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Synthesis, Structure, and Heck Cyclization of the *syn*- and *anti*-Atropisomers of *N*-Acyl-*N*-(4-methyl-3,6-dihydro-2*H*-pyran-3-yl)-2-iodo-4,6-dimethylaniline

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Abstract—The reaction of 2-iodo-2,4-dimethylaniline with 3,4-dibromo-4-methyltetrahydro-2*H*-pyran, followed by treatment with acetyl bromide or 4-nitrobenzoyl chloride, gave *syn*- and *anti*-atropisomers of *N*-(2-iodo-4,6-dimethylphenyl)-*N*-(4-methyl-3,6-dihydro-2*H*-pyran-3-yl)acetamide and *N*-(2-iodo-4,6-dimethylphenyl)-*N*-(4-methyl-3,6-dihydro-2*H*-pyran-3-yl)-4-nitrobenzamide. Heating of the acetamide derivative with palladium(II) acetate in the presence of copper(II) acetate and *N*,*N*,*N*′,*N*′-tetramethylethane-1,2-diamine resulted in heterocyclization to *N*-acetyl-4a,6,8-trimethyl-1,4a,9,9a-tetrahydropyrano[3,4-*b*]indole.

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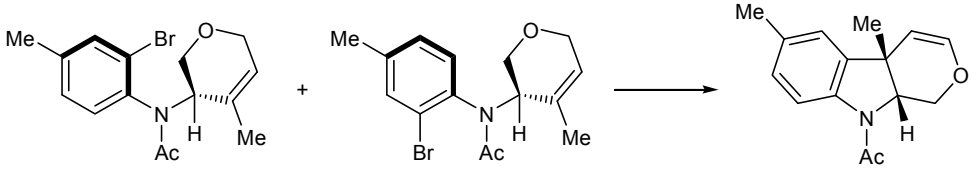
Some pyranoindole heterocycles were found to inhibit hepatitis C virus (HCV) NS5B Δ^{21} polymerase [1, 2]; therefore, synthesis of analogous structures seems to be desirable [3, 4]. We previously synthesized pyranoindole **II** by cyclization of a mixture of *syn*- and *anti*-atropisomers of *N*-pyranyl-substituted aniline **I** [5] (Scheme 1).

In the present work we studied analogous cyclization of sterically crowded homologs of **I**, whose reversible isomerization is hindered. For this purpose, we have synthesized *N*-pyranyl-substituted *o*-iodoanilines. The bromination of 4-methyl-3,6-dihydro-2*H*-pyran (**III**) gave dibromide **IV** which was brought into condensation (without isolation) with 2-iodo-4,6-dimethylaniline (**V**) on heating in boiling triethylamine. Compound **V** was preliminarily prepared by iodination of xylidine **VIII** with benzyltriethylammonium dichloroiodate (**IX**) [6]. The condensation product, *N*-pyranylaniline **VI**, was subjected to acylation with acetyl bromide or 4-nitrobenzoyl chloride in the presence of K_2CO_3 to obtain amides **VIIa** and **VIIb** as mixtures of *syn*- and *anti*-atropisomers (Scheme 2) which can be separated by HPLC (**VIIa**) or column chromatography on silica gel (**VIIb**).

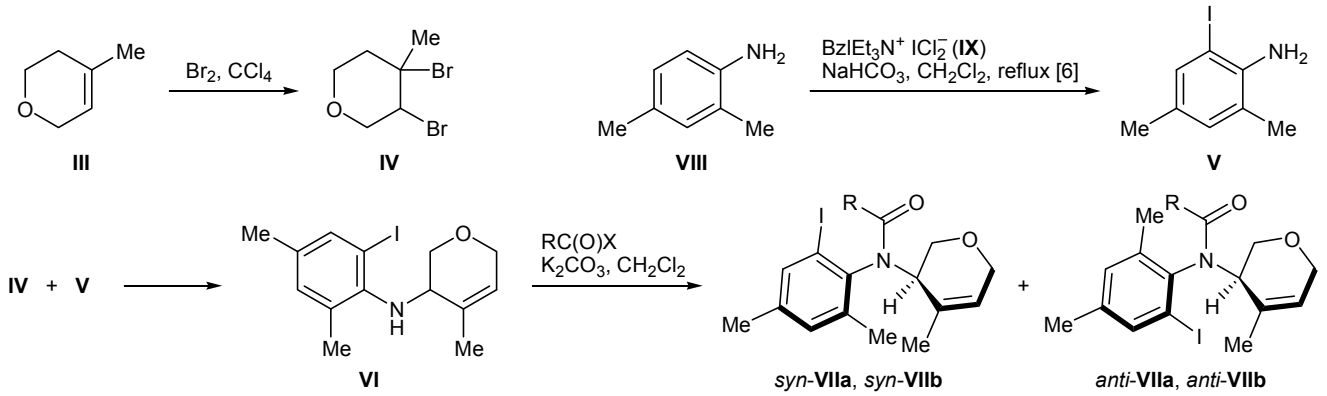
The structure of the products was determined on the basis of their spectral parameters and elemental compositions, as well as by X-ray analysis of *anti*-**VIIb** (see figure). The X-ray diffraction data were used to interpret spectral parameters of the atropisomers. The indole nitrogen atom has almost planar bond configuration (the sum of the bond angles is 356.9°). The dihydropyran ring adopts a *half-chair* conformation which is typical of such rings. The nitro group is coplanar to the benzene ring, while the carbonyl group

Scheme 1.



anti-**I** *syn*-**I** **II**, 70%

Scheme 2.



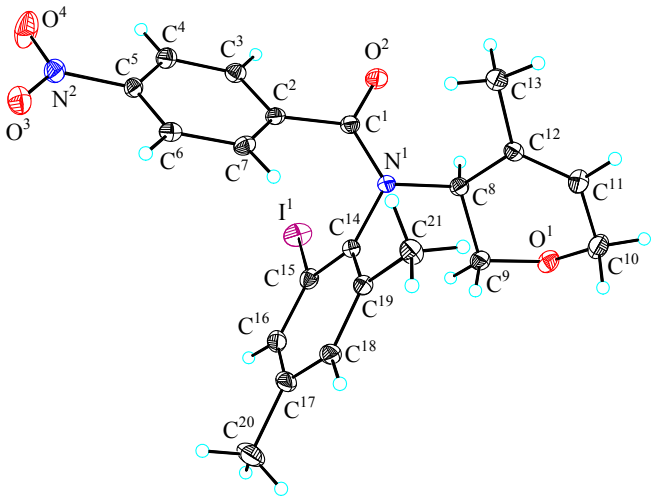
R = Me (**a**), 4-$O_2NC_6H_4$ (**b**).

deviates from the benzene ring plane [the torsion angle $O^2C^1C^2C^3$ is 26.6(2)°], presumably owing to steric repulsion between the benzene rings. The C^1–C^2 bond [1.509(2) Å] is considerably longer than the corresponding standard bond in conjugated systems (1.470 Å) [7]. The bond length distribution in the benzene ring conforms well to the effect of only the nitro group: the C^5–C^6 and C^5–C^4 bonds are shorter [1.387(2) and 1.383(2) Å], the C^2–C^3 and C^2–C^7 bonds are longer [1.400(2) and 1.401(2) Å], and the C^6–C^7 and C^3–C^4 bonds occupy intermediate position [1.392(2) and 1.384(2) Å]. Despite the presence of a carbonyl oxygen atom as lone electron pair donor and iodine atom as lone electron pair acceptor, no intermolecular halogen bond is formed in crystal, and the crystal packing is stabilized by conventional van der Waals interactions.

The *syn* and *anti* isomers of **VIIb** displayed in the ^{1}H NMR spectra appreciable difference between the chemical shifts of methyl protons in the dihydropyranyl fragment; the 4a-Me signal of *anti*-**VIIb** appears in a weaker field (δ 2.01 ppm) relative to the corresponding signal of *syn*-**VIIb** (δ 1.64 ppm). The difference in the chemical shifts of 3-H is also significant. The 3-H proton of *syn*-**VIIb** resonates as a broadened singlet at δ 4.61 ppm. The 3-H singlet in the spectrum of the major *anti*-atropisomer is also broadened, and it appears at δ 5.14 ppm. The difference in the chemical shifts of 5-H is not so large: δ 5.60 and 5.65 ppm (s) for *syn*-**VIIb** and *anti*-**VIIb**, respectively. Thus, the 3-H, 5-H, and 4a-CH_3 signals of *anti*-**VIIb** are observed in a weaker field relative to those of *syn*-**VIIb**.

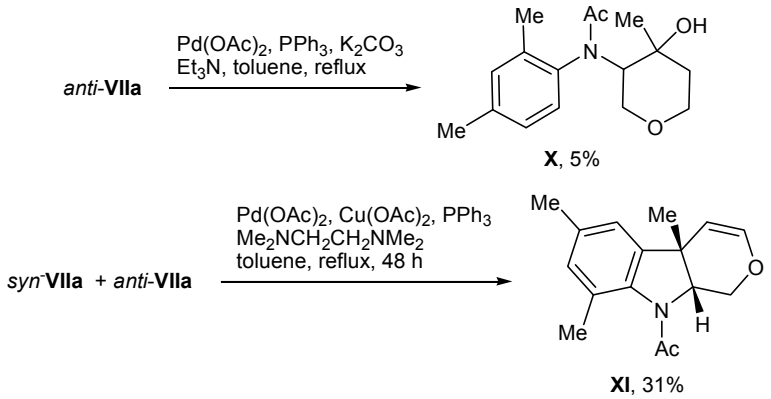
Analysis of the ^{1}H NMR spectra of atropisomers of **VIIa** and **VIIb** also revealed a fairly strong effect of the acyl fragment on the position of the 5-H and 3-H signals. The 3-H proton in the nitrobenzoyl derivative (*syn*-**VIIb**) resonates in a weak field (δ 4.61 ppm), whereas the 3-H signal of *syn*-**VIIa** is located at δ 3.55 ppm (Δδ ≈ 1.06 ppm) and is partially overlapped by the 2-H_B signal. The 3-H and 5-H signals of *anti*-**VIIa** (δ 3.84 and 7.03 ppm, respectively) are displaced downfield relative to the corresponding signals of *syn*-**VIIa** (5-H, δ 6.43 ppm). The general trend in the variation of the chemical shifts of 3-H, 5-H, and 4a-CH_3 in the *anti* atropisomers of **VIIa** and **VIIb** is retained regardless of the nature of the *N*-acyl group.

The synthetic potential of the obtained compounds turned to be limited. Our attempts to cyclize *syn*-**VIIa** and *anti*-**VIIa** to pyranoindole structure by the action of palladium(II) acetate in the presence of copper(II) acetate (or without it), triphenylphosphine, triethylamine, and potassium carbonate were unsuccessful. In the reaction with *anti*-**VIIa**, the conversion was very poor, and we isolated only a small amount of com-



Structure of the molecule of *N*-(2-iodo-4,6-dimethylphenyl)-*N*-(4-methyl-3,6-dihydro-2*H*-pyran-3-yl)-4-nitrobenzamide (*anti*-**VIIb**) according to the X-ray diffraction data.

Scheme 3.



pound **X** (Scheme 3) whose structure was confirmed by elemental analysis and spectral data. By adding *N*,*N*,*N*′,*N*′-tetramethylethane-1,2-diamine to the catalytic system in the reaction with a mixture of *syn*-**VIIa** and *anti*-**VIIa** we succeeded in isolating pyranoindole **XI** with a poor yield (31%), whereas a considerable amount (35%) of unreacted initial compounds was recovered.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded from solutions in $CDCl_3$ on a Bruker Avance III 500 spectrometer at 500.13 and 125.73 MHz, respectively, using tetramethylsilane as internal reference. The elemental compositions were determined on a Hewlett Packard 185B CHN Analyzer. HPLC/MS analyses were carried out on a Shimadzu LCMS-2010EV instrument (atmospheric pressure chemical ionization; eluent methanol–water, 50:50). Semipreparative HPLC was performed using a Luna Silica column (250×10 mm, grain size 10 μm; eluent isopropyl alcohol, flow rate 4 ml/min; detection at λ 254 nm). The purity of liquid samples was checked by GLC on a Khromos GKh-1000 instrument (flame ionization detector; carrier gas helium; 1-m×3-mm column packed with 5% of SE 30 on Chromaton N-AW). Qualitative TLC analyses were carried out on Sorbfil PTSKh-AF-V-UF plates; spots were visualized under UV light (λ 254 nm) and by treatment with iodine vapor. Silica gel MN Kieselgel 60 (140–270 mesh) was used for column chromatography.

Single crystals of *anti*-**VIIb** suitable for X-ray analysis were obtained by slow crystallization from *tert*-butyl methyl ether. Monoclinic crystals, $C_{21}H_{21}IN_2O_4$ (*M* 492.307), with the following unit cell parameters (at 100 K): a = 16.4859(7), b = 7.3644(3), c = 16.7884(7) Å; V = 2001.77(14) Å^3; space group $P2_1/n$; Z = 4; d_{calc} = 1.634 g/cm^3; μ = 1.629 mm^{-1}. Total of 31518 reflection intensities were measured on a Bruker SMART APEX II diffractometer (λMoK_α irradiation, θ_{max} = 33.5°) at 100 K from a 0.32×0.26×0.18-mm^3 single crystal. The initial set of reflection intensities was processed using SAINT and SADABS built in APEX2 [8]; a correction for absorption was applied. The structure was solved by the direct method and was refined against F^2_{hkl} by the least-squares procedure in full-matrix anisotropic approximation for non-hydrogen atoms. The positions of hydrogen atoms were calculated on the basis of geometry considerations and were refined according to the riding model [U_{iso}(H) = nU_{eq}(C), where n = 1.5 for methyl carbon atoms, and n = 1.2 for the other carbon atoms]. Intensities of 7843 independent reflections (R_{int} = 0.0366) were included in the refinement procedure. The final divergence factors were wR_2 = 0.0668 for all independent reflections and R_1 = 0.0300 for 6231 reflections with $I > 2\sigma(I)$. All calculations were performed on an IBM PC using SHELXTL software package [9]. The coordinates of atoms and their temperature factors were deposited to the Cambridge Crystallographic Data Centre (entry no. CCDC 916683) and are available at *http://www.ccdc.cam.ac.uk/products/csd/request/*.

***N*-(2-Iodo-4,6-dimethylphenyl)-4-methyl-3,6-dihydro-2*H*-pyran-3-amine (VI).** A solution of 1.44 ml of bromine in 10 ml of carbon tetrachloride was added dropwise under stirring at 0°C to a solution of 3 g (30.6 mmol) of 4-methyl-3,6-dihydro-2*H*-pyran in 10 ml of carbon tetrachloride. The reaction was complete almost instantaneously. The solution was evaporated under reduced pressure, a solution of 3 g (12 mmol) of 2-iodo-4,6-dimethylaniline (**V**) in 30 ml of triethylamine was added to the residue, and the

mixture was heated for 10 h under reflux. When the initial amine disappeared (TLC), the mixture was cooled to room temperature, a solution of 10 g of sodium hydroxide in 100 ml of water was added, and the mixture was thoroughly stirred and treated with 100 ml of benzene. The organic phase was separated, dried over KOH, and evaporated under reduced pressure, and the residue was subjected to column chromatography on silica gel using benzene as eluent. Yield 2.31 g (55%), viscous material, R_f 0.52 (C_6H_6). ^{1}H NMR spectrum, δ, ppm: 1.85 q (3H, CH_3, J = 2.0 Hz), 2.18 s (3H, CH_3), 2.23 s (3H, CH_3), 3.93–4.27 m (6H, 6-H, 2-H, 3-H, NH), 5.61–5.68 m (1H, 3-H), 6.84 s (1H, 3′-H), 7.36 s (1H, 5′-H). ^{13}C NMR spectrum, δ_C, ppm: 19.7, 19.8, 21.2 (CH_3); 54.2 (C^5); 65.6, 67.8 ($C^{2'}$, $C^{6'}$), 94.8 (C^2); 123.3, 132.4, 137.9 ($C^{3'}$, $C^{5'}$, C^3); 130.6, 133.2; 134.0, 144.3 ($C^{1'}$, $C^{4'}$, $C^{6'}$, C^4). Found, %: C 48.84; H 5.18; I 36.85; N 3.99. $C_{14}H_{18}INO$. Calculated, %: C 48.99; H 5.29; I 36.98; N 4.08.

***N*-(2-Iodo-4,6-dimethylphenyl)-*N*-(4-methyl-3,6-dihydro-2*H*-pyran-3-yl)acetamide (*syn*-VIIa).** Acetyl bromide, 1.12 ml (16 mmol), was added under stirring to a solution of 2.74 g (8 mmol) of amine **VI** in 5 ml of anhydrous methylene chloride in the presence of 2.2 g (16 mmol) of potassium carbonate. The mixture was stirred for 4 h, treated with 20 ml of a saturated solution of sodium hydrogen carbonate, stirred, and extracted with 100 ml of methylene chloride. The organic phase was separated, washed with water (2×20 ml), dried over Na_2SO_4, and evaporated under reduced pressure, and the residue was subjected to column chromatography on silica gel using benzene to benzene–ethyl acetate (4:1) as eluents (gradient elution) to isolate 1.79 g (73% calculated on the 80% conversion) of a mixture of *syn*-**VIIa** and *anti*-**VIIa**. From the preceding fractions we also isolated 0.55 g (20%) of unreacted initial amine **VI**. The isomer mixture was separated by HPLC. Yield of *syn*-**VIIb** 0.59 g (24%), transparent glassy material, R_f 0.45 (PhH–EtOAc, 9:1). ^{1}H NMR spectrum, δ, ppm: 1.61 s (3H, CH_3), 1.77 s (3H, CH_3), 2.26 s (3H, CH_3), 2.29 s (3H, CH_3), 3.45 d.d (1H, 2-H_A, J = 3.7, 10.6 Hz), 3.52–3.61 m (2H, 2-H_B, 3-H), 3.90 d.t (1H, 6-H_A, J = 2.4, 10.9 Hz), 4.05 d.d (1H, 6-H_B, J = 9.7, 10.9 Hz), 6.43 br.s (1H, 5-H), 7.10 s (1H, 5′-H), 7.65 s (1H, 3′-H). Found, %: C 49.77; H 5.16; I 32.85; N 3.49. $C_{16}H_{20}INO_2$. Calculated, %: C 49.88; H 5.23; I 32.94; N 3.64.

***N*-(2-Iodo-4,6-dimethylphenyl)-*N*-(4-methyl-3,6-dihydro-2*H*-pyran-3-yl)acetamide (*anti*-VIIa).** Yield 0.34 g (14%), transparent glassy material, R_f 0.35 (PhH–EtOAc, 9:1). ^{1}H NMR spectrum, δ, ppm: 1.43 s (3H, CH_3), 1.85 s (3H, CH_3), 2.25 s (3H, CH_3), 2.37 s (3H, CH_3), 3.23 d.d (1H, 2-H_A, J = 4.0, 11.0 Hz), 3.62 d.d.d (1H, 2-H_B, J = 1.0, 2.6, 11.0 Hz), 3.84 d.d (1H, 3-H, J = 3.8, 11.0 Hz), 3.95 d.t (1H, 6-H_A, J = 2.9, 11.0 Hz), 4.20 t (1H, 6-H_B, J = 10.9 Hz), 7.03 br.s (1H, 5-H), 7.10 s (1H, 5′-H), 7.64 s (1H, 3′-H). Found, %: C 49.79; H 5.14; I 32.82; N 3.55. $C_{16}H_{20}INO_2$. Calculated, %: C 49.88; H 5.23; I 32.94; N 3.64.

***N*-(2-Iodo-4,6-dimethylphenyl)-*N*-(4-methyl-3,6-dihydro-2*H*-pyran-3-yl)-4-nitrobenzamide (*syn*-VIIb).** 4-Nitrobenzoyl chloride, 0.52 g (2.8 mmol), was added under stirring to a solution of 0.74 g (2.16 mmol) of amine **VI** in 3 ml of anhydrous pyridine. The mixture was stirred for 4 h, 20 ml of water and 200 ml of methylene chloride were added, the mixture was stirred, the organic phase was separated, washed in succession with 4% aqueous HCl (170 ml), water (50 ml), and a saturated solution of $NaHCO_3$, dried over Na_2SO_4, and evaporated under reduced pressure, and the residue was subjected to column chromatography on silica gel. From the first fractions we isolated 0.178 g (24%) of unreacted amine **VI** (conversion 76%). The subsequent elution gave 0.21 g (26% calculated on the 76% conversion) of *syn*-**VIIb** as an amorphous material, R_f 0.7 (PhH–EtOAc, 9:1). ^{1}H NMR spectrum, δ, ppm: 1.64 s (3H, CH_3), 2.16 s (3H, CH_3), 2.55 s (3H, CH_3), 3.76 d.d (1H, 2-H_A, J = 3.7, 12.0 Hz), 4.07–4.39 m (2H, 2-H_B, 6-H_A), 4.48 d.d (1H, 6-H_B, J = 3.7, 12.0 Hz), 4.61 br.s (1H, 3-H), 5.61 br.s (1H, 5-H), 6.97 s (1H, 5′-H), 7.34 s (1H, 3′-H), 7.65 d (2H, H_{arom}, J = 9.0 Hz), 8.02 d (2H, H_{arom}, J = 9.0 Hz). Found, %: C 51.08; H 4.20; I 25.64; N 5.58. $C_{21}H_{21}IN_2O_4$. Calculated, %: C 51.23; H 4.30; I 25.78; N 5.69.

***N*-(2-Iodo-4,6-dimethylphenyl)-*N*-(4-methyl-3,6-dihydro-2*H*-pyran-3-yl)-4-nitrobenzamide (*anti*-VIIb)** was isolated by further elution. Yield 0.37 g (46% for the 76% conversion), R_f 0.6 (PhH–EtOAc, 9:1), mp 119°C (from *tert*-butyl methyl ether). ^{1}H NMR spectrum, δ, ppm: 2.01 s (3H, CH_3), 2.17 s (3H, CH_3), 2.44 s (3H, CH_3), 3.67 d.d (1H, 2-H_A, J = 4.3, 12.6 Hz), 3.97 d.d (1H, 2-H_B, J = 5.0, 12.6 Hz), 4.04–4.11 m (2H, 6-H_A, 6-H_B), 5.14 br.s (1H, 3-H), 5.66 br.s (1H, 5-H), 6.95 s (1H, 5′-H), 7.35 s (1H, 3′-H), 7.61 d (2H, H_{arom}, J = 8.9 Hz), 8.01 d (2H, H_{arom}, J = 8.9 Hz). Found, %: C 51.11; H 4.22; I 25.67; N 5.59. $C_{21}H_{21}IN_2O_4$.Calculated, %: C 51.23; H 4.30; I 25.78; N 5.69.

***N*-(2,4-Dimethylphenyl)-*N*-(4-hydroxy-4-methyltetrahydro-2*H*-pyran-3-yl)acetamide (X).** Com-

pound *anti*-**VIIa**, 0.324 g (0.84 mmol), was dissolved in 2 ml of toluene, 19 mg (0.084 mmol) of $Pd(OAc)_2$, 44 mg (0.017 mmol) of PPh_3, 0.28 ml (0.3 mmol) of Et_3N, 41.4 mg (0.3 mmol) of K_2CO_3, and 182 mg (1 mmol) of $Cu(OAc)_2$ were added, and the mixture was heated for 48 h under reflux (TLC), and evaporated under reduced pressure. The residue was subjected to chromatography on silica gel using benzene as eluent. First fractions contained 0.262 g (81%) of unreacted *anti*-**VIIa**. The subsequent elution gave 0.017 g (5%) of compound **X**, R_f 0.4 (PhH–EtOAc, 9 : 1). ^{1}H NMR spectrum, δ, ppm: 1.62 s, 2.11 s, 2.12 s, 2.23 s (CH_3); 1.75 d.d.d (1H, 5-H_{ax}, J = 4.9, 12.0, 14.8 Hz), 2.81 d.t (1H, 5-H_{eq}, J = 1.8, 14.8 Hz), 3.37 t (1H, 2-H_{ax}, J = 11.0 Hz), 3.44 d.d (1H, 3-H, J = 3.9, 10.4 Hz), 3.52 d.t (1H, 6-H_{ax}, J = 2.0, 12.0 Hz), 3.73 s (1H, OH), 3.78 d.d.d (1H, 6-H_{eq}, J = 1.8, 4.8, 12.0 Hz), 3.89 d.d (1H, 2-H_{eq}, J = 4.0, ^2J = 11.0 Hz), 6.59 d (1H, 6′-H, J = 8.1 Hz), 6.89 s (1H, 3′-H), 6.91 d (1H, 5′-H, J = 8.1 Hz). ^{13}C NMR spectrum, δ_C, ppm:17.2, 20.2, 22.1, 22.6 (CH_3); 34.5 (C^5), 57.3 (C^3), 63.3 (C^6), 66.5 (C^2), 81.7 (C^4), 109.8 ($C^{6'}$); 121.7, 126.2, 142.5 ($C^{1'}$, $C^{2'}$, $C^{4'}$); 127.5 ($C^{7'}$); 131.2 ($C^{3'}$), 170.0 (C=O). Mass spectrum: m/z 278 $[M + H]^+$. Found, %: C 69.18; H 8.28; N 4.97. $C_{16}H_{23}NO_3$. Calculated, %: C 69.29; H 8.36; N 5.05. M 277.36.

1-(4a,6,8-Trimethyl-1,4a,9,9a-tetrahydropyrano-[3,4-*b*]indol-9-yl)ethanone (XI). To 0.371 g (0.96 mmol) of a mixture of *syn*-**VIIa** and *anti*-**VIIa** we added 23 mg (0.1 mmol) of $Pd(OAc)_2$, 52.5 mg (0.2 mmol) of PPh_3, 0.45 ml (0.3 mmol) of *N*,*N*,*N*′,*N*′-tetramethylethane-1,2-diamine, and 182 mg (1 mmol) of $Cu(OAc)_2$. The mixture was heated for 48 h under reflux (TLC) and evaporated under reduced pressure, and the residue was subjected to chromatography on silica gel using benzene as eluent. Yield 0.05 g (31% calculated on the 65% conversion), R_f 0.45 (PhH–EtOAc, 10:1). ^{1}H NMR spectrum, δ, ppm: 1.30 s (3H, CH_3), 2.27 s (3H, CH_3), 2.27 s (3H, CH_3), 3.30 s (3H, CH_3), 3.37 t (1H, 1-H_{ax}, J = 10.9 Hz), 4.15 d.d (1H, 1-H_{eq}, J = 6.1, 10.9 Hz), 4.22–4.32 m (1H, 9a-H), 5.07 d (1H, 4-H, J = 6.1 Hz), 6.42 d (1H, 3-H, J = 6.1 Hz), 6.79 s (1H, 7-H), 6.85 s (1H, 5-H). ^{13}C NMR spectrum, δ_C, ppm: 20.1, 20.9, 22.9, 27.5 (CH_3); 42.0 (C^{4a}), 64.1 (C^{9a}), 65.6 (C^1), 106.3 (C^4), 120.7 (C^5), 130.6 (C^7); 133.7, 135.5, 135.7, 136.2 (C^6, C^8, C^{8a}, C^{4b}); 143.4 (C^3), 169.8 (C=O). Found, %: C 74.52; H 7.35; N 5.36. $C_{16}H_{19}NO_2$. Calculated, %: C 74.68; H 7.44; N 5.44.

Further elution gave 0.13 g (35%) of unreacted mixture of atropisomers *syn*-**VIIa** and *anti*-**VIIa** at a ratio of 1 : 1.

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