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Five-Membered 2,3-Dioxoheterocycles: XCVI.* Reactions of 3-Aroylpyrrolo[1,2-*a*]quinoxaline-1,2,4(5*H*)-triones with Substituted 1,3,3-trimethyl-2-azaspiro[4.5]dec-1-enes

V. V. Konovalova[a], K. A. Galata[a], Yu. V. Shklyaev[a], and A. N. Maslivets[b]

[a]*Institute of Technical Chemistry, Ural Branch, Russian Academy of Sciences,Perm, Russia*
[b]*Perm State National Research University, Perm, 614990 Russia*
e-mail: koh2@psu.ru

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Abstract—3-Aroylpyrrolo[1,2-*a*]quinoxaline-1,2,4(5*H*)-triones react with 1,3,3,7,9-pentamethyl-2-azaspiro-[4.5]deca-1,6,9-trien-8-one and 2',5',5'-trimethyl-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-4-one providing 3-aroyl-2-hydroxy-3a-{(3,3,7,9-tetramethyl-8-oxo-2-azaspiro[4.5]deca-1,6,9-trien-1-yl)methyl}pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-diones and 3-aroyl-2-hydroxy-3a-{(5',5'-dimethyl-4-oxo-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-2'-yl)methyl}pyrrolo[1,2-*a*]-quinoxaline-1,4(3a*H*,5*H*)-diones.

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Recyclization and heterocyclization reactions under the action of binucleophilic reagents of substituted 4-acyl-1*H*-pyrrole-2,3-diones, in particular, those fused with azaheterocycles at the [*e*] side, are a convenient method of building up a wide range of fused heterocycles and spirobisheterocyclic systems [2, 3].

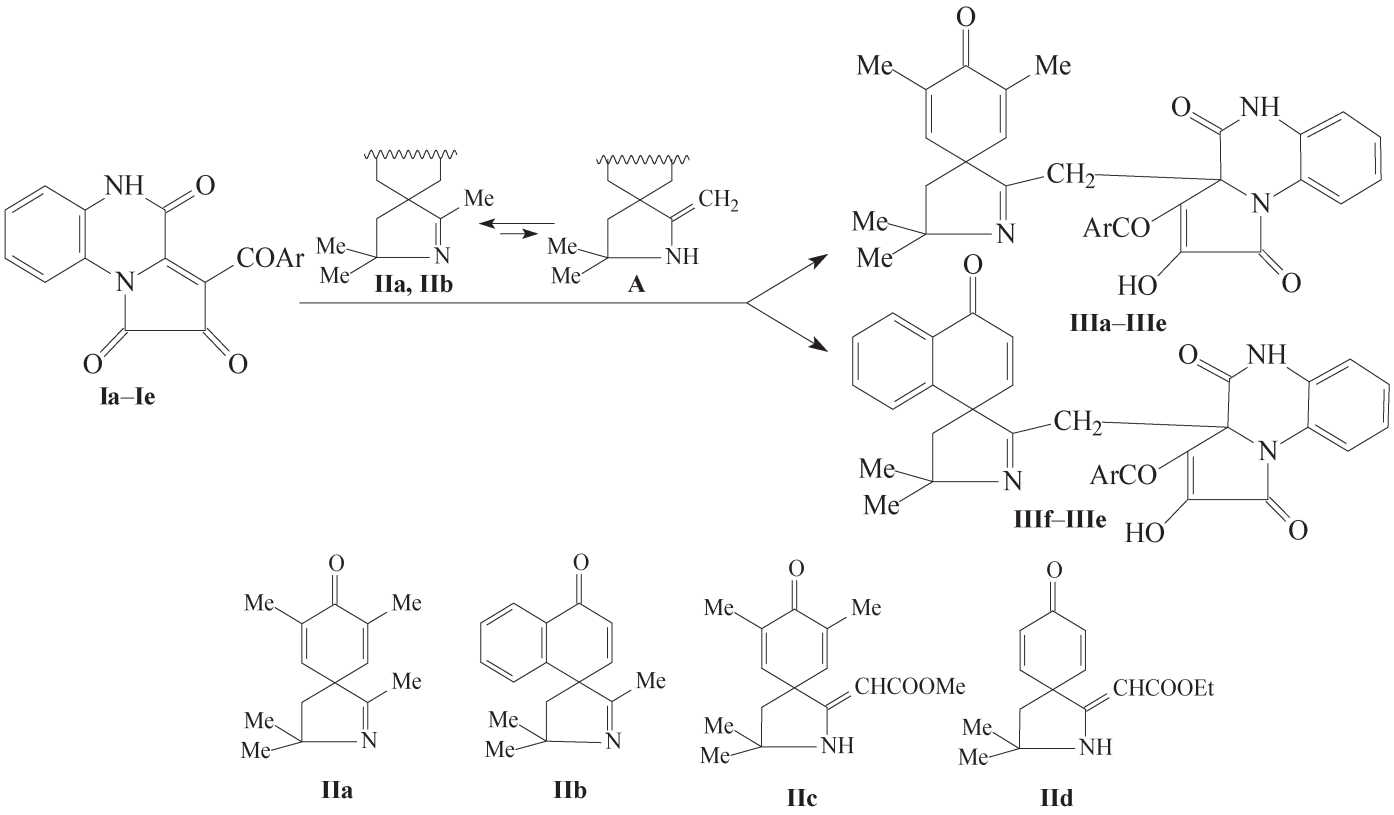
We showed formerly that 4-acyl-1*H*-pyrrole-2,3-diones fused with 1,4-benzoxazinone fragment (3-aroylpyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones) reacted with a spiroheterocyclic enamine (2',5',5'-trimethyl-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-4-one) as with a 1,3-CH,NH-binucleophile along the scheme with a successive attack of groups β-CH and NH on the enamino fragment of the enamino form of the spironaphthalenepyrrolone at the carbon atoms in the positions *3a* and *4* of pyrrolobenzoxazinetriones, respectively, with the cleavage of the C^4–O^5 bond of the oxazine ring of pyrrolobenzoxazinetriones and the formation of substituted dispiro[azol-2,2'-(azolo[1,2-*a*]azol)-7',1''-(dihydronaphthalenes)] [4]. In extension of the investigation of this process we studied the reaction of 4-acyl-1*H*-pyrrole-2,3-diones fused with 1,4-quinoxalinone fragment {3-aroylpyrrolo[1,2-*a*]quinoxaline-1,2,4(5*H*)-triones} with spiropyrrolines.

The reactions of 3-aroylpyrrolo[1,2-*a*]quinoxaline-1,2,4(5*H*)-triones **Ia–Ie** with substituted 1,3,3-trimethyl-2-azaspiro[4.5]dec-1-enes **IIa, IIb** [5, 6] in a ratio 1 : 1 at boiling in anhydrous acetonitrile for 10–30 min (till the disappearance of the bright violet color of initial pyrroloquinoxalinetriones) afforded in high yields the products of addition of the activated β-CH group of the tautomeric enamino form **A** of compounds **IIa, IIb** to the carbon atom in the position *3a* of pyrroloquinoxalinetriones **Ia–Ie**, 3-aroyl-2-hydroxy-3a-{(3,3,7,9-tetramethyl-8-oxo-2-azaspiro[4.5]deca-1,6,9-trien-1-yl)methyl}- **IIIa–IIIe** and 3-aroyl-2-hydroxy-3a-{(5',5'-dimethyl-4-oxo-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-2'-yl)methyl}pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-diones **IIIf–IIIj**.

Compounds **IIIa–IIIj** are colorless crystalline substances of high melting points (melting with decomposition), readily soluble in DMSO and DMF, sparingly soluble in alcohols and haloalkanes, insoluble in water and alkanes, showing a positive test (cherry-red color) for the presence of enol hydroxyl with the alcohol solution of iron(III) chloride.

IR spectra of compounds **IIIa–IIIj** contain the absorption of the stretching vibrations of the enol OH group as a broad band in the region 3216–3230 cm^{-1}, a broad band of the NH group at 3155–3169 cm^{-1}, bands of two lactam carbonyl groups C^1=O and C^4=O in the

* For communication XCV, see [1].



Ar = Ph (**a**, **f**), 4-MeC_6H_4 (**b**, **g**), 4-$MeOC_6H_4$ (**c**, **h**), 4-ClC_6H_4 (**d**, **i**), 4-BrC_6H_4 (**e**, **j**).

region 1689–1692 cm^{-1}, of aroyl carbonyl group, of the carbonyl group of the cyclohexadiene fragment C^8=O (for compounds **IIIa–IIIe**) or of the naphthalenone fragment C^4=O (for compounds **IIIf–IIIj**) in the region 1641–1659 cm^{-1}.

In the ^{1}H NMRspectra of compounds **IIIa–IIIj** in DMSO-d_6 alongside with the proton signals from the aromatic rings and the groups attached to them two singlets are present of the protons of two methyl groups in the position *3* (for compounds **IIIa–IIIe**) or *5′* (for compounds **IIIf–IIIj**) of the pyrrolidine fragment in the region 1.16–1.33 and 1.22–1.40 ppm, a doublet of doublets of the C^4H_2 group of the pyrrolidine fragment (for compounds **IIIa–IIIe**) in the region 1.83–2.12 ppm and the $C^{4'}H_2$ group of the pyrrolidine fragment (2.03–2.31 ppm for compounds **IIIf–IIIj**), a doublet of doublets of the CH_2 group in the region 3.32–3.37 (for compounds **IIIa–IIIe**) and 1.82–3.17 ppm (for compounds **IIIf–IIIj**), a singlet from the proton of NH group in the region 10.67–10.84 ppm, and a broadened singlet of the proton of enol OH group in the region 11.41–11.80 ppm.

The spiropyrroline fragment in the synthesized compounds **IIIa–IIIj** exists in the imine form, although formerly only its presence in the enamine form [7], has been observed, apparently because of the lack in compounds **IIIa–IIIj** of intramolecular hydrogen bonds, which may stabilize the enamine form.

Further intramolecular cyclization of substituted 3a-(pyrrolylmethyl)pyrroloquinoxalinediones **IIIa–IIIj**, which has been observed for 3a-(pyrrolylmethyl)pyrrolobenzoxazinediones [4] does not occur with the former compounds evidently due to the decreased electrophilicity of the carbonyl carbon atom of the C^4=O group as compared to the electrophilicity of the ester carbon atom.

We attempted to modify the structure of initial 1,3,3-trimethyl-2-azaspiro-[4.5]dec-1-enes in order to change the regiodirection of their reaction with pyrroloquinoxalinetriones. It turned out that the reaction of 3-aroylpyrrolo[1,2-*a*]quinoxaline-1,2,4(5*H*)-triones **Ia–Ie** with 1,3,3-trimethyl-2-azaspiro[4.5]dec-1-enes **IIc**, **IId** containing an additional ester group under analogous reaction conditions did not occur due to increased steric hindrances at the reaction site caused by the additional functional group.

EXPERIMENTAL

IR spectra were recorded on an IR Fourier spectrophotometer IFS 66 (Bruker) from mulls in mineral oil. ^{1}H and ^{13}C NMRspectra were registered on a spectrometer Mercury-300BB (operating frequency 300 MHz) in DMSO-d_6, internal reference HMDS. The homogeneity of compounds synthesized was confirmed by TLC on Sorbfil plates, eluents ethyl acetate–benzene, 1 : 5, ethyl acetate; spots visualized with 0.5% chloranil solution in toluene.

3-Benzoyl-2-hydroxy-3a-{(3,3,7,9-tetramethyl-8-oxo-2-azaspiro[4.5]deca-1,6,9-trien-1-yl)methyl}pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIIa). A solution of 1.0 mmol of reagent **Ia** and 1.0 mmol of spirocompound **IIa** in 20 mL of anhydrous acetonitrile was boiled for 20 min, cooled, the separated precipitate was filtered off. Yield 97%, mp 242–243°C (ethyl acetate). IR spectrum, cm^{-1}: 3220 br (OH), 3161 br (NH), 1691 (C^1=O, C^4=O), 1643 (COPh, C^8=O$_{cyclohex.}$). ^{1}H NMRspectrum, δ, ppm: 1.16 s, 1.23 s (6H, 2Me), 1.47 s (3H, Me), 1.83 s (3H, Me), 1.83, 2.09 d.d (2H, C^{4}H$_2$, J 16.5 Hz), 3.33, 3.36 d.d (2H, CH$_2$, J 6.3 Hz), 6.00 s (1H, H^6), 6.50 s (1H, H^{10}), 7.04–7.83 group of signals (9H, Ph, C$_6$H$_4$), 10.79 s (1H, NH), 11.57 br.s (1H, OH). Found, %: C 71.61; H 5.58; N 7.66. $C_{32}H_{29}N_3O_5$. Calculated, %: C 71.76; H 5.46; N 7.85.

Compounds **IIIb–IIIj** were similarly obtained.

2-Hydroxy-3a-{(3,3,7,9-tetramethyl-8-oxo-2-azaspiro[4.5]deca-1,6,9-trien-1-yl)methyl}-3-(4-toluoyl)pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIIb). Yield 96%, mp 187–188°C (dichloroethane). IR spectrum, cm^{-1}: 3221 br (OH), 3162 br (NH), 1690 (C^1=O, C^4=O), 1651 (COC$_6$H$_4$Me-4, C^8=O$_{cyclohex.}$). ^{1}H NMRspectrum, δ, ppm: 1.16 s, 1.23 s (6H, 2Me), 1.49 s (3H, Me), 1.83 s (3H, Me), 1.83, 2.12 d.d (2H, C^{4}H$_2$, J 16.8 Hz), 2.37 s (3H, 4-MeC$_6$H$_4$), 3.32, 3.37 d.d (2H, CH$_2$, J 6.4 Hz), 6.00 s (1H, H^6), 6.49 s (1H, H^{10}), 7.04–7.83 group of signals (8H, 2C$_6$H$_4$), 10.75 s (1H, NH), 11.41 br.s (1H, OH). ^{13}C NMRspectrum, δ, ppm: 15.72, 15.89 (7-Me, 9-Me), 21.24 (4-MeC$_6$H$_4$), 29.92 (CH$_2$), 30.42, 30.72 (C^{3}Me$_2$), 45.53 (2H, C^{4}H$_2$), 63.80 (C^{3}Me$_2$), 65.08 (C^{3a}), 74.59 (C^5$_{spiro}$), 115.84 (C^{3}COAr), 120.90–135.81 (C^{Ar}), 134.40, 134.74 (C^{7,9}$_{cyclohex.}$), 145.07, 145.73 (C^{6}H, C^{10}H$_{cyclohex.}$), 151.62 (C^{2}OH), 162.76 (C^1), 163.85, 164.96 (C^1=O, C^4=O), 185.41 (C^8$_{cyclohex.}$), 190.68 (COAr). Found, %: C 72.10; H 5.89; N 7.47. $C_{33}H_{31}N_3O_5$. Calculated, %: C 72.11; H 5.69; N 7.65.

2-Hydroxy-3-(4-methoxybenzoyl)-3a-{(3,3,7,9-tetramethyl-8-oxo-2-azaspiro[4.5]deca-1,6,9-trien-1-yl)methyl}pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIIc). Yield 92%, mp 186–187°C (ethyl acetate). IR spectrum, cm^{-1}: 3229 br (OH), 3168 br (NH), 1691 (C^1=O, C^4=O), 1648 (COC$_6$H$_4$OMe-4, C^8=O$_{cyclohex.}$). ^{1}H NMRspectrum, δ, ppm: 1.20 s, 1.27 s (6H, 2Me), 1.52 s (3H, Me), 1.87 s (3H, Me), 1.87, 2.12 d.d (2H, C^{4}H$_2$, J 16.8 Hz), 3.34, 3.37 d.d (2H, CH$_2$, J 6.3 Hz), 3.41 s (3H, 4-MeOC$_6$H$_4$), 6.05 s (1H, H^6), 6.54 s (1H, H^{10}), 7.08–7.87 group of signals (8H, 2C$_6$H$_4$), 10.84 s (1H, NH), 11.80 br.s (1H, OH). Found, %: C 70.01; H 5.52; N 7.26. $C_{33}H_{31}N_3O_6$. Calculated, %: C 70.07; H 5.52; N 7.43.

2-Hydroxy-3a-{(3,3,7,9-tetramethyl-8-oxo-2-azaspiro[4.5]deca-1,6,9-trien-1-yl)methyl}-3-(4-chlorobenzoyl)pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIId). Yield 96%, mp 260–261°C (ethyl acetate). IR spectrum, cm^{-1}: 3230 br (OH), 3168 br (NH), 1690 (C^1=O, C^4=O), 1647 (COC$_6$H$_4$Cl-4, C^8=O$_{cyclohex.}$). ^{1}H NMR spectrum, δ, ppm: 1.20 s, 1.27 s (6H, 2Me), 1.51 s (3H, Me), 1.87 s (3H, Me), 1.87, 2.12 d.d (2H, C^{4}H$_2$, *J* 16.7 Hz), 3.34, 3.37 d.d (2H, CH$_2$, J 6.3 Hz), 6.05 s (1H, H^6), 6.53 s (1H, H^{10}), 7.08–7.87 group of signals (8H, 2C$_6$H$_4$), 10.83 s (1H, NH), 11.74 br.s (1H, OH). Found, %: C 67.31; H 5.00; N 7.23. $C_{32}H_{28}ClN_3O_5$. Calculated, %: C 67.42; H 4.95; N 7.37.

3-(4-Bromobenzoyl)-2-hydroxy-3a-{(3,3,7,9-tetramethyl-8-oxo-2-azaspiro[4.5]deca-1,6,9-trien-1-yl)methyl}pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIIe). Yield 94%, mp 272–273°C (dichloroethane). IR spectrum, cm^{-1}: 3229 br (OH), 3169 br (NH), 1692 (C^1=O, C^4=O), 1641 (COC$_6$H$_4$Br-4, C^8=O$_{cyclohex.}$). ^{1}H NMRspectrum, δ, ppm: 1.16 s, 1.22 s (6H, 2Me), 1.47 s (3H, Me), 1.82 s (3H, Me), 1.83, 2.12 d.d (2H, C^{4}H$_2$, J 16.7 Hz), 3.32, 3.37 d.d (2H, CH$_2$, J 6.4 Hz), 6.01 s (1H, H^6), 6.48 s (1H, H^{10}), 7.04–7.82 group of signals (8H, 2C$_6$H$_4$), 10.77 s (1H, NH), 11.71 br.s (1H, OH). Found, %: C 62.43; H 4.79; N 6.82. $C_{32}H_{28}BrN_3O_5$. Calculated, %: C 62.55; H 4.59; N 6.84.

3-Benzoyl-2-hydroxy-3a-{(5',5'-dimethyl-4-oxo-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-2'-yl)methyl}pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIIe). Yield 96%, mp 236–237°C (dichloroethane). IR spectrum, cm^{-1}: 3219 br (OH), 3161 br (NH), 1689 (C^1=O, C^4=O), 1653 (COPh, C^4=O$_{naphth.}$). ^{1}H NMRspectrum, δ, ppm: 1.29 s, 1.36 s (6H, 2Me), 2.04, 2.27 d.d (2H, C$^{4'}$H$_2$, *J* 14.3 Hz), 1.83, 3.16 d.d (2H, CH$_2$, *J* 16.7 Hz),

5.76 s (1H, $C^3H_{naphth.}$), 6.28 s (1H, $C^2H_{naphth.}$), 7.01–7.98 group of signals (13H, Ph, $2C_6H_4$), 10.70 s (1H, NH), 11.54 br.s (1H, OH). Found, %: C 73.16; H 4.96; N 7.53. $C_{34}H_{27}N_3O_5$. Calculated, %: C 73.24; H 4.88; N 7.54.

2-Hydroxy-3a-{(5',5'-dimethyl-4-oxo-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-2'-yl)methyl}-3-(4-tolyl)pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIIg). Yield 97%, mp 220–222°C (dichloroethane). IR spectrum, cm^{-1}: 3223 br (OH), 3158 br (NH), 1690 (C^1=O, C^4=O), 1659 (COC_6H_4Me-4, C^4=O_{naphth}). ^{1}H NMRspectrum, δ, ppm: 1.29 s, 1.35 s (6H, 2Me), 2.03, 2.26 d.d (2H, $C^{4'}H_2$, *J* 14.0 Hz), 2.37 s (3H, 4-MeC_6H_4), 1.83, 3.16 d.d (2H, CH_2, *J* 17.0 Hz), 5.76 s (1H, $C^3H_{naphth.}$), 6.27 s (1H, $C^2H_{naphth.}$), 7.00–7.97 group of signals (12H, $3C_6H_4$), 10.67 s (1H, NH), 11.41 br.s (1H, OH). Found, %: C 73.53; H 5.22; N 7.24. $C_{35}H_{29}N_3O_5$. Calculated, %: C 73.54; H 5.11; N 7.35.

2-Hydroxy-3a-{(5',5'-dimethyl-4-oxo-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-2'-yl)methyl}-3-(4-methoxybenzoyl)pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIIh). Yield 91%, mp 233–234°C (dichloroethane). IR spectrum, cm^{-1}: 3216 br (OH), 3155 br (NH), 1691 (C^1=O, C^4=O), 1655 (COC_6H_4OMe-4, C^4=O_{naphth}). ^{1}H NMRspectrum, δ, ppm: 1.33 s, 1.40 s (6H, 2Me), 2.08, 2.31 d.d (2H, $C^{4'}H_2$, *J* 14.3 Hz), 1.85, 3.17 d.d (2H, CH_2, *J* 16.5 Hz), 3.39 c (3H, 4-$MeOC_6H_4$), 5.80 s (1H, $C^3H_{naphth.}$), 6.33 s (1H, $C^2H_{naphth.}$), 7.05–8.01 group of signals (12H, $3C_6H_4$), 10.77 s (1H, NH), 11.78 br.s (1H, OH). Found, %: C 71.44; H 5.05; N 7.05. $C_{35}H_{29}N_3O_6$. Calculated, %: C 71.54; H 4.97; N 7.15.

2-Hydroxy-3a-{(5',5'-dimethyl-4-oxo-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-2'-yl)methyl}-3-(4-chlorobenzoyl)pyrrolo[1,2-*a*]quinoxaline-1,4(3a*H*,5*H*)-dione (IIIi). Yield 92%, mp 233–234°C (dichloroethane). IR spectrum, cm^{-1}: 3217 br (OH), 3156 br (NH), 1690 (C^1=O, C^4=O), 1657 (COC_6H_4Cl-4, C^4=$O_{naphth.}$). ^{1}H NMRspectrum, δ, ppm: 1.28 s, 1.35 s (6H, 2Me), 2.03, 2.26 d.d (2H, $C^{4'}H_2$, *J* 13.8 Hz), 1.82, 3.13 d.d (2H, CH_2, *J* 16.5 Hz), 5.76 s (1H, $C^3H_{naphth.}$), 6.29 s (1H, $C^2H_{naphth.}$), 7.01–7.97 group of signals (12H, $3C_6H_4$), 10.71 c (1H, NH), 11.71 br.s (1H, OH). Found, %: C 68.91; H 4.51; N 6.99. $C_{34}H_{26}ClN_3O_5$. Calculated, %: C 68.98; H 4.43; N 7.10.

3-(4-Bromobenzoyl)-2-hydroxy-3a-{(5',5'-dimethyl-4-oxo-4',5'-dihydro-4*H*-spiro[naphthalene-1,3'-pyrrol]-2'-yl)methyl}pyrrolo[1,2-*a*]-quinoxaline-1,4(3a*H*,5*H*)-dione (IIIj). Yield 96%, mp 230–231°C (dichloroethane). IR spectrum, cm^{-1}: 3219 br (OH), 3156 br (NH), 1691 (C^1=O, C^4=O), 1656 (COC_6H_4Br-4, C^4=$O_{naphth.}$). ^{1}H NMRspectrum, δ, ppm: 1.29 s, 1.36 s (6H, 2Me), 2.03, 2.27 d.d (2H, $C^{4'}H_2$, *J* 14.1 Hz), 1.82, 3.12 d.d (2H, CH_2, *J* 16.8 Hz), 5.77 s (1H, $C^3H_{naphth.}$), 6.29 s (1H, $C^2H_{naphth.}$), 7.01–7.98 group of signals (12H, $3C_6H_4$), 10.71 s (1H, NH), 11.72 br.s (1H, OH). Found, %: C 64.02; H 4.25; N 6.60. $C_{34}H_{26}N_3O_5Br$. Calculated, %: C 64.16; H 4.12; N 6.60.

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