.90; N 7.93. $[M]^+$ 356.2245. $C_{25}H_{28}N_2$. Calculated, %: C 84.23; H 7.91; N 7.86. *M* 356.2252.

2-Cyclohexyl-*N*-(1*H*-indol-7-ylmethylidene)-aniline (IVe). Yield 50%, oily substance. IR spectrum: ν 1621 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.00–1.80 m (6H, CH_2), 1.80–2.35 m (4H, CH_2), 3.31m (1H, CH), 6.76 m (1H, 3'-H), 7.00–7.70 m (7H, 2'-H, 3-H, 4-H, 5-H, 5'-H, 6-H, 6'-H), 7.93 d (1H, 4'-H, *J* = 8 Hz), 8.36 s (1H, N=CH), 10.96 br.s (1H, NH). Found, %: C 83.05; H 7.30; N 9.31. $[M]^+$ 302.1782. $C_{21}H_{22}N_2$. Calculated, %: C 83.40; H 7.33; N 9.26. *M* 302.1783.

2-Cyclohexyl-*N*-(1*H*-indol-7-ylmethylidene)-6-methylaniline (IVf). Yield 55%, mp 65–66°C. IR spectrum: ν 1627 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.10–1.45 m (6H, CH_2), 1.50–1.90 m (4H, CH_2), 2.14 s (3H, CH_3), 2.63 m (1H, CH), 6.55 m (1H, 3'-H), 6.86–7.35 m (6H, 2'-H, 3-H, 4-H, 5-H, 5'-H, 6'-H), 7.72 d (1H, 4'-H, *J* = 7.5 Hz), 8.36 s (1H, N=CH), 10.70 br.s (1H, NH). Found, %: C 84.17; H 7.83; N 8.92. $[M]^+$ 316.1983. $C_{22}H_{24}N_2$. Calculated, %: C 83.50; H 7.65; N 8.85. *M* 316.1940.

2-Cyclohexyl-*N*-(1*H*-indol-7-ylmethylidene)-4,6-dimethylaniline (IVg). Yield 85%, mp 149–150°C. IR spectrum: ν 1625 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.05–1.45 m (6H, CH_2), 1.55–1.85 m (4H, CH_2), 2.22 s (3H, CH_3), 2.28 s (3H, CH_3), 2.60 m (1H, CH), 6.53 m (1H, 3'-H), 6.77 s (1H, 5-H), 6.81 s (1H, 3-H), 7.10–7.30 m (3H, 2'-H, 5'-H, 6'-H), 7.71 d (1H, 4'-H, *J* = 7.5 Hz), 8.33 s (1H, N=CH), 10.71 br.s (1H, NH). Found, %: C 83.69; H 7.80; N 8.23. $[M]^+$ 330.2101. $C_{23}H_{26}N_2$. Calculated, %: C 83.59; H 7.93; N 8.48. *M* 330.2096.

2,6-Dicyclohexyl-*N*-(1*H*-indol-7-ylmethylidene)-aniline (IVh). Yield 87%, mp 160–162°C. IR spectrum: ν 1626 cm^{-1} (N=C). ^1H NMR spectrum, δ, ppm: 1.00–1.50 m (12H, CH_2), 1.50–1.90 m (8H, CH_2), 2.58 m (2H, CH), 6.56 m (1H, 3'-H), 6.86–7.35 m (6H, 2'-H, 3-H, 4-H, 5-H, 5'-H, 6'-H), 7.74 d (1H, 4'-H, *J* = 7.5 Hz), 8.36 s (1H, N=CH), 10.70 br.s (1H, NH). Found, %: C 84.45; H 8.55; N 7.26. $[M]^+$ 384.2562. $C_{27}H_{32}N_2$. Calculated, %: C 84.33; H 8.39; N 7.28. *M* 384.2565.

***o*-Nitrobenzaldehyde dibutyl acetal (VI).** A mixture of 37.8 g (0.25 mol) of *o*-nitrobenzaldehyde, 56.5 ml (0.62 mol) of butan-1-ol, 75 ml of freshly

distilled toluene, and 0.13 g of *p*-toluenesulfonic acid monohydrate was heated under reflux in a flask equipped with a Dean–Stark trap for 12 h until 4–4.5 ml of water separated. The mixture was treated with 10 ml of a saturated aqueous solution of $NaHCO_3$ and 60 ml of a saturated aqueous solution of sodium chloride. The organic phase was separated and dried over calcined potassium carbonate, the solvent was distilled off under reduced pressure on a rotary evaporator, and the residue was distilled under reduced pressure (oil pump), a fraction with bp 160–163°C (2–3 mm) being collected. Yield 63.3 g (90%). ^{1}H NMR spectrum, δ, ppm: 0.88 t (6H, CH_3, J = 7.5 Hz), 1.35 m (4H, C**H**$_2$CH$_3$), 1.52 m (4H, OC**H**$_2$CH$_2$), 3.47 m (4H, OCH$_2$C**H**$_2$), 5.93 s (1H, CH), 7.36 t.d (1H, 4-H, J = 7.5, 1.5 Hz), 7.48 t.d (1H, 5-H, J = 7.5, 1.5 Hz), 7.64 d.d (1H, 6-H, J = 7.5, 1.5 Hz), 7.72 d.d (1H, 3-H, J = 7.5, 1.5 Hz). Found, %: C 64.14; H 8.34; N 4.92. $[M]^+$ 281.1624. $C_{15}H_{23}NO_4$. Calculated, %: C 64.03; H 8.24; N 4.98. M 281.1627.

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