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Prototropic Generation of Azomethine Ylides from Esters of *N*-benzylideneaminoacids, and 1,3-Dipolar Cycloaddition Thereof *

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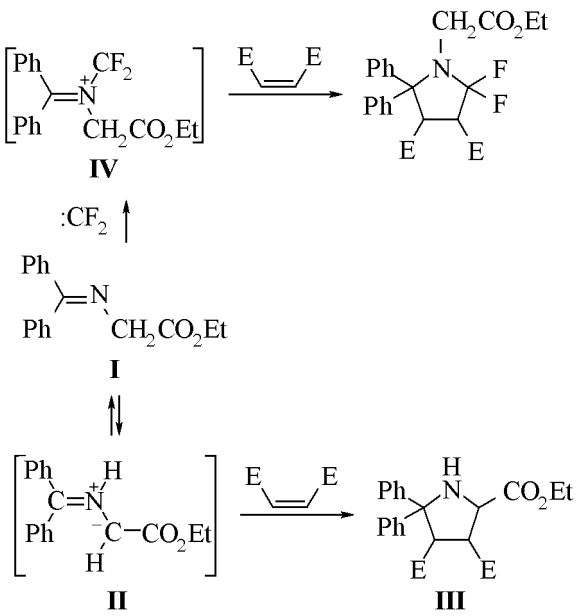
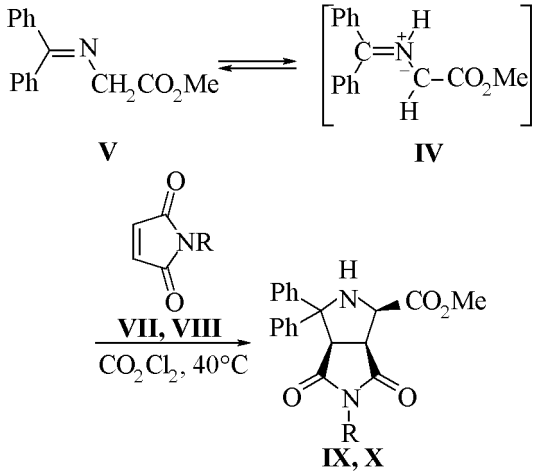
Abstract—Esters of *N*-benzhydrylideneglycin and -alanine undergo prototropic isomerization to yield azomethine ylides that react stereoselectively with N-substituted maleimides and fumaronitrile affording pyrrole derivatives.

Imines of a general formula $ArCH{=}NCHRCO_2R'$, derivatives of benzaldehyde and aminoacids esters, are known to be able to undergo prototropic isomerization into unstable azomethine ylides $ArCH{=}N^+HC^-RCO_2R'$. The latter enter into cycloaddition with various dipolarophiles [1, 2]. The ease of azomethine ylide formation depends on the basicity of the nitrogen atom and the acidity of CH group in the α-position to the nitrogen [1]. Taking into account that pK_a of ethyl *N*-benzylideneglycinate ($PhCH{=}NCH_2CO_2Et$) and ethyl *N*-benzhydrylideneglycinate ($Ph_2C{=}NCH_2CO_2Et$) amount respectively to 19.5 and 18.7 [3] and also that the ylide from the latter compound would possess large resonance stabilization it is presumable that the benzophenone imines would easier undergo the prototropic isomerization into azomethine ylides. Yet the cycloaddition of the *N*-benzhydrylidene derivatives should be more sterically hindered than the reaction of the corresponding benzylidene derivatives. The publications on this topic are virtually lacking. An article was recently published where the addition product **III** to dimethyl maleate of ylide **II** formed at prototropic isomerization of ethyl *N*-benzhydrylideneglycinate (**I**) was obtained as side product under conditions used for generation of difluoro-substituted ylide **IV** from difluorocarbene and imine **I** [4].



E = CO_2Me

In the present study we aimed at investigation of synthetic opportunities provided by cycloaddition of azomethine ylides forming at prototropic isomeriza-



R = Et (**VII, IX**), 4-$CH_3OC_6H_4$ (**VIII, X**).

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tion of azomethines originating from benzophenone and aminoacids esters, and also at optimization of conditions for generating difluoro-substituted ylides of **IV** type from imines derived from aminoacids and difluorocarbene [4–9]. To this end we studied the reactions of *N*-benzhydrylideneglycin and -alanine esters with a number of activated ethylenes.

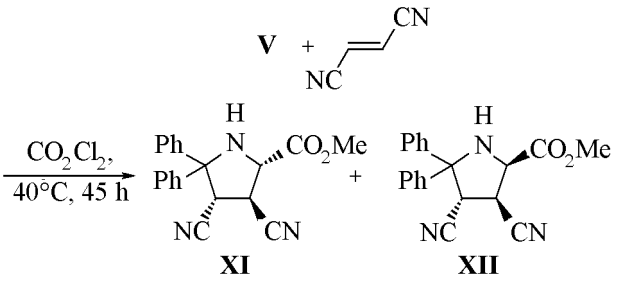
The reaction of imine **V** with maleimides **VII, VIII** in boiling dichloromethane gives rise in 80% yield to an only stereoisomer of the two probable perhydropyrrolo[3,4-*c*]pyrroles **IX, X**. The composition and structure of the products were confirmed by elemental analysis and spectral data.

The configuration of compounds **IX, X** was deduced from the spin-spin coupling constants of protons H^1, H^{6a} amounting to 7.5 Hz. It is known that in the spectra of analogous perhydropyrrolopyrrolecarboxylates with *trans*-configuration at the C^1, C^{6a} atoms the coupling constants for H^1–H^{6a} atoms are close to zero, and in the compounds with *cis*-configuration they equal to 8–9 Hz [10, 11].

The use of fumaronitrile as dipolarophile in the reaction with imine **V** resulted in a mixture of stereoisomeric products **XI, XII** in 56 and 24% yield respectively. The stereochemistry of these products was established from the measurement of nuclear Overhauser effect. With the main isomer **XI** the irradiation of the H^2 proton increased the intensity of the signal from proton H^3 (2%) and that of the signal at δ 7.55 ppm, *J* 7 Hz (3%) corresponding to the *ortho*-protons in the phenyl ring *trans*-directed with respect to the methoxycarbonyl group. The irradiation of the H^3 proton resulted only in a slight (1–2%) increase of the signals from the protons $H^{2,4}$. Yet at the irradiation of the H^4 proton significantly (13%) increased the intensity of the same aromatic protons (δ 7.55 ppm, *J* 7 Hz) as at the irradiation of the H^2 proton. This evidences the *cis*-orientation of the H^2 and H^4 protons.

In the spectrum of isomer **XII** significantly (8%) grew the intensity of H^2 proton at the irradiation of the H^3 proton. The irradiation of H^2 and H^4 protons provided considerable changes in the proton signals of the phenyl rings: On irradiation of the H^2 proton the signal at δ 7.54 ppm, *J* 7 Hz increased by 10%, and on irradiation of H^4 proton grew by 10% the signal of the ortho-protons from another phenyl ring (δ 7,45 ppm, *J* 7 Hz). These findings show that the protons H^2 and H^4 occur in *trans*-orientation, and H^2 and H^3 in *cis*-orientation.

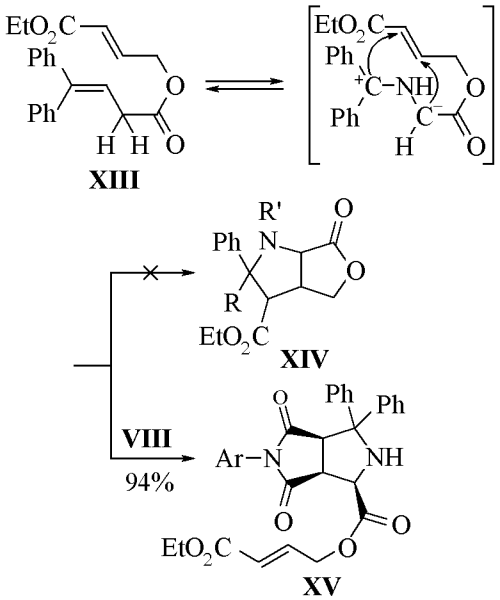
Basing on the above data, taking into account the *trans*-configuration of the fumaronitrile, and stereospecific character of the azomethine ylides cycloaddition we conclude that compound **XI** is 2,3-*trans*-3,4-*trans*-isomer, and compound **XII** 2,3-*cis*-3,4-*trans*-isomer.



XI **XII**

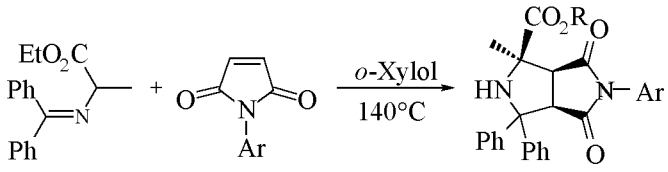
We attempted to carry out intramolecular cycloaddition of ylide generated from the ester of *N*-benzhydrylideneglycin. To this end was synthesized imine **XIII** containing in the molecule a dipolar fragment, a double bond activated with ethoxycarbonyl group. However cycloadduct **XIV** was not obtained at boiling compound **XIII** either in dichloromethane or in *o*-xylene. It is hardly probable that this failure is due to low reactivity of the C=C bond activated with a single electron-acceptor group. It is known, for instance, that ylide obtained from methyl *N*-(naphth-2-yl)-3-allylthioalaninate and containing even nonactivated terminal C=C bond cleanly underwent the intramolecular cycloaddition to afford triazabicyclo[3.3.0]-octane system [12]. The too short chain (3 atoms) connecting dipole and dipolarophile also cannot explain the result obtained since an example exists of compound **XIV** (R = H, R' = *i*-Pr) formation in high yield by intramolecular cycloaddition of an azomethine ylide generated from the corresponding aziridine [13].

Yet the intermolecular reaction of compound **XIII** with imide **VIII** afforded the corresponding product



XV in high yield. Therefore the lack of intramolecular reaction should be ascribed to steric factors since there is no way to take the conformation required by cycloaddition process due to the presence of geminal phenyl groups at the ylide carbon.

Unlike glycin derivatives those of alanine **XVI, XVII** fail to react with imides **VII, XVIII** on boiling in dichloromethane. Yet at higher temperature (boiling in xylene) the corresponding adducts were obtained in 60 and 71% yield respectively. The lesser reactivity of compounds **XVI, XVII** may be due both to lower acidity of the CH group in the α-position with respect to nitrogen (pK_a $PhCH{=}NCHMeCO_2Et$ and $Ph_2C{=}NCHMeCO_2Et$ equals respectively to 18.7 and 22.8 [2]) and to increased steric hindrance to cycloaddition.

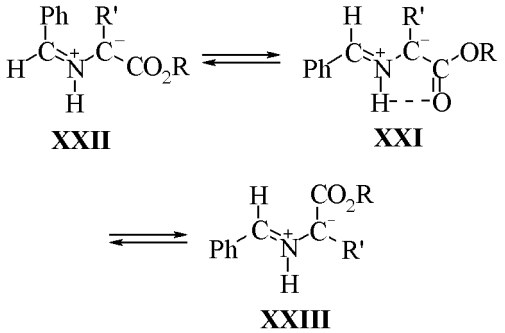


XVI, XVII **VII, VIII** **XIX, XX**

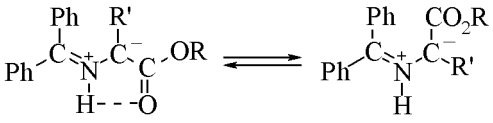
R = Et (**XVI, XIX**), Me (**XVII, XX**); Ar = 4-$MeOC_6H_4$ (**VII, XIX**), 4-ClC_6H_4 (**XVIII, XX**).

The stereochemistry of compound **XX** follows from its NMR spectrum ^1H–^1H NOESY (see Figure). The spectrum indicates a strong effect for *cis*-located protons H^{6a} and H^{3a} and also for methyl group with this protons, whereas with the H^{6a} proton the interaction is stronger. The methyl group interacts besides with the ortho-protons of the *cis*-oriented phenyl ring that also are in strong interaction with the proton H^{6a}. The data indicate *cis*-orientation of the methyl group and the protons H^{6a} and H^{3a}. The similar parameters of the ^1H NMR spectra of compounds **XVII** and **XVIII** evidence the similarity of their stereochemistry.

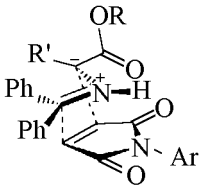
The high stereoselectivity of the reaction between the N-benzylidene derivatives of the α-aminoacids



XXII **XXI** **XXIII**

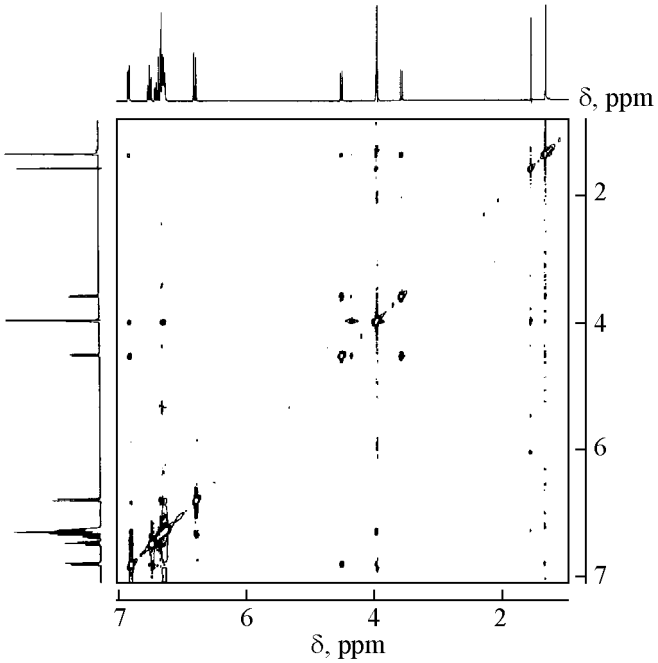


XXIV **XXV**



XXVI

esters and maleimides is ascribed to the higher velocity of addition to this dipolarophile of the primary ylide **XXI** than the rate of this ylide isomerization into ylides **XXII** and **XXIII** [1, 2]. Here the reaction proceeds through an *endo* transition state. With N-benzhydrylidene derivatives arise products **IX, X, XV, XIX**, and **XX** of the same stereochemistry. Taking into account that by steric reasons the primary ylide **XXIV** should be less prone to isomerize into compound **XXV** than ylide **XXI** into compound **XXII**, and that *exo*-cycloaddition with ylide **XXV** should be less favorable than the *endo*-addition with ylide **XXIV** it is presumable that in the case of N-benzhydrylidene derivatives of alanine and glycin esters the reaction also proceeds via *endo* transition state of **XXVI** type.



^1H–^1H NOESY Spectrum of compound **XX**.

Thus the esters of *N*-benzhydrylideneglycin and -alanine undergo a prototropic isomerization into azomethine ylides. The latter react stereoselectively with *N*-aryl- and *N*-alkylmaleimides and with fumaronitrile furnishing pyrrole derivatives. The reaction with the glycin derivatives occurs with considerable velocity already at room temperature whereas isomerization of the alanine derivatives requires more rigid conditions. The data obtained allow the following conclusion: To avoid the effect of prototropic isomerization in preparation of cycloaddition products from azomethine ylides formed from difluorocarbene and the *N*-benzhydrylideneglycin esters [4] the difluorocarbene should be generated by express-procedure with the active lead [9] whereas with the alanine derivatives the common methods are sufficient [7].

EXPERIMENTAL

IR spectra were measured on UR-20 spectrometer from solutions in CCl_4 or $CHCl_3$, the cell 400 μ thick. NMR spectra were registered on Bruker DPX-300 instrument (^1H, 300 MHz; ^{13}C, 75 MHz, internal reference residual signal of $CHCl_3$ in the solvent $CDCl_3$, δ_C 76.7 ppm). Elemental analyses were carried out with CHN-analyzer HP-185B. The reaction progress was monitored by TLC on Silufol UV-254 plates and by GLC on a chromatograph LKhM-80 (glass columns 1.8 and 2.5 m long, stationary phase 5% SE-30 on Chromaton-N-Super). The separation of reaction mixtures by column chromatography was performed on silica gel 5–40 μ.

Ethyl *E*-[2-(*N*-benzhydrylidenamino)acetoxy]but-2-enoate (XIII). To a solution of 5.95 g (16.6 mmol) of tosylate (*E*)-$H_2NCH_2CO_2CH_2CH{=}CHCO_2Et$ in 50 ml of dichloromethane was added 3 g (16.6 mol) of diphenylketimine. The reaction mixture in a stoppered flat-bottom flask was stirred with a magnetic stirrer for 24 h. On completing the reaction the solution was filtered through the glass frit filter, the precipitate was washed with dichloromethane, and the filtrate was evaporated on the rotary evaporator. We obtained 5.52 g (95%) practically pure imine **XI** as light yellow oily fluid. IR spectrum (CCl_4), ν, cm^{-1}: 3065 w, 3030 w, 2985, 2940 w, 2910 w, 1765 s, 1740 s, 1670, 1630, 1450, 1310 s, 1280 s, 1170 s, 1050. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.31 t (3H, CH_3, *J* 7.1 Hz), 4.22 q [2H, CH_2(Et), *J* 7.1 Hz], 4.29 s (2H, CH_2N), 4.83 d.d (2H, CH_2, *J* 4.9, 1.8 Hz), 6.05 d.d (1H, C^2H, *J* 15.9, 1.8 Hz), 6.95 d.d (1H, C^3H, *J* 15.9, 4.9 Hz), 7.19–7.50 m (10H, H arom). ^{13}C NMR spectrum, δ_C, ppm: 13.7 (CH_3), 55.1 (C–N), 60.2, 62.5 (OCH_2), 122.1, 127.2, 127.7, 128.4, 128.6, 130.2, 135.5, 138.7, 140.5 (C=C, C_{Ph}), 165.4 (C=N), 169.6, 171.9 (CO). Found, %: C 71.87; H 5.96; N 3.71. $C_{21}H_{21}NO_4$. Calculated, %: C 71.78; H 6.02; N 3.99.

Imine reactions with dipolarophiles. A mixture of equimolar amounts (1.5–2 mmol) of an imine and a dipolarophile in 10 ml of dichloromethane was boiled under reflux till disappearance of the initial compounds (TLC monitoring) (24–45 h). The reaction mixture was evaporated, and the crystals obtained were recrystallized. The isomers obtained in reaction with fumaronitrile were separated by column chromatography on silica gel (eluent hexane–ethyl ether).

Methyl-(±)-(1*S*,3*aS*,6*aR*)-4,6-dioxo-3,3-diphenyl-5-ethylperhydropyrrolo[3,4-*c*]pyrrole-1-carboxylate (IX). mp 185–186°C (MeOH). IR spectrum ($CHCl_3$), ν, cm^{-1}: 3350 w, 3045 w, 2955 w, 2880 w, 2850 w, 1770 w, 1740, 1710 s, 1600 w, 1450, 1410, 1380 w, 1350, 1320 w, 1250 w, 1125 w. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.07 t (3H, CCH_3 *J* 7.1 Hz), 2.77 d (1H, NH, *J* 7.1 Hz), 3.43 m (2H, CH_2), 3.55 t (1H, H^{6a}, *J* 7.5 Hz), 3.80 d.d (1H, H^1, *J* 7.1, 7.5 Hz), 3.84 s (3H, CH_3O), 4.14 d (1H, H^{3a}, *J* 7.5 Hz), 7.2–7.5 m (10H, H arom). ^{13}C NMR spectrum, δ_C, ppm: 12.6 (CH_3), 33.8 (CH_2), 48.4, 52.0, 52.1, 59.4 ($C^{1,3a,6a}$, CH_3O), 72.8 (C^4), 126.1, 126.9, 127.2, 127.3, 127.5, 128.4, 141.1, 144.2 (C_{Ph}), 170.3, 174.4, 175.2 (CO). Found, %: C 69.68; H 5.83; N 7.45. $C_{22}H_{22}N_2O_5$. Calculated, %: C 69.83; H 5.86; N 7.40.

Methyl-(±)-(1*S*,3*aS*,6*aR*)-5-(4-methoxyphenyl)-4,6-dioxo-3,3-diphenylperhydropyrrolo-[3,4-*c*]-pyrrole-1-carboxylate (X). mp 175–176°C (Et_2O–CH_2Cl_2). IR spectrum ($CHCl_3$), ν, cm^{-1}: 3350 w, 3070 br.w, 2955 w, 2845 w, 1780 w, 1740, 1710 s, 1610 w, 1520, 1445, 1390, 1340 w, 1305 w, 1260, 1170, 1135, 1040. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.93 d (1H, NH, *J* 7.0 Hz), 3.71 t (1H, H^{6a}, *J* 7.5 Hz), 3.79 s (3H, CH_3O), 3.84 s (3H, CH_3O), 3.91 d.d (1H, H^1, *J* 7.0, 7.5 Hz), 4.32 d (1H, H^{3a}, *J* 7.5 Hz), 6.8–7.4 m (14H, H arom). ^{13}C NMR spectrum, δ_C, ppm: 48.7, 52.0, 52.5, 55.1, 59.8 ($C^{1,3a,6a}$, $2CH_3O$), 73.3 (C^3), 114.0, 124.0, 126.1, 126.9, 127.1, 127.3, 127.4, 127.6, 128.5, 141.1, 144.2, 159.1 (Ar), 170.3, 173.8, 174.8 (CO). Found, %: C 70.90; H 5.32; N 6.14. $C_{27}H_{24}N_2O_5$. Calculated, %: C 71.04; H 5.30; N 6.14.

Methyl-(±)-(2*S*,3*S*,4*S*)-5,5-diphenyl-3,4-dicyanotetrahydro-1*H*-pyrrole-2-carboxylate (XI). mp 128–129°C (ether). IR spectrum ($CHCl_3$), ν, cm^{-1}: 3370 w, 3300 w, 2960, 2930, 2880, 2860, 2260 w, 1760 s, 1450, 1330. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 3.15 d (1H, NH, *J* 7.5 Hz), 3.43 d.d (1H, H^3, *J* 9.6, 12.0 Hz), 3.79 d (1H, H^4, *J* 12.0 Hz), 3.86 s (3H, CH_3O), 4.45 d.d (1H, H^2, *J* 7.5, 9.6 Hz), 7.28–7.43 m (6H, H arom), 7.48 m (2H, H^o, *J* 7.1 Hz), 7.55 m (2H, H^o, *J* 7.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 36.4, 44.6 ($C^{3,4}$), 53.0 (CH_3O), 61.2 (C^2), 74.9 (C^5), 116.4, 116.6 (CN), 125.4, 126.9, 128.0, 128.1, 128.4, 128.9, 140.9, 141.1 (Ph), 169.5 (CO). Found, %: C 72.91; H 5.09; N 12.54. $C_{20}H_{17}N_3O_2$. Calculated, %: C 72.49; H 5.17; N 12.68.

Methyl-(±)-(2*R*,3*S*,4*S*)-5,5-diphenyl-3,4-dicyanotetrahydro-1*H*-pyrrole-2-carboxylate (XII). mp 135–136°C (ether). IR spectrum ($CHCl_3$), ν, cm^{-1}: 3310 w, 2960, 2930, 2880, 2860, 2260 w, 1760 s, 1450, 1350. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 3.31 d (1H, NH, *J* 8 Hz), 3.69 d.d (1H, H^3, *J* 7.1, 5.7 Hz), 4.19 d.d (1H, H^2, *J* 7.1, 8 Hz), 3.92 s (3H, CH_3O), 4.30 d (1H, H^4, *J* 5.7 Hz), 7.28–7.48 m (8H, H arom), 7.54 m (2H, H^o, *J* 7.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 36.4, 43.2 ($C^{3,4}$), 53.2 (CH_3O), 60.1 (C^2), 73.7 (C^5), 115.2, 115.7 (CN), 125.6, 126.8, 128.0, 128.2, 128.3, 128.8, 139.1, 141.7 (Ph), 170.6 (CO). Found, %: C 72.68; H 5.13; N 12.77. $C_{20}H_{17}N_3O_2$. Calculated, %: C 72.49; H 5.17; N 12.68.

(*E*)-3-Ethoxycarbonylprop-2-enyl-(±)-(1*S*,3*aS*,6*aR*)-5-(4-methoxyphenyl)-4,6-dioxo-3,3-diphenylperhydropyrrolo[3,4-*c*]pyrrole-1-carboxylate (XV). IR spectrum (CCl_4), ν, cm^{-1}: 3065 w, 3030 w, 2985 w, 2940 w, 1730 s, 1740 s, 1515, 1445, 1390, 1310, 1245, 1180, 1050. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.30 t (3H, CCH_3, *J* 7.1 Hz), 2.88 br.s (NH), 3.75 d.d (1H, H^{6a}, *J* 7.1, 7.5 Hz), 3.78 s (3H, $6CH_3O$), 3.93 d (1H, H^1, *J* 7.1 Hz), 4.30 d (1H, H^{3a}, *J* 7.5 Hz), 4.21 q [2H, CH_2(Et), *J* 7.1 Hz], 4.83 d.d.d (1H, CH_2, *J* 15.9, 4.9, 1.8 Hz), 4.94 d.d.d (1H, CH_2, *J* 15.9, 4.9, 1.8 Hz), 6.10 d.t (1H, C^2H, *J* 15.9, 1.8 Hz), 6.99 d.t (1H, C^3H, *J* 15.9, 4.9 Hz), 7.88–7.34 m (14H, H arom). ^{13}C NMR spectrum, δ_C, ppm: 13.8 (CH_3), 48.5, 52.2 ($C^{3a,6a}$), 55.1 (C^1), 59.5, 60.2, 63.2 (OCH_2, OCH_3), 73.2 (C^3), 114.0, 122.5, 126.1, 127.0, 127.1, 127.4, 127.5, 127.6, 128.5, 140.1, 141.1, 144.1, 159.1, (C=C, Ar), 165.4, 169.4, 173.7, 174.8 (CO). Found, %: C 69.38; H 5.59; N 4.95. $C_{27}H_{24}N_2O_5$. Calculated, %: C 69.30; H 5.45; N 5.05.

Ethyl-(±)-(1*S*,3*aS*,6*aR*)-1-methyl-5-(4-methoxyphenyl)-4,6-dioxo-3,3-diphenylperhydropyrrolo[3,4-*c*]pyrrole-1-carboxylate (XIX). mp 91–93°C (cyclohexane). IR spectrum ($CHCl_3$), ν, cm^{-1}: 3290 w, 3050 w, 2930, 2860, 1820, 1730, 1520, 1450, 1390, 1040. ^1H ($CDCl_3$), δ, ppm: 1.33 s (3H, CH_3), 1.41 t (1H, CH_3, *J* 7.2 Hz), 3.55 d (1H, H^{6a}, *J* 8.2 Hz), 3.78 s (3H, CH_3O), 4.04 br.s (1H, NH), 4.49 d (1H, H^{3a}, *J* 8.2 Hz), 6.76–6.87 m (4H, H arom), 7.23–7.37 m (6H, H arom), 7.43–7.47 m (2H, H arom), 7.79–7.81 m (2H, H arom). ^{13}C NMR spectrum, δ_C, ppm: 13.8, 27.4 (Me), 55.0, 57.2, 57.9, 61.9, 67.5 ($C^{1,3a,6a}$, CH_3O, CH_2O), 75.1 (C^3), 113.8, 123.8, 126.7, 126.8, 126.9, 127.2, 127.4, 127.9, 143.2, 147.6, 158.9 (Ar), 172.8, 173.7, 174.6 (CO). Found, %: C 74.20; H 7.21; N 4.88. $C_{29}H_{28}N_2O_5 \cdot C_6H_{12}$. Calculated, %: C 73.92; H 7.09; N 4.93.

Methyl-(±)-(1*S*,3*aS*,6*aR*)-1-methyl-4,6-dioxo-3,3-diphenyl-5-(4-chlorophenyl)perhydropyrrolo-[3,4-*c*]pyrrole-1-carboxylate (XX). mp 196–197°C (ethyl acetate–hexane). IR spectrum ($CHCl_3$), ν, cm^{-1}: 3390 w, 3050 w, 2960, 1820, 1730, 1500, 1460, 1380, 1290, 1150, 1100. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.34 s (3H, CH_3), 3.57 d (1H, H^{6a}, *J* 7.9 Hz), 3.95 s (3H, CH_3O), 3.97 br.s (1H, NH), 4.50 d (1H, H^{3a}, *J* 7.9 Hz), 6.76–6.79 m (2H, H arom, *J* 8.3 Hz), 7.23–7.38 m (8H, H arom), 7.43–7.48 m (2H, H arom), 7.76–7.79 m (2H, H arom, *J* 7.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 27.3 (Me), 52.7, 57.0, 57.8, 67.5 ($C^{1,3a,6a}$, CH_3O), 75.0 (C^3), 126.7, 126.9, 127.0, 127.3, 128.0, 128.7, 129.5, 133.7, 143.0, 147.5 (Ar), 173.1, 173.2, 174.2 (CO). Found, %: C 68.56; H 4.92; N 5.71. $C_{27}H_{23}ClN_2O_4$. Calculated, %: C 68.28; H 4.88; N 5.90.

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