ISSN 1070-4280, Russian Journal of Organic Chemistry, 2008, Vol. 44, No. 4, pp. 582–586. © Pleiades Publishing, Ltd., 2008.
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Five-Membered 2,3-Dioxo Heterocycles: LVI.* Reaction of 3-Aroyl-1*H*-pyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones with Acyclic Enamino Ketones

N. L. Racheva, M. A. Belova, and A. N. Maslivets

Perm State University, ul. Bukireva 15, Perm, 614990 Russia
e-mail: koh2@psu.ru

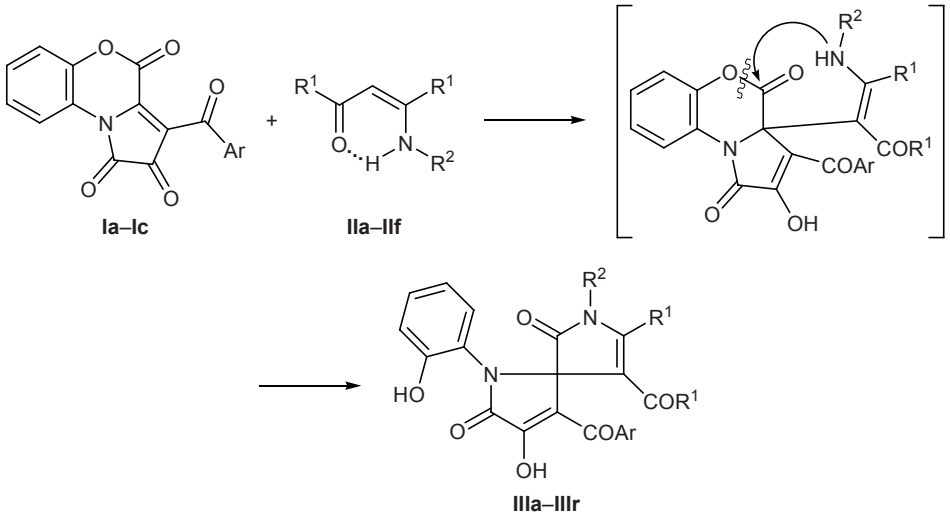
Received May 16, 2006

Abstract—3-Aroyl-1*H*-pyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones react with 4-arylaminopent-3-en-2-ones and 3-amino-1,3-diphenylprop-2-en-1-ones to give substituted 4-aroyl-3-hydroxy-1-(*o*-hydroxyphenyl)-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-diones.

DOI: 10.1134/S1070428008040192

Recyclizations and heterocyclizations of 4-acyl-1*H*-pyrrole-2,3-diones, including those fused to a nitrogen-containing heteroring at the *a* side, by the action of difunctional nucleophiles are widely used as an accessible method for building up various fused heterocyclic systems [2, 3]. We previously showed that 4-acyl-1*H*-pyrrole-2,3-diones fused to 1,4-benzoxazine system, namely 3-aroyl-1*H*-pyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones **Ia**–**Ic** [4], react with cyclic enamino ketones (3-amino-5,5-dimethylcyclohex-2-en-1-ones, both N-substituted and N-unsubstituted) as with 1,3-C,N-binucleophiles. The reaction involves successive attack by the β-CH and NH groups of enamino ketone on the C^{3a} and C^4 carbon atoms, respectively,

Scheme 1.



Ia–Ic **IIa–IIf** **IIIa–IIIr**

I, Ar = Ph (**a**), 4-$MeOC_6H_4$ (**b**), 4-BrC_6H_4 (**c**); **II**, R^1 = Me, R^2 = Ph (**a**), 4-$MeOC_6H_4$ (**b**), 4-ClC_6H_4 (**c**), 4-BrC_6H_4 (**d**); R^1 = Ph, R^2 = $PhCH_2$ (**e**), 4-$EtOC_6H_4$ (**f**); **III**, R^1 = Me (**a**–**l**), Ph (**m**–**r**); Ar = R^2 = Ph (**a**); Ar = 4-$MeOC_6H_4$, R^2 = Ph (**b**); Ar = 4-BrC_6H_4, R^2 = Ph (**c**); Ar = Ph, R^2 = 4-$MeOC_6H_4$ (**d**); Ar = R^2 = 4-$MeOC_6H_4$ (**e**); Ar = 4-BrC_6H_4, R^2 = 4-$MeOC_6H_4$ (**f**); Ar = Ph, R^2 = 4-ClC_6H_4 (**g**); Ar = 4-$MeOC_6H_4$, R^2 = 4-ClC_6H_4 (**h**); Ar = 4-BrC_6H_4Br, R^2 = 4-ClC_6H_4 (**i**); Ar = Ph, R^2 = 4-BrC_6H_4 (**j**); Ar = 4-$MeOC_6H_4$, R^2 = 4-BrC_6H_4 (**k**); Ar = R^2 = 4-BrC_6H_4 (**l**); Ar = Ph, R^2 = $PhCH_2$ (**m**); Ar = 4-$MeOC_6H_4$, R^2 = $PhCH_2$ (**n**); Ar = 4-BrC_6H_4, R^2 = $PhCH_2$ (**o**); Ar = Ph, R^2 = 4-$EtOC_6H_4$ (**p**); Ar = 4-$MeOC_6H_4$, R^2 = 4-$EtOC_6H_4$ (**q**); Ar = 4-BrC_6H_4, R^2 = 4-$EtOC_6H_4$ (**r**).

* For communication LV, see [1].

of pyrrolobenzoxazinetrione and is accompanied by cleavage of the oxazine ring at the C^4–O^5 bond to give 3′-aroyl-4′-hydroxy-1′-(*o*-hydroxyphenyl)-6,6-dimethyl-6,7-dihydrospiro[indole-3,2′-pyrrole]-2,4,5′-(1*H*,1′*H*,5*H*)-triones [5] whose structure was proved by X-ray analysis.

In continuation of our studies on reactions of hetareno[*a*]pyrrole-2,3-diones with difunctional nucleophiles, in the present work we examined reactions of 3-aroyl-1*H*-pyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones **Ia**–**Ic** with acyclic enamino ketones, 4-arylaminopent-3-en-2-ones **IIa**–**IId** and N-substituted 3-amino-1,3-diphenylprop-2-en-1-ones **IIe** and **IIf**. Unlike cyclic analogs, acyclic enamino ketones **IIa**–**IIf** exist as *Z* isomers with intramolecular hydrogen bond formed by the NH proton and ketone carbonyl oxygen atom, and the β-CH and NH groups are oriented at different sides with respect to the double C=C bond, which makes compounds **II** difficult to react as binucleophiles. Cyclic enamino ketones exist as *E* isomers where the β-CH and NH groups are arranged at the same side of the double bond, and such arrangement favors their behavior as binucleophiles.

By heating equimolar amounts of pyrrolobenzoxazinetriones **Ia**–**Ic** and enamino ketones **IIa**–**IIf** in boiling anhydrous benzene for 3–7 min (until dark violet color typical of initial compounds **I** disappeared) we obtained in high yields substituted 4-aroyl-3-hydroxy-1-(*o*-hydroxyphenyl)-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-diones **IIIa**–**IIIr**** (Scheme 1). Compounds **IIIa**–**IIIr** were isolated as colorless or light yellow crystalline substances melting at high temperature with decomposition; they are readily soluble in DMF and DMSO, poorly soluble in most other organic solvents, and insoluble in saturated hydrocarbons and water. Compounds **IIIa**–**IIIr** showed a positive test (cherry color) for enolic and phenolic hydroxy groups with an alcoholic solution of iron(III) chloride.

The IR spectra of spirobipyrroles **IIIa**–**IIIr** contained absorption bands due to stretching vibrations of the OH groups (a broad band in the region 3040–3170 cm^{-1}), lactam carbonyl groups (one or two peaks at 1700–1769 cm^{-1}), and acetyl and aroyl carbonyl groups (two peaks at 1620–1688 cm^{-1} in the spectra of **IIIa**–**IIIl** and at 1620–1682 cm^{-1} in the spectra of **IIIm**–**IIIr**). Compounds **IIIa**–**IIIr** showed in the ^1H NMR spectra (DMSO-d_6) signals from protons in the aromatic rings and substituents attached thereto, singlets from the 8-methyl group and 9-acetyl group at δ 2.04–2.08 and 2.17–2.21 ppm, respectively (**IIIa**–**IIII**), a doublet of doublets from the diastereotopic benzylic protons at δ 4.39–4.80 ppm (**IIIm**–**IIIo**), a singlet from the phenolic hydroxy proton at δ 9.94–10.20 (**IIIa**–**IIII**) or 9.53–9.76 ppm (**IIIm**–**IIIr**), and a broadened singlet from the enolic hydroxy proton at δ 12.00–12.70 ppm.

Presumably, in the first step activated β-CH group in acyclic enamino ketone **II** adds at the carbon atom in the 3a-position of pyrrolobenzoxazinetrione **I**, as in reactions of the latter with common nucleophiles [2–4]. Next follows isomerization of the enamino fragment from *Z* configuration to *E* and pyrrole ring closure as a result of intramolecular attack by the amino group on the lactone carbonyl carbon atom (C^4) in the oxazine ring of **I**, which is accompanied by cleavage of the C^4–O^5 bond. The described reaction may be regarded as a fairly rare example of regioselective synthesis of difficultly accessible spiro-fused bipyrrole heterocyclic system with various substituents in several positions of both pyrrole rings.

EXPERIMENTAL

The IR spectra were recorded on a UR-20 spectrometer from samples dispersed in mineral oil. The ^1H and ^{13}C NMR spectra were measured on a Bruker AM-400 instrument (400 MHz for ^1H) from solutions in DMSO-d_6 using tetramethylsilane as internal reference. The purity of the products was checked by thin-layer chromatography on Silufol plates using ethyl acetate or ethyl acetate–benzene (1:5) as eluent; spots were detected by treatment with iodine vapor.

9-Acetyl-4-benzoyl-3-hydroxy-1-(2-hydroxyphenyl)-8-methyl-7-phenyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIa). A solution of 1.0 mmol of compound **Ia** and 1.0 mmol of enamine **IIa** in 10 ml of anhydrous benzene was heated for 3 min under reflux. The mixture was cooled, and the precipitate was filtered off. Yield 88%, mp 198–199°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3050 br (OH), 1765 (C^6=O), 1705 (C^2=O), 1640 and 1626 (4-C=O, 9-C=O). ^1H NMR spectrum, δ, ppm: 2.07 s (3H, Me), 2.21 s (3H, MeCO), 6.89–7.85 m (14H, H_{arom}), 10.00 s (1H, 2′-OH), 12.70 br.s (1H, 3-OH). Found, %: C 70.40; H 4.67; N 5.62. $C_{29}H_{22}N_2O_6$. Calculated, %: C 70.44; H 4.48; N 5.67.

Compounds **IIIb**–**IIIr** were synthesized in a similar way.

** For preliminary communication, see [6].

9-Acetyl-3-hydroxy-1-(2-hydroxyphenyl)-4-(4-methoxybenzoyl)-8-methyl-7-phenyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIb). Yield 97%, mp 209–212°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3160 br (OH), 1754 (C^6=O), 1703 (C^2=O), 1667 and 1628 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.05 s (3H, Me), 2.18 s (3H, MeCO), 3.86 s (3H, OMe), 6.82–7.87 m (13H, H_{arom}), 9.98 s (1H, 2′-OH), 12.53 br.s (1H, 3-OH). Found, %: C 68.59; H 4.71; N 5.36. $C_{30}H_{24}N_2O_7$. Calculated, %: C 68.70; H 4.61; N 5.34.

9-Acetyl-4-(4-bromobenzoyl)-3-hydroxy-1-(2-hydroxyphenyl)-8-methyl-7-phenyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIc). Yield 87%, mp 210–211°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3170 br (OH), 1753 (C^6=O), 1703 (C^2=O), 1666 and 1631 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.07 s (3H, Me), 2.20 s (3H, MeCO), 6.85–7.76 m (13H, H_{arom}), 10.02 s (1H, 2′-OH), 12.40 br.s (1H, 3-OH). Found, %: C 60.87; H 3.63; Br 14.07; N 5.01. $C_{29}H_{21}BrN_2O_6$. Calculated, %: C 60.75; H 3.69; Br 13.94; N 4.89.

9-Acetyl-4-benzoyl-3-hydroxy-1-(2-hydroxyphenyl)-7-(4-methoxyphenyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIId). Yield 85%, mp 228–230°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3100 br (OH), 1764 (C^6=O), 1713 (C^2=O), 1665, 1634 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.05 s (3H, Me), 2.19 s (3H, MeCO), 3.81 s (3H, OMe), 6.83–8.03 m (13H, H_{arom}), 9.99 s (1H, 2′-OH), 12.70 br.s (1H, 3-OH). Found, %: C 68.77; H 4.54; N 5.27. $C_{30}H_{24}N_2O_7$. Calculated, %: C 68.70; H 4.61; N 5.34.

9-Acetyl-3-hydroxy-1-(2-hydroxyphenyl)-4-(4-methoxybenzoyl)-7-(4-methoxyphenyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIe). Yield 87%, mp 220–222°C (from BuOAc). IR spectrum, ν, cm^{-1}: 3150 br (OH), 1732 (C^6=O, C^2=O), 1688 and 1638 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.04 s (3H, Me), 2.17 s (3H, MeCO), 3.81 s (3H, OMe), 3.87 s (3H, OMe), 6.82–7.87 m (12H, C_6H_4), 9.98 s (1H, 2′-OH), 12.50 br.s (1H, 3-OH). Found, %: C 67.84; H 4.82; N 5.02. $S_{31}N_{26}N_2O_8$. Calculated, %: C 67.74; H 4.73; N 5.05.

9-Acetyl-4-(4-bromobenzoyl)-3-hydroxy-1-(2-hydroxyphenyl)-7-(4-methoxyphenyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIf). Yield 86%, mp 209–210°C (from BuOAc). IR spectrum, ν, cm^{-1}: 3110 br (OH), 1765 (C^6=O), 1704 (C^2=O), 1663 and 1626 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.04 s (3H, Me), 2.18 s (3H, MeCO), 3.81 s (3H, OMe), 6.82–7.75 m (12H, H_{arom}), 9.99 s (1H, 2′-OH), 12.50 br.s (1H, 3-OH). Found, %: C 59.78; H 3.93; Br 13.41; N 4.55. $C_{30}H_{23}BrN_2O_7$. Calculated, %: C 59.71; H 3.84; Br 13.24; N 4.64.

9-Acetyl-4-benzoyl-7-(4-chlorophenyl)-3-hydroxy-1-(2-hydroxyphenyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIg). Yield 88%, mp 200–203°C (from benzene). IR spectrum, ν, cm^{-1}: 3170 br (OH), 1748 (C^6=O), 1711 (C^2=O), 1668 and 1627 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.07 s (3H, Me), 2.20 s (3H, MeCO), 6.86–7.92 m (13H, H_{arom}), 10.20 s (1H, 2′-OH), 12.40 br.s (1H, 3-OH). Found, %: C 65.69; H 4.21; Cl 6.65; N 5.39. $C_{29}H_{21}ClN_2O_6$. Calculated, %: C 65.85; H 4.00; Cl 6.70; N 5.30.

9-Acetyl-7-(4-chlorophenyl)-3-hydroxy-1-(2-hydroxyphenyl)-4-(4-methoxybenzoyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIh). Yield 87%, mp 216–217°C (from benzene). IR spectrum, ν, cm^{-1}: 3160 br (OH), 1744 (C^6=O), 1703 (C^2=O), 1669 and 1626 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.07 s (3H, Me), 2.20 s (3H, MeCO), 3.86 s (3H, OMe), 6.86–7.87 m (12H, H_{arom}), 10.00 s (1H, 2′-OH), 12.50 br.s (1H, 3-OH). Found, %: C 64.58; H 4.09; Cl 6.39; N 4.92. $C_{30}H_{23}ClN_2O_7$. Calculated, %: C 64.46; H 4.15; Cl 6.34; N 5.01.

9-Acetyl-4-(4-bromobenzoyl)-7-(4-chlorophenyl)-3-hydroxy-1-(2-hydroxyphenyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIi). Yield 89%, mp 199–201°C (from BuOAc). IR spectrum, ν, cm^{-1}: 3150 br (OH), 1759 (C^6=O), 1708 (C^2=O), 1673 and 1638 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.08 s (3H, Me), 2.18 s (3H, MeCO), 6.82–7.75 m (12H, H_{arom}), 10.00 s (1H, 2′-OH), 12.50 br.s (1H, 3-OH). Found, %: C 57.33; H 3.30; Br 13.24; Cl 5.79; N 4.57. $C_{29}H_{20}BrClN_2O_6$. Calculated, %: C 57.30; H 3.32; Br 13.15; Cl 5.83; N 4.61.

9-Acetyl-4-benzoyl-7-(4-bromophenyl)-3-hydroxy-1-(2-hydroxyphenyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIj). Yield 90%, mp 203–204°C (from benzene). IR spectrum, ν, cm^{-1}: 3170 br (OH), 1748 (C^6=O), 1711 (C^2=O), 1672 and 1628 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.07 s (3H, Me), 2.21 s (3H, MeCO), 6.80–7.93 m (13H, H_{arom}), 9.94 s (1H, 2′-OH), 12.40 br.s (1H, 3-OH). Found, %: C 60.68; H 3.77; Br 13.78; N 5.02.

$C_{29}H_{21}BrN_2O_6$. Calculated, %: C 60.75; H 3.69; Br 13.94; N 4.89.

9-Acetyl-7-(4-bromophenyl)-3-hydroxy-1-(2-hydroxyphenyl)-4-(4-methoxybenzoyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIk). Yield 89%, mp 220–221°C (from benzene). IR spectrum, ν, cm^{-1}: 3130 br (OH), 1733 (C^6=O), 1713 (C^2=O), 1661 and 1620 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.07 s (3H, Me), 2.18 s (3H, MeCO), 3.86 s (3H, OMe), 6.81–7.87 m (12H, H_{arom}), 10.00 s (1H, 2′-OH), 12.00 br.s (1H, 3-OH). Found, %: C 59.62; H 3.78; Br 13.30; N 4.57. $C_{30}H_{23}BrN_2O_7$. Calculated, %: C 59.71; H 3.84; Br 13.24; N 4.64.

9-Acetyl-4-(4-bromobenzoyl)-7-(4-bromophenyl)-3-hydroxy-1-(2-hydroxyphenyl)-8-methyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIl). Yield 88%, mp 225–226°C (from benzene). IR spectrum, ν, cm^{-1}: 3070 br (OH), 1736 (C^6=O), 1719 (C^2=O), 1661 and 1626 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 2.08 s (3H, Me), 2.18 s (3H, MeCO), 6.81–7.79 m (12H, H_{arom}), 10.00 s (1H, 2′-OH), 12.40 br.s (1H, 3-OH). Found, %: C 53.37; H 3.11; Br 24.58; N 4.23. $C_{29}H_{20}Br_2N_2O_6$. Calculated, %: C 53.40; H 3.09; Br 24.50; N 4.29.

4,9-Dibenzoyl-7-benzyl-3-hydroxy-1-(2-hydroxyphenyl)-8-phenyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIm). Yield 90%, mp 279–281°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3040 br (OH), 1727 (C^6=O, C^2=O), 1682 and 1632 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 4.39 d.d and 4.80 d.d (1H each, C**H**$_2$Ph, J = 16.5 Hz), 6.62–7.89 m (24H, H_{arom}), 9.64 s (1H, 2′-OH), 12.40 br.s (1H, 3-OH). Found, %: C 75.98; H 4.52; N 4.34. $C_{40}H_{28}N_2O_6$. Calculated, %: C 75.94; H 4.46; N 4.43.

9-Benzoyl-7-benzyl-3-hydroxy-1-(2-hydroxyphenyl)-4-(4-methoxybenzoyl)-8-phenyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIn). Yield 89%, mp 272–273°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3130 br (OH), 1736 (C^6=O), 1709 (C^2=O), 1681 and 1628 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 3.88 s (3H, OMe), 4.39 d.d and 4.80 d.d (1H each, C**H**$_2$Ph, J = 16.5 Hz), 6.66–7.90 m (23H, H_{arom}), 9.53 s (1H, 2′-OH), 12.20 br.s (1H, 3-OH). Found, %: C 74.37; H 4.63; N 4.14. $C_{41}H_{30}N_2O_7$. Calculated, %: C 74.31; H 4.56; N 4.23.

9-Benzoyl-7-benzyl-4-(4-bromobenzoyl)-3-hydroxy-1-(2-hydroxyphenyl)-8-phenyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIo). Yield 91%, mp 284–285°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3050 br (OH), 1730 (C^6=O, C^2=O), 1677 and 1636 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 4.39 d.d and 4.79 d.d (1H each, C**H**$_2$Ph, J = 16.3 Hz), 6.62–7.81 m (23H, H_{arom}), 9.66 s (1H, 2′-OH), 12.60 br.s (1H, 3-OH). Found, %: C 67.61; H 3.79; Br 11.25; N 3.86. $C_{40}H_{27}BrN_2O_6$. Calculated, %: C 67.52; H 3.82; Br 11.23; N 3.94.

4,9-Dibenzoyl-7-(4-ethoxyphenyl)-3-hydroxy-1-(2-hydroxyphenyl)-8-phenyl-1,7-diazaspiro[4.4]-nona-3,8-diene-2,6-dione (IIIp). Yield 92%, mp 269–270°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3150 br (OH), 1769 (C^6=O), 1728 (C^2=O), 1668 and 1634 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 1.27 t (3H, CH_3, J = 6.9 Hz), 4.03 q (2H, OCH_2, J = 7.0 Hz), 6.61–7.92 m (23H, H_{arom}), 9.76 s (1H, 2′-OH), 12.40 br.s (1H, 3-OH). Found, %: C 74.26; H 4.61; N 4.29. $C_{41}H_{30}N_2O_7$. Calculated, %: C 74.31; H 4.56; N 4.23.

9-Benzoyl-7-(4-ethoxyphenyl)-3-hydroxy-1-(2-hydroxyphenyl)-4-(4-methoxybenzoyl)-8-phenyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIq). Yield 90%, mp 258–259°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3140 br (OH), 1726 (C^6=O), 1700 (C^2=O), 1665 and 1630 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 1.28 t (3H, Me, J = 6.9 Hz), 3.88 s (3H, OMe), 3.95 q (2H, OCH_2, J = 7.1 Hz), 6.59–7.92 m (22H, H_{arom}), 9.72 s (1H, 2′-OH), 12.20 br.s (1H, 3-OH). Found, %: C 72.73; H 4.69; N 4.10. $C_{42}H_{32}N_2O_8$. Calculated, %: C 72.82; H 4.66; N 4.04.

9-Benzoyl-4-(4-bromobenzoyl)-7-(4-ethoxyphenyl)-3-hydroxy-1-(2-hydroxyphenyl)-8-phenyl-1,7-diazaspiro[4.4]nona-3,8-diene-2,6-dione (IIIr). Yield 91%, mp 262–263°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3150 br (OH), 1736 (C^6=O), 1723 (C^2=O), 1671 and 1620 (4-C=O, 9-C=O). ^{1}H NMR spectrum, δ, ppm: 1.27 t (3H, Me, J = 6.9 Hz), 3.95 q (2H, OCH_2, J = 6.9 Hz), 6.60–7.86 m (22H, H_{arom}), 9.76 s (1H, 2′-OH), 12.60 br.s (1H, 3-OH). Found, %: C 66.37; H 3.99; Br 10.81; N 3.74. $C_{41}H_{29}BrN_2O_7$. Calculated, %: C 66.40; H 3.94; Br 10.77; N 3.78.

This study was performed under financial support by the Russian Foundation for Basic Research (project no. 07-03-96036).

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