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Five-Membered 2.3-Dioxo Heterocycles: LXVIII.* Three Pathways in the Reaction of Methyl 3-Aroyl-1-aryl-4,5-dioxo-4,5-dihydro-1*H*-pyrrole-2-carboxylates with 3-Amino-5,5-dimethylcyclohex-2-en-1-one

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Abstract—Methyl 3-aroyl-1-aryl-4,5-dioxo-4,5-dihydro-1*H*-pyrrole-2-carboxylates reacted with 3-amino-5,5-dimethylcyclohex-2-en-1-one having no substituent on the nitrogen atom to give 3-aroyl-4-arylamino-6′,6′-dimethyl-6′,7′-dihydro-5*H*-spiro[furan-2,3′-indole]-2′,4′,5′(1′*H*,5′*H*)-triones or methyl 12-aroyl-11-aryl-9-hydroxy-5,5-dimethyl-3,10-dioxo-8,11-diazatricyclo[7.2.1.0^{2,7}]dodec-2(7)-ene-1-carboxylates. The latter underwent thermal recyclization to 3′-aroyl-1′-aryl-4′-hydroxy-6,6-dimethyl-6,7-dihydrospiro[indole-3,2′-pyrrole]-2,4,5′(1*H*,1′*H*,5*H*)-triones.

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Monocyclic 1*H*-pyrrole-2,3-diones are known to react with difunctional nucleophiles to form various five-, six-, and seven-membered aza heterocycles, as well as fused and spiro heterocyclic systems [2, 3]. We previously showed that methyl 3-aroyl-1-aryl-4,5-dioxo-4,5-dihydro-1*H*-pyrrole-2-carboxylates react with N-alkyl- and N-aryl-substituted acyclic, carbocyclic, and heterocyclic enamino ketones (ethyl 3-anilinobut-2-enoates [4], 3-alkylamino- and 3-arylamino-5,5-dimethylcyclohex-2-en-1-ones [5], and 1-methyl-3,4-dihydrobenzo[*f*]isoquinolines [6]) as with 1,3-C,N-binucleophiles via initial attack by the β-CH group in the enamino fragment of enamino ketone at C^2 of pyrroledione and subsequent closure of new pyrrole ring as a result of intramolecular attack by the NH group in the enamino fragment on the ester carbonyl carbon atom and elimination of methanol. These reactions lead to the formation of spiro heterocyclic systems such as 1,7-diazaspiro[4.4]nona-3,8-dienes [4], spiro[indole-3,2′-pyrroles] [5], and spiro[benzo[*h*]-pyrrolo[2,1-*a*]isoquinoline-2,2′-pyrroles] [6].

In continuation of our studies on chemical transformations of monocyclic 1*H*-pyrrole-2,3-diones by the action of difunctional nucleophiles, in the present work we examined reactions of methyl 3-aroyl-1-aryl-4,5-dioxo-4,5-dihydro-1*H*-pyrrole-2-carboxylates **Ia**–**Id** with 3-amino-5,5-dimethylcyclohex-2-en-1-one (**II**), which is a potential 1,3-C,N-binucleophile (cyclic enamino ketone). Pyrrolediones **Ia**–**Id** reacted with an equimolar amount of enamino ketone **II** on heating in boiling anhydrous benzene or chloroform over a period of 1–10 min (until intense red color typical of initial pyrrolediones disappeared) to give methyl 12-aroyl-11-aryl-9-hydroxy-5,5-dimethyl-3,10-dioxo-8,11-diazatricyclo[7.2.1.0^{2,7}]dodec-2(7)-ene-1-carboxylates **IIIa**–**IIIc**** and 3-aroyl-4-arylamino-6′,6′-dimethyl-6′,7′-dihydro-5*H*-spiro[furan-2,3′-indole]-2′,4′,5′(1′*H*,5*H*)-triones **IVa** and **IVb** (Scheme 1). Compounds **IIIa**–**IIIc** were isolated as colorless or light yellow high-melting crystalline substances that are readily soluble in DMSO and DMF, poorly soluble in other common organic solvents, and insoluble in water and saturated hydrocarbons. Compounds **IIIa**–**IIIc** showed a negative color test for enolic hydroxy goup with an alcoholic solution of $FeCl_3$.

The IR spectra of **IIIa**–**IIIc** contained absorption bands due to stretching vibrations of the NH group at 3330–3384 cm^{-1}, OH group at 3090–3130 cm^{-1} (broad

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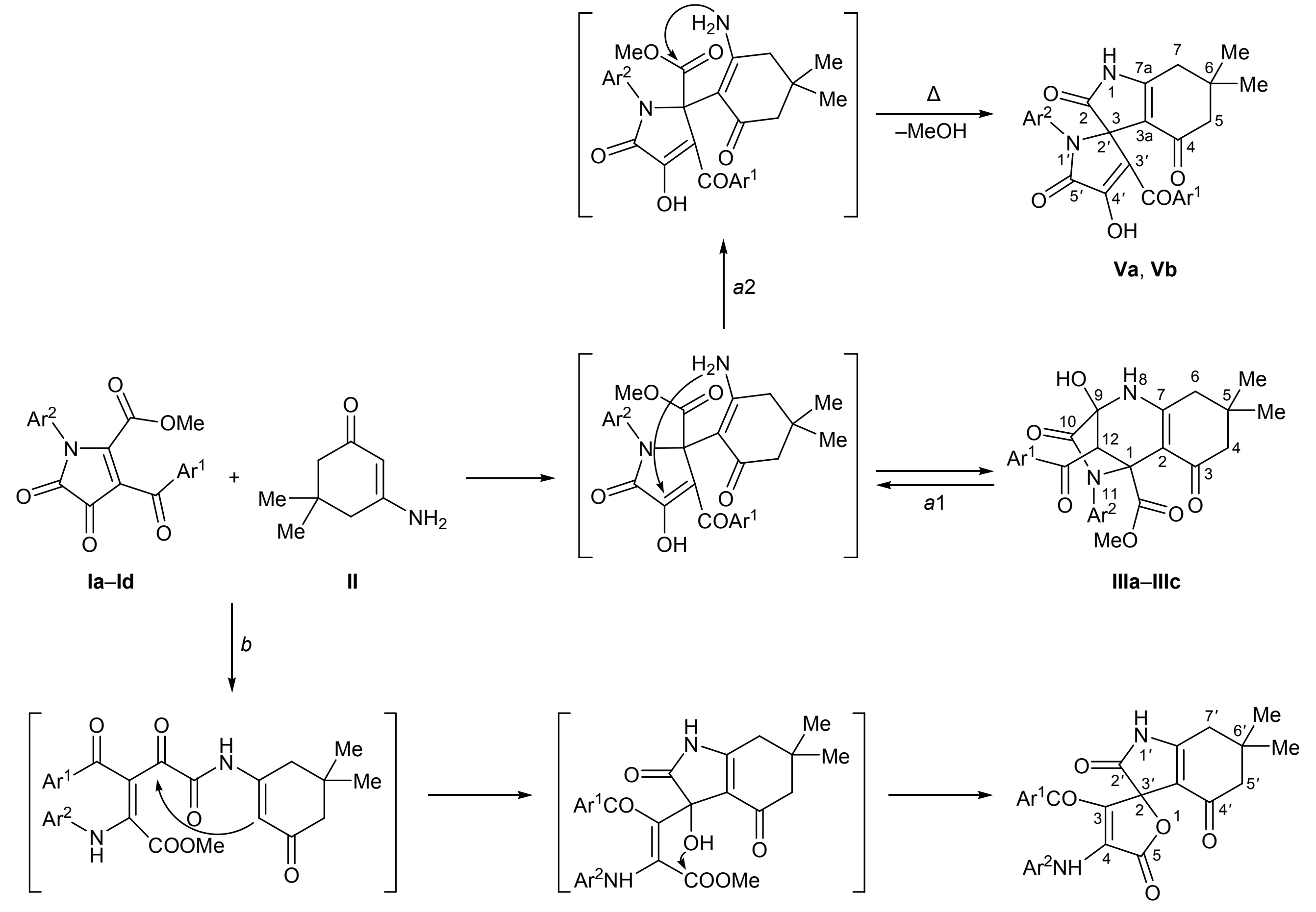
* For communication LXVII, see [1].

** For preliminary communication, see [7].

Scheme 1.



Ia–Id **II** **IIIa–IIIc** **Va**, **Vb** **IVa**, **IVb**

I, $Ar^1 = Ar^2 = Ph$ (**a**); $Ar^1 = Ph$, $Ar^2 = C_6H_4Me\text{-}4$ (**b**); $Ar^1 = Ph$, $Ar^2 = C_6H_4Cl\text{-}4$ (**c**); $Ar^1 = C_6H_4OEt\text{-}4$, $Ar^2 = C_6H_4Me\text{-}4$ (**d**); **III**, $Ar^1 = Ar^2 = Ph$ (**a**), $C_6H_4Me\text{-}4$ (**b**), $C_6H_4Cl\text{-}4$ (**c**); **IV**, $Ar^1 = Ph$, $Ar^2 = C_6H_4Cl\text{-}4$ (**a**); $Ar^1 = C_6H_4OEt\text{-}4$, $Ar^2 = C_6H_4Me\text{-}4$ (**b**); **V**, $Ar^1 = Ar^2 = Ph$ (**a**); $Ar^1 = Ph$, $Ar^2 = C_6H_4Me\text{-}4$ (**b**).

band), lactam carbonyl C^{10}=O at 1748–1756 cm^{-1}, ester carbonyl at 1726–1732 cm^{-1}, and enone carbonyl group C^3=O in the dimedone fragment and ketone (aroyl) carbonyl group in the region 1623–1665 cm^{-1}. In the ^1H NMR spectra of **IIIa**–**IIIc** we observed signals from protons in aromatic rings and substituents attached thereto, two singlets from protons in two nonequivalent methyl groups (δ 0.72–0.89 ppm), two doublets of doublets from protons in two nonequivalent methylene groups in the cyclohexene fragment (δ 1.90–2.30 ppm), a singlet from the methoxycarbonyl group (δ 3.78–3.79 ppm), a singlet from 12-H (δ 5.11–5.15 ppm), a singlet from the hydroxy proton (δ 6.71–6.81 ppm), and a singlet from the NH proton at δ 7.27–7.47 ppm. The spectral parameters of bridged compounds **IIIa**–**IIIc** are very similar to those of model 13-allyl-20-(4-bromobenzoyl)-12-hydroxy-16,16-dimethyl-3-phenyl-3,10,13-triazapentacyclo-$[10.7.1.0^{1,10}.0^{4,9}.0^{14,19}]$eicosa-4,6,8,14(19)-tetraene-2,11,18-trione whose structure was proved by X-ray analysis [8].

Compounds **IVa** and **IVb** are yellow high-melting crystalline substances; they are readily soluble in DMSO and DMF, difficultly soluble in other organic solvents, and insoluble in water and saturated hydrocarbons. They showed a negative color test for enolic hydroxy group with an alcoholic solution of iron(III) chloride. Compounds **IVa** and **IVb** displayed in the IR spectra absorption bands due to stretching vibrations of the NH group (a broad band in the region 3380–3200 cm^{-1}), lactone carbonyl group C^5=O in the furan ring and lactam carbonyl group $C^{2'}$=O in the indole fragment (1786–1730 cm^{-1}), and enone carbonyl group $C^{4'}$=O in the dimedone fragment and ketone carbonyl group in the aroyl fragment (1670–1630 cm^{-1}). The ^{1}H NMR spectra of **IVa** and **IVb** contained signals from protons in the aromatic rings and substituents attached thereto, two singlets from protons in two nonequivalent methyl groups (δ 0.78–1.05 ppm), two doublets of doublets from protons in two nonequivalent methylene groups in the cyclohexene fragment (δ 1.90–2.69 ppm), and two singlets from the NH protons in the lactam and secondary amine fragments at δ 8.97–9.30 and 11.24–11.35 ppm, respectively. The spectral parameters of spiro compounds **IVa** and **IVb** are very similar to those of model 4-arylamino-3-aroyl-1′-(4-methoxyphenyl)-6′,6′-dimethyl-2,2′,3′,4′,-5,5′,6′,7′-octahydro-1′*H*-spiro[furan-2,3′-indole]-2′,4′,5-trione whose structure was proved by X-ray analysis [9].

Bridged compounds **IIIa**–**IIIc** are likely to be formed via initial orbital-controlled [2, 3, 10–12] addition of the β-CH group in the enamino fragment of **II** at the C^2 carbon atom of dioxo ester **Ia**–**Ic** (path *a*) and subsequent attack by the amino group on C^4 (path *a*1; Scheme 1). No compounds analogous to **III** were detected previously in the reactions of pyrolediones **I** with N-substituted enamino ketones, presumably due to increased steric hindrances at the substituted nitrogen atom.

Path *b* leading to formation of compounds **IVa** and **IVb** involves initial charge-controlled [2, 3, 12] addition of the amino nitrogen atom in enamine **II** at the C^5 carbon atom in pyrrolediones **Ic** and **Id**. Next follows opening of the pyrrole ring at the N^1–C^5 bond and intramolecular closure of new pyrrole ring via nucleophilic addition of the β-CH group in the enamino fragment to the ketone carbonyl group. Hemiacetal hydroxy group thus formed attacks the ester carbonyl carbon atom, leading to closure of furan ring with elimination of methanol. Analogous reaction scheme was observed previously as side process [9].

On heating in boiling 1,2-dichloroethane over a period of 10–12 min, bridged compounds **IIIa** and **IIIb** underwent recyclization to 3′-aroyl-1′-aryl-4′-hydroxy-6,6-dimethyl-6,7-dihydrospiro[indole-3,2′-pyrrole]-2,4,5′(1*H*,1′*H*,5*H*)-triones **Va** and **Vb** (Scheme 1). Spiro compounds **Va** and **Vb** were isolated as colorless high-melting crystalline substances that are readily soluble in DMSO and DMF, poorly soluble in other organic solvents, and insoluble in water and saturated hydrocarbons; they showed a positive color test for enolic hydroxy group with an alcoholic solution of iron(III) chloride.

In the IR spectra of **Va** and **Vb**, absorption bands due to stretching vibrations of the NH and OH groups were located in the region 3495–3090 cm^{-1}, lactam carbonyl group C^2=O in the indole fragment and C$^{5'}$=O group in the pyrrole fragment gave rise to absorption band at 1748–1709 cm^{-1}, and stretching vibrations of the enone (C^4=O) and benzoyl carbonyl groups had a frequency of 1665–1625 cm^{-1}. The ^{1}H NMR spectra of **Va** and **Vb** contained signals from protons in the aromatic rings and substituents attached thereto, two singlets from protons in two nonequivalent methyl groups (δ 0.60–0.86 ppm), two doublets of doublets from protons in two nonequivalent methylene groups in the cyclohexene fragment (δ 1.95–2.06 ppm), and two singlets from the NH and OH protons at δ 11.08–11.14 and 12.42–12.45 ppm, respectively. The spectral parameters of compounds **Va** and **Vb** are very similar to those of model 3′-benzoyl-1-cyclohexyl-4′-hydroxy-6,6-dimethyl-1′-phenyl-1′,2,3,4,5,5′,6,7-octahydro-1*H*,2′*H*-spiro[indole-3,2′-pyrrole]-2,4,5′-trione [5] and 3′-benzoyl-4′-hydroxy-11,11-dimethyl-1′-phenyl-1,2,2′,5′,10,11-hexahydrospiro[benzo[*h*]pyrrolo[2,1-*a*]isoquinoline-2,2′-pyrrole]-1,5′-dione [6] whose structure was proved by X-ray analysis.

Presumably, compounds **IIIa** and **IIIb** in solution undergo thermally induced cleavage of the hemiaminal NH–C(OH) bond and closure of new pyrrole ring via intramolecular attack by the primary amino group in the enamino fragment on the ester carbonyl group (path *a*2) with elimination of methanol (Scheme 1). Analogous scheme of reaction of pyrrolediones like **I** with N-alkyl- and N-aryl-substituted acyclic, cyclic, and heterocyclic enamino ketones was observed by us most frequently [4–6].

Thus we have revealed three different pathways in the reactions of methyl 3-aroyl-1-aryl-4,5-dioxo-4,5-dihydro-1*H*-pyrrole-2-carboxylates with 3-amino-5,5-dimethylcyclohex-2-en-1-one.

EXPERIMENTAL

The IR spectra were recorded on FSM-1201 and Bruker IFS-66 spectrometers from samples dispersed in mineral oil. The ^{1}H and ^{13}C NMR spectra were measured on a Bruker AM-400 spectrometer (at 400 MHz for ^{1}H) from solutions in DMSO-d_6 using tetramethylsilane as internal reference. The purity of the products was checked by TLC on Silufol plates using ethyl acetate or ethyl acetate–benzene (1:5) as eluent; chromatograms were developed by treatment with iodine vapor.

Methyl 12-benzoyl-9-hydroxy-5,5-dimethyl-3,10-dioxo-11-phenyl-8,11-diazatricyclo[7.2.1.0^{2,7}]dodec-2(7)-ene-1-carboxylate (IIIa). A solution of 1.0 mmol of compound **Ia** and 1.0 mmol of enamine **II** in 10 ml of anhydrous benzene was heated for 1 min under reflux. The mixture was cooled, and the precipitate was filtered off. Yield 3.75 g (79%), mp 178–180°C (decomp., from ethyl acetate). IR spectrum, ν, cm^{-1}: 3384 (NH), 3090 br (OH), 1751 (C^{10}=O), 1732 (COOMe), 1663, 1630 (C^3=O, COPh). ^{1}H NMR spectrum, δ, ppm: 0.73 s and 0.86 s (3H each, Me), 1.91 d.d and 1.98 d.d (1H each, 6-H, *J* = 16.1 Hz), 2.18 d.d and 2.29 d.d (1H each, 4-H, *J* = 16.5 Hz), 3.79 s (3H, OMe), 5.15 s (1H, 12-H), 6.75 s (1H, OH), 7.44 s (1H, NH), 7.12–7.82 m (10H, H$_{\text{arom}}$). Found, %: C 68.38; H 5.50; N 5.92. $C_{27}H_{26}N_2O_6$. Calculated, %: C 68.34; H 5.52; N 5.90.

Compounds **IIIb** and **IIIc** were synthesized in a similar way.

Methyl 12-benzoyl-9-hydroxy-5,5-dimethyl-11-(4-methylphenyl)-3,10-dioxo-8,11-diazatricyclo[7.2.1.0^{2,7}]dodec-2(7)-ene-1-carboxylate (IIIb). Yield 37%, mp 173–175°C (decomp., from ethyl acetate). IR spectrum, ν, cm^{-1}: 3365 (NH), 3110 br (OH), 1748 (C^{10}=O), 1730 (COOMe), 1665, 1626 (C^3=O, COPh). ^{1}H NMR spectrum, δ, ppm: 0.73 s and 0.89 s (3H each, Me), 1.92 d.d and 1.99 d.d (1H each, 6-H, *J* = 16.0 Hz), 2.18 d.d and 2.30 d.d (1H each, 4-H, *J* = 16.4 Hz), 2.33 s (3H, C_6H_4**Me**), 3.79 s (3H, OMe), 5.14 s (1H, 12-H), 6.71 s (1H, OH), 7.27 s (1H, NH), 6.98–7.82 m (9H, H$_{\text{arom}}$). ^{13}C NMR spectrum, δ$_\text{C}$, ppm: 20.60 ($\mathbf{C}H_3C_6H_4$), 27.37 (5-CH$_3$), 32.18 (C^5), 42.04 (C^6), 49.55 (C^4), 54.09 (CH$_3$O), 57.30 (C^{12}), 59.65 (C^1), 100.62 (C^9), 115.85 (C^2), 125.33–138.23 (C$_{\text{arom}}$), 158.84 (C^7), 164.70 (MeO**C**O), 167.82 (C^{10}), 187.66 (Ph**C**O), 195.46 (C^3). Found, %: C 68.99; H 5.66; N 5.92. $C_{28}H_{28}N_2O_6$. Calculated, %: C 68.84; H 5.78; N 5.73.

Methyl 12-benzoyl-11-(4-chlorophenyl)-9-hydroxy-5,5-dimethyl-3,10-dioxo-8,11-diazatricyclo[7.2.1.0^{2,7}]dodec-2(7)-ene-1-carboxylate (IIIc). Yield 25%, mp 182–184°C (decomp., from methanol). IR spectrum, ν, cm^{-1}: 3330 (NH), 3130 br (OH), 1756 (C^{10}=O), 1726 (COOMe), 1659, 1623 (C^3=O, COPh). ^{1}H NMR spectrum, δ, ppm: 0.72 s and 0.85 s (3H each, Me), 1.90 d.d and 1.98 d.d (1H each, 6-H, *J* = 16.3 Hz), 2.18 d.d and 2.30 d.d (1H each, 4-H, *J* = 16.7 Hz), 3.78 s (3H, OMe), 5.11 s (1H, 12-H), 6.81 s (1H, OH), 7.47 s (1H, NH), 7.16–7.82 m (9H, H$_{\text{arom}}$). Found, %: C 63.77; H 4.91; Cl 6.99; N 5.54. $C_{27}H_{25}ClN_2O_6$. Calculated, %: C 63.72; H 4.95; Cl 6.97; N 5.50.

3-Benzoyl-4-(4-chlorophenylamino)-6′,6′-dimethyl-6′,7′-dihydro-5*H*-spiro[furan-2,3′-indole]-2′,4′,5(1′*H*,5′*H*)-trione (IVa). A solution of 1.0 mmol of compound **Ic** and 1.0 mmol of enamino ketone **II** in 10 ml of anhydrous benzene was heated for 10 min under reflux. The mixture was cooled, and the precipitate was filtered off. Yield 54%, mp 182–185°C (decomp., from ethyl acetate). IR spectrum, ν, cm^{-1}: 3200 br (NH), 1786 (C^5=O), 1765 (C$^{2'}$=O), 1638 (C$^{4'}$=O, COPh). ^{1}H NMR spectrum, δ, ppm: 0.80 s and 1.05 s (3H each, Me), 1.93 d.d and 2.27 d.d (1H each, 7-H, *J* = 16.0 Hz), 2.41 d.d and 2.69 d.d (1H each, 5-H, *J* = 18.1 Hz), 6.65–7.38 m (9H, H$_{\text{arom}}$), 9.30 s (1H, 1′-H), 11.35 s (1H, 4-NH). Found, %: C 65.49; H 4.24; Cl 7.61; N 5.68. $C_{26}H_{21}ClN_2O_5$. Calculated, %: C 65.48; H 4.44; Cl 7.43; N 5.87.

6′,6′-Dimethyl-4-(4-methylphenylamino)-3-(4-ethoxybenzoyl)-6′,7′-dihydro-5*H*-spiro[furan-2,3′-indole]-2′,4′,5(1′*H*,5′*H*)-trione (IVb) was synthesized in a similar way. Yield 67%, mp 198–200°C (decomp., from ethyl acetate). IR spectrum, ν, cm^{-1}: 3380 (NH), 1730 (C^5=O, C$^{2'}$=O), 1670, 1630 (C$^{4'}$=O, COAr). ^{1}H NMR spectrum, δ, ppm: 0.78 s and 1.04 s (3H each, Me), 1.28 t (3H, $C\mathbf{H}_3CH_2$, *J* = 7.0 Hz), 1.90 d.d and 2.24 d.d (1H each, 7-H, *J* = 16.1 Hz), 2.06 s (3H, $C_6H_4C\mathbf{H}_3$), 2.39 d.d and 2.66 d.d (1H each, 5-H, *J* = 18.4 Hz), 4.00 q (2H, OCH$_2$, *J* = 7.1 Hz), 6.57–8.21 m (8H, H$_{\text{arom}}$), 8.97 s (1H, 1′-H), 11.24 s (1H, 4-NH). ^{13}C NMR spectrum, δ$_\text{C}$, ppm: 14.41 ($\mathbf{C}H_3$), 20.56 ($\mathbf{C}H_3C_6H_4$), 27.34 (5-CH$_3$), 32.17 (C$^{6'}$), 42.00 (C$^{7'}$), 49.55 (C$^{5'}$), 63.49 (CH$_2$O), 77.55 (C^2, C$^{3'}$), 113.85 (C$^{3a'}$), 126.48–131.40 (C$_{\text{arom}}$), 130.71 (C^3), 138.15 (C^4), 158.77 (C^5), 164.66 (C$^{2'}$), 167.72 (C$^{7a'}$), 186.16 (C$_6$H$_4$CO), 195.44 (C$^{4'}$). Found, %: C 69.53; H 5.64; N 5.43. $C_{29}H_{28}N_2O_6$. Calculated, %: C 69.59; H 5.64; N 5.60.

3′-Benzoyl-4′-hydroxy-6,6-dimethyl-1′-phenyl-6,7-dihydrospiro[indole-3,2′-pyrrole]-2,4,5′(1*H*,1′*H*,5*H*)-trione (Va). A solution of 1.0 mmol of compound **Ia** and 1.0 mmol of enamine **II** in 10 ml of anhydrous benzene was heated for 10 min under reflux. The mixture was cooled, and the precipitate was filtered off. Yield 54%, mp 185–187°C (decomp., from ethyl acetate). IR spectrum, ν, cm^{-1}: 3383, 3247 br (NH, OH), 1732, 1715 (C^2=O, C$^{5'}$=O), 1665, 1628 (C^4=O, COPh). ^{1}H NMR spectrum, δ, ppm: 0.60 s and 0.86 s (3H each, Me), 1.94 d.d and 2.05 d.d (1H each, 7-H, *J* = 16.1 Hz), 2.21 d.d and 2.42 d.d (1H each, 5-H, *J* = 18.0 Hz), 7.03–7.87 m (9H, H$_{\text{arom}}$), 11.14 s (1H, NH), 12.42 s (1H, OH). Found, %: C 70.41; H 5.06; N 6.19. $C_{26}H_{22}N_2O_5$. Calculated, %: C 70.58; H 5.01; N 6.33.

3′-Benzoyl-4′-hydroxy-6,6-dimethyl-1′-(4-methylphenyl)-6,7-dihydrospiro[indole-3,2′-pyrrole]-2,4,5′(1*H*,1′*H*,5*H*)-trione (Vb). Yield 67%, mp 253–255°C (decomp., ethyl acetate). IR spectrum, ν, cm^{-1}: 3495, 3090 br (NH, OH), 1748, 1709 (C^2=O, $C^{5'}$=O), 1662, 1625 (C^4=O, COPh). ^{1}H NMR spectrum, δ, ppm: 0.66 s and 0.86 s (3H each, Me), 1.95 d.d and 2.06 d.d (1H each, 7-H, J = 16.1 Hz), 2.29 s (3H, $C_6H_4C\mathbf{H}_3$), 2.21 d.d and 2.41 d.d (2H, 5-H, J = 18.4 Hz), 6.89–7.23 m (4H, C_6H_4), 7.46–7.66 m (5H, C_6H_5), 11.08 s (1H, NH), 12.45 s (1H, OH). Found, %: C 71.16; H 5.10; N 6.16. $C_{27}H_{24}N_2O_5$. Calculated, %: C 71.04; H 5.30; N 6.14.

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