

Five-Membered 2,3-Dioxo Heterocycles: XLVI.* Reaction of 5-Aryl-4-quinoxaliny-2,3-dihydrofuran-2,3-diones with Aldehydes and Ketones. Molecular and Crystalline Structure of 5-(3-*p*-Tolylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spiro-2'-adamantan-4-one

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Abstract—Aroyl(quinoxaliny)ketenes generated by thermolysis of 5-aryl-4-(3-arylquinoxalin-2-yl)-2,3-dihydrofuran-2,3-diones act as dienes in [4+2]-cycloaddition at the carbonyl group of aldehydes and ketones to afford 2-substituted 6-aryl-5-(3-arylquinoxalin-2-yl)-4*H*-1,3-dioxin-4-ones. The structure of 5-(3-*p*-tolylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spiro-2'-adamantan-4-one was proved by X-ray analysis.

4,5-Disubstituted 2,3-dihydrofuran-2,3-diones are thermally unstable. On heating to a temperature approaching their melting point (130–140°C), these compounds undergo decarbonylation to give the corresponding acylketenes; in solution, the temperature of decarbonylation decreases by 50–70°C. Acylketenes generated in such a way are capable of reacting with various dienophiles, following [4+2]-cycloaddition pattern, while in the absence of reaction partner, the path of their stabilization is determined by substituents present therein. The most usual stabilization path is [4+2]-cycloaddition (cyclodimerization) where one acylketene molecule acts as a diene through its conjugated carbonylketene moiety (O=C–C=C=), and the other behaves as dienophile (ketene C=C bond). This stabilization path leading to substituted 2-pyranones is typical of aryl(aroyl)-, aroyl(halo)-, and aroyl(methyl)ketenes formed by thermolysis of 4,5-diaryl-, 5-aryl-4-halo-, and 5-aryl-4-methyl-2,3-dihydrofuran-2,3-diones, respectively [2, 3]. Dibenzoylketene generated by thermal decarbonylation of 4-benzoyl-5-phenyl-2,3-dihydrofuran-2,3-dione also reacts according to the above scheme, but the corresponding primary product, 2-pyranone derivative, loses CO₂ molecule or undergoes [1,3]-shift of the benzoyl group

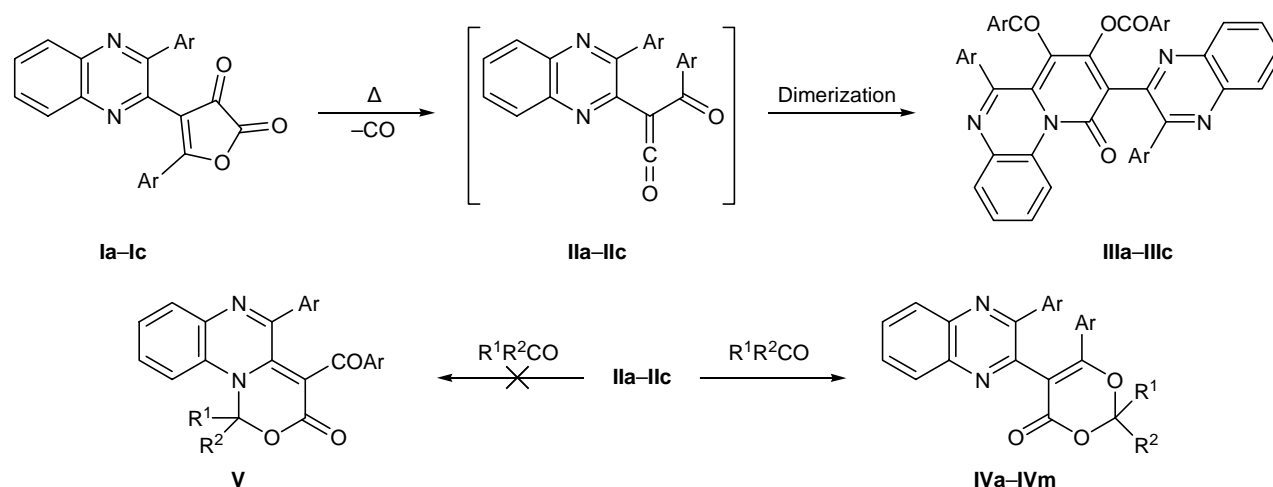
[4]. Stabilization of dipivaloylketene generated by thermolysis of 5-*tert*-butyl-4-pivaloyl-2,3-dihydrofuran-2,3-dione is achieved via [4+2]-cycloaddition of the carbonylketene fragment of one molecule to the carbonyl group of the pivaloyl or ketene moiety of the other molecule [5]. By contrast, thermolysis of 4-[α -(arylimino)benzyl]-5-phenyl-2,3-dihydrofuran-2,3-diones gives rise to benzoyl(imido)ketenes which are stabilized via intramolecular 6 π -electrocyclic ring closure, leading to substituted quinolin-4-ones [6].

Thermolysis of substituted 5-aryl-4-(3-arylquinoxalin-2-yl)-2,3-dihydrofuran-2,3-diones **Ia–Ic** is also accompanied by decarbonylation to give aroyl(3-arylquinoxalin-2-yl)ketenes **IIa–IIc** which can be classed with aroyl(imido)ketenes. In the absence of other reaction partners, they undergo [4+2]-cyclodimerization where one aroylketene molecule acts as diene through the conjugated imido)ketene bond sequence –N=C–C=C=, and the other acts as dienophile through the ketene –C=C= bond. As a result, 4-aryloxy-2-(3-arylquinoxalin-2-yl)-1*H*-pyrido[1,2-*a*]quinoxalin-1-ones **IIIa–IIIc** are formed [7, 8] (Scheme 1).

With the goal of estimating the reactivity of carbonyl compounds in intermolecular cycloaddition with ketenes **IIa–IIc** in comparison with ketenes themselves (i.e., cyclodimerization of the latter), as

* For communication XLV, see [1].

Scheme 1.



I–III, Ar = Ph (**a**), *p*-MeC₆H₄ (**b**), 2,5-Me₂C₆H₃ (**c**); **IV**, Ar = Ph, R¹ = *p*-BrC₆H₄, R² = H (**a**); Ar = *p*-MeC₆H₄, R¹ = *p*-BrC₆H₄, R² = H (**b**); Ar = Ph, R¹ = *p*-MeOC₆H₄, R² = H (**c**); Ar = Ph, R¹ = 3,4-(MeO)₂C₆H₃, R² = H (**d**); Ar = Ph, R¹ = *m*-O₂NC₆H₄, R² = H (**e**); Ar = Ph, R¹ = Me₂CHCH₂, R² = H (**f**); Ar = Ph, R¹R² = (CH₂)₄ (**g**); Ar = *p*-MeC₆H₄, R¹R² = (CH₂)₄ (**h**); Ar = 2,5-Me₂C₆H₃, R¹R² = (CH₂)₄ (**i**); Ar = Ph, R¹R² = (CH₂)₅ (**j**); Ar = *p*-MeC₆H₄, R¹R² = (CH₂)₅ (**k**); Ar = Ph, R¹R² = (CH₂)₆ (**l**); Ar = *p*-MeC₆H₄, R¹R² = 2-adamantylidene (**m**).

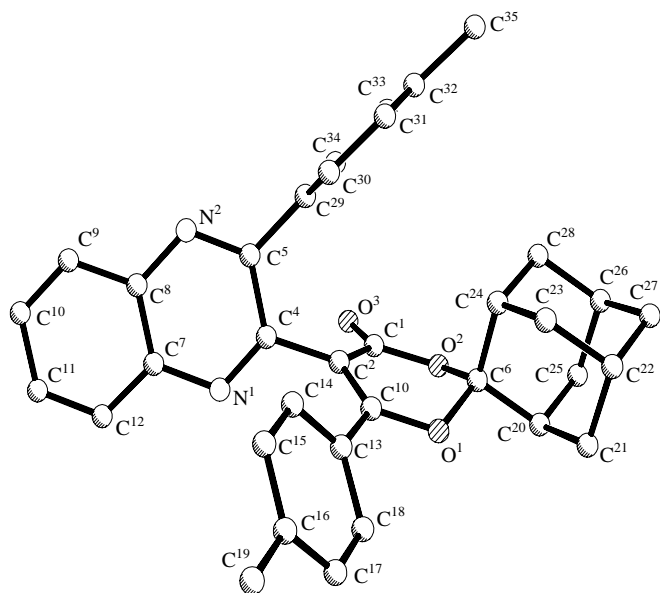
well as the regio- and stereoselectivity of these reactions, in the present work we made an attempt to trap intermediate ketenes **II** by aldehydes and ketones.

Heating of mixtures of furandiones **Ia–Ic** and carbonyl compounds in a high-boiling aprotic solvent at 130–140°C (i.e., at a temperature corresponding to decarbonylation of initial furandiones) for 15–20 min

led to formation of the corresponding [4+2]-adducts whose spectral parameters did not allow us to choose between two alternative isomeric structures **IV** and **V** (Scheme 1). On the basis of the results of X-ray diffraction study of adamantane derivative **IVm** we unambiguously identified the cycloaddition products as 2-substituted 6-aryl-5-(3-arylquinoxalin-2-yl)-4H-1,3-dioxin-4-ones **IVa–IVm** [9].

Obviously, aroyl(imido)ketenes **IIa–IIc** generated by thermal decarbonylation of furandiones **Ia–Ic** react as dienes at the carbonyl group of aldehydes and ketones, and the conjugated aroylketene bond system O=C–C=C= is involved. It remains unclear why the regioselectivity of this reaction differs from that observed in the cyclodimerization of **II** [7, 8]; the problem is now under discussion.

The ¹H NMR spectra of compounds **IVa–IVm** in DMSO-*d*₆ contain signals from aromatic protons in the region δ 6.64–8.69 ppm, a group of signals from the polymethylene and adamantane fragments at δ 1.42–2.30 ppm (compounds **IVg–IVm**), a doublet δ 1.04 ppm from the methyl groups and a multiplet at δ 1.85 ppm from the isobutyl radical (**IVf**), singlets from methyl protons at δ 1.84–2.37 ppm (**IVb**, **IVh**, **IVi**, **IVk**, and **IVm**), a singlet from methoxy protons at δ 3.83 ppm (**IVc** and **IVd**), and a singlet from the 2-H proton in the 1,3-dioxine fragment at δ 6.22–7.10 ppm (**IVa–IVf**). In the ¹H NMR spectra of all compounds



Structure of the molecule of 6-*p*-tolyl-5-(3-*p*-tolylquinoxalin-2-yl)-4H-1,3-dioxine-2-spiro-2'-adamantan-4-one (**IVm**) according to the X-ray diffraction data.

IVa–IVm, a four-proton multiplet separates from the set of aromatic proton signals. This multiplet is typical of an *AA'BB'* system formed by protons at C⁵–C⁸ of the quinoxaline fragment, which is very consistent with the assigned structure. In the ¹³C NMR spectrum of compound **IVi** in CDCl₃ we observed signals from carbon atoms of the phenyl groups (δ_c 128.09–141.38 ppm), hexamethylene fragment (δ_c 21.34–39.49 ppm), C², C⁴, C⁵, and C⁶ of the dioxine ring (δ_c 111.11, 163.93, 106.69, and 161.26 ppm, respectively), and C² and C³ of the quinoxaline ring (δ_c 147.72 and 155.63 ppm, respectively).

Figure shows the general view of molecule **IVm** according to the X-ray diffraction data, and the principal bond lengths and bond angles in the molecule are listed in table. The structure is characterized by clearly localized double bonds N¹=C⁴, N²=C⁵, and C²=C³ (1.312, 1.314, and 1.354 Å, respectively). The quinoxaline fragment is planar. The plane of the *p*-tolyl group on C⁵ is orthogonal to the quinoxaline ring plane, and its orientation is bisector. The dioxine ring adopts an *envelope* conformation folded along the O¹...O² axis; the folding angle is 43.5°. The plane formed by the O¹, O², C¹, C², and C³ atoms with the quinoxaline ring plane forms an angle of 74.3° (the torsion angle C¹C²C⁴C⁵ is –77.7°), and the adamantane fragment declines from the plane including the above five atoms toward the *p*-tolyl substituent in the quinoxaline moiety. The tolyl group in the dioxine fragment is turned through an angle of –40.6° (C²C³C¹³C¹⁴) relative to the latter. Molecules **IVm** in crystal do not give rise to hydrogen bonds or other shortened intermolecular contacts.

EXPERIMENTAL

The IR spectra of were recorded on a UR-20 instrument from samples dispersed in mineral oil. The ¹H NMR spectra** were obtained on a Bruker AM-400 spectrometer (400 MHz) using DMSO-*d*₆ as solvent and HMDS as internal reference. The purity of products **IVa–IVm** was checked by TLC on Silufol plates using hexane–ethyl acetate (5:1) as eluent.

2-*p*-Bromophenyl-6-phenyl-5-(3-phenylquinoxalin-2-yl)-4*H*-1,3-dioxin-4-one (IVa). A solution of 1 mmol of furandione **Ia** [7, 8] and 1.1 mmol of *p*-bromobenzaldehyde in 5 ml of dry *p*-xylene was

Some bond lengths (*d*, Å) and bond angles (ω , deg) in the molecule of 6-*p*-tolyl-5-(3-*p*-tolylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spiro-2'-adamantan-4-one

Bond	<i>d</i>	Bond	<i>d</i>
O ¹ –C ³	1.357(2)	O ¹ –C ⁶	1.433(2)
O ² –C ¹	1.358(2)	O ² –C ⁶	1.437(2)
O ³ –C ¹	1.211(2)	N ¹ –C ⁴	1.312(2)
N ¹ –C ⁷	1.374(2)	N ² –C ⁵	1.314(2)
N ² –C ⁸	1.372(2)	C ¹ –C ²	1.457(2)
C ² –C ³	1.354(2)	C ² –C ⁴	1.491(3)
C ³ –C ¹³	1.471(2)	S ⁴ –C ⁵	1.439(2)
C ⁵ –C ²⁹	1.490(2)	C ⁶ –C ²⁰	1.511(3)
C ⁶ –C ²⁴	1.525(2)	C ⁷ –C ⁸	1.405(3)
Angle	ω	Angle	ω
C ³ O ¹ C ⁶	116.60(12)	C ¹ O ² C ⁶	117.94(13)
C ⁴ N ¹ C ⁷	116.62(16)	C ⁵ N ² C ⁸	117.10(16)
O ³ C ¹ O ²	117.99(17)	O ³ C ¹ C ²	124.54(18)
O ² C ¹ C ²	117.29(14)	C ³ C ² C ¹	118.48(16)
C ³ C ² C ⁴	125.10(15)	C ¹ C ² C ⁴	115.75(14)
C ² C ³ O ¹	120.16(14)	C ² C ³ C ¹³	128.48(16)
O ¹ C ³ C ¹³	111.30(13)	N ¹ C ⁴ C ⁵	122.40(17)
N ¹ C ⁴ C ²	118.28(15)	C ⁵ C ⁴ C ²	119.27(15)
N ² C ⁵ C ⁴	121.46(16)	N ² C ⁵ C ²⁹	117.87(15)
C ⁴ C ⁵ C ²⁹	120.67(16)	O ¹ C ⁶ O ²	108.84(13)
O ¹ C ⁶ C ²⁰	107.37(13)	O ² C ⁶ C ²⁰	107.49(14)
O ¹ C ⁶ C ²⁴	111.36(13)	O ² C ⁶ C ²⁴	110.94(13)
C ²⁰ C ⁶ C ²⁴	110.68(15)		

heated for 20 min at 138–140°C. The mixture was cooled, and the precipitate was filtered off and recrystallized from acetonitrile. Yield 79%, mp 141–142°C (from acetonitrile). IR spectrum, ν , cm^{–1}: 1735 (C⁶=O). ¹H NMR spectrum, δ , ppm: 6.50 s (1H, CH), 6.68–7.50 m (14H, C₆H₅, C₆H₄), 7.92 m (2H, 6-H, 7-H, quinoxaline), 8.11 d and 8.20 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 67.32; H 3.59; Br 14.95; N 5.28. C₃₀H₁₉BrN₂O₃. Calculated, %: C 67.30; H 3.58; Br 14.92; N 5.23. Compounds **IVb–IVm** were synthesized in a similar way.

2-*p*-Bromophenyl-6-*p*-tolyl-5-(3-*p*-tolylquinoxalin-2-yl)-4*H*-1,3-dioxin-4-one (IVb). Yield 56%, mp 198–200°C (from acetonitrile). IR spectrum, ν , cm^{–1}: 1755 (C⁶=O). ¹H NMR spectrum, δ , ppm: 2.22 s (3H, Me), 2.37 s (3H, Me), 6.82 s (1H, CH), 6.90–7.78 m (12H, C₆H₄), 7.92 m (2H, 6-H, 7-H, quinoxaline), 8.11 d and 8.19 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 68.20; H 4.12; Br 14.77; N 4.88.

** NMR studies were performed at the *Ural-YaMR* Center under support by the Russian Foundation for Basic Research (project no. 00-03-40 139).

$C_{32}H_{23}BrN_2O_3$. Calculated, %: C 68.21; H 4.11; Br 14.18; N 4.97.

2-*p*-Methoxyphenyl-6-phenyl-5-(3-phenylquinoxalin-2-yl)-4*H*-1,3-dioxin-4-one (IVc). Yield 64%, mp 185–186°C (from acetonitrile). IR spectrum, ν , cm^{-1} : 1765 ($C^6=O$). 1H NMR spectrum, δ , ppm: 3.83 s (3H, OMe), 6.76 s (1H, CH), 6.93–7.66 m (14H, C_6H_5 , C_6H_4), 7.94 m (2H, 6-H, 7-H, quinoxaline), 8.12 d and 8.19 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 76.55; H 4.55; N 5.88. $C_{31}H_{23}N_2O_4$. Calculated, %: C 76.53; H 4.56; N 5.76.

2-(3,4-Dimethoxyphenyl)-6-phenyl-5-(3-phenylquinoxalin-2-yl)-4*H*-1,3-dioxin-4-one (IVd). Yield 18%, mp 145–147°C (from acetonitrile). IR spectrum, ν , cm^{-1} : 1750 ($C^6=O$). 1H NMR spectrum, δ , ppm: 3.83 s (6H, MeO), 6.75 s (1H, CH), 6.96–7.47 m (13H, C_6H_5 , C_6H_3), 7.94 m (2H, 6-H, 7-H, quinoxaline), 8.13 d and 8.19 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 74.42; H 4.70; N 5.38. $C_{32}H_{24}N_2O_3$. Calculated, %: C 74.41; H 4.68; N 5.42.

2-*m*-Nitrophenyl-6-phenyl-5-(3-phenylquinoxalin-2-yl)-4*H*-1,3-dioxin-4-one (IVe). Yield 21%, mp 138–140°C (from acetonitrile). IR spectrum, ν , cm^{-1} : 1745 ($C^6=O$). 1H NMR spectrum, δ , ppm: 7.10 s (1H, CH), 6.99–8.59 m (18H, C_6H_5 , C_6H_4). Found, %: C 71.85; H 3.83; N 8.40. $C_{30}H_{19}N_3O_5$. Calculated, %: C 71.85; H 3.82; N 8.35.

2-Isobutyl-6-phenyl-5-(3-phenylquinoxalin-2-yl)-4*H*-1,3-dioxin-4-one (IVf). Yield 53%, mp 148–150°C (from acetonitrile). IR spectrum, ν , cm^{-1} : 1740 ($C^6=O$). 1H NMR spectrum, δ , ppm: 1.04 d (6H, Me), 1.85 m (3H, CH–CH₂), 6.22 s (1H, CH), 6.91–7.44 m (10H, C_6H_5), 7.93 m (2H, 6-H, 7-H, quinoxaline), 8.11 d and 8.20 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 77.03; H 5.55; N 6.50. $C_{28}H_{24}N_2O_3$. Calculated, %: C 77.04; H 5.54; N 6.42.

6-Phenyl-5-(3-phenylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spirocyclopentan-4-one (IVg). Yield 79%, mp 141–142°C (from cyclohexane). IR spectrum, ν , cm^{-1} : 1740 ($C^6=O$). 1H NMR spectrum, δ , ppm: 1.77–2.29 m (8H, CH₂), 6.98–7.44 m (10H, C_6H_5), 7.93 m (2H, 6-H, 7-H, quinoxaline), 8.12 d and 8.19 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 77.39; H 5.12; N 6.51. $C_{28}H_{22}N_2O_3$. Calculated, %: C 77.40; H 5.10; N 6.42.

6-*p*-Tolyl-5-(3-*p*-tolylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spirocyclopentan-4-one (IVh). Yield 64%, mp 145–147°C (from cyclohexane). IR spectrum, ν , cm^{-1} : 1745 ($C^6=O$). 1H NMR spectrum, δ , ppm: 1.76–

2.30 m (8H, CH₂), 2.20 s (3H, Me), 2.37 s (3H, Me), 6.95–7.24 m (8H, C_6H_4), 7.92 m (2H, 6-H, 7-H, quinoxaline), 8.11 d and 8.16 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 77.91; H 5.66; N 6.04. $C_{30}H_{26}N_2O_3$. Calculated, %: C 77.90; H 5.67; N 6.06.

6-(2,5-Dimethylphenyl)-5-[3-(2,5-dimethylphenyl)quinoxalin-2-yl]-4*H*-1,3-dioxine-2-spirocyclopentan-4-one (IVi). Yield 54%, mp 188–190°C (from cyclohexane). IR spectrum, ν , cm^{-1} : 1740 ($C^6=O$). 1H NMR spectrum, δ , ppm: 1.64–2.28 m (8H, CH₂), 1.86 s (6H, Me), 1.86 s (6H, Me), 6.64–7.21 m (6H, C_6H_3), 7.92 m (2H, 6-H, 7-H, quinoxaline), 8.08 d and 8.22 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 78.33; H 6.17; N 5.80. $C_{32}H_{30}N_2O_3$. Calculated, %: C 78.34; H 6.16; N 5.71.

6-Phenyl-5-(3-phenylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spirocyclohexan-4-one (IVj). Yield 52%, mp 153–155°C (from cyclohexane). IR spectrum, ν , cm^{-1} : 1740 ($C^6=O$). 1H NMR spectrum, δ , ppm: 1.47–2.14 m (10H, CH₂), 7.05–7.43 m (10H, C_6H_5), 7.94 m (2H, 6-H, 7-H, quinoxaline), 8.13 d and 8.18 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 77.67; H 5.37; N 6.33. $C_{29}H_{24}N_2O_3$. Calculated, %: C 77.66; H 5.39; N 6.25.

6-*p*-Tolyl-5-(3-*p*-tolylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spirocyclohexan-4-one (IVk). Yield 75%, mp 196–198°C (from cyclohexane). IR spectrum, ν , cm^{-1} : 1738 ($C^6=O$). 1H NMR spectrum, δ , ppm: 1.49–2.13 m (10H, CH₂), 2.24 s (3H, Me), 2.35 s (3H, Me), 7.01–7.10 m (8H, C_6H_4), 7.92 m (2H, 6-H, 7-H, quinoxaline), 8.12 d and 8.16 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 78.15; H 5.91; N 5.79. $C_{31}H_{28}N_2O_3$. Calculated, %: C 78.13; H 5.92; N 5.88.

6-Phenyl-5-(3-phenylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spirocycloheptan-4-one (IVl). Yield 61%, mp 170–171°C (from cyclohexane). IR spectrum, ν , cm^{-1} : 1740 ($C^6=O$). 1H NMR spectrum, δ , ppm: 1.42–2.28 m (12H, CH₂), 6.94–7.30 m (10H, C_6H_5), 7.92 m (2H, 6-H, 7-H, quinoxaline), 8.11 d and 8.16 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 77.88; H 5.66; N 6.11. $C_{30}H_{26}N_2O_3$. Calculated, %: C 77.90; H 5.67; N 6.06.

6-*p*-Tolyl-5-(3-*p*-tolylquinoxalin-2-yl)-4*H*-1,3-dioxine-2-spiro-2'-adamantan-4-one (IVm). Yield 72%, mp 229–230°C (from ethyl acetate). IR spectrum, ν , cm^{-1} : 1725 ($C^6=O$). 1H NMR spectrum, δ , ppm: 1.70–2.04 m (14H, adamantane), 2.23 s (3H, Me), 2.36 s (3H, Me), 7.04–7.21 m (8H, C_6H_4), 7.91 m (2H, 6-H, 7-H, quinoxaline), 8.11 d and 8.16 d (2H, 5-H, 8-H, quinoxaline). Found, %: C 79.57; H 6.14;

N 5.29. C₃₅H₃₂N₂O₃. Calculated, %: C 79.52; H 6.10; N 5.30.

X-Ray diffraction study of a single crystal of compound IVm. Well defined triclinic crystals, C₃₅H₃₂N₂O₃, with the following unit cell parameters: $a = 10.678(2)$, $b = 11.288(2)$, $c = 13.363(3)$ Å; $\alpha = 108.07(3)$, $\beta = 110.97(3)$, $\gamma = 95.02(3)^\circ$; $V = 1393.6(5)$ Å³; $M = 528.63$; $Z = 2$; $d_{\text{calc}} = 1.260$ g/cm³; space group $P-1$. The unit cell parameters and reflection intensities were measured on a KM-4 (KUMA DIFFRACTION) automatic four-circle diffractometer (CuK α irradiation, scan range $3.8 < \Theta < 80.2^\circ$). The structure was solved by the direct statistical method. All hydrogen atoms, including those of the methyl groups, were localized by the difference synthesis of electron density. The structure was refined by the least-squares procedure in full-matrix anisotropic (isotropic for hydrogen atoms) approximation from 3343 reflections with $I > 2\sigma(I)$ (total of 5200 reflections were measured) to $R = 0.0421$; GOF = 0.966. No correction for absorption was introduced ($\mu = 0.635$ mm⁻¹). The calculations were performed with the aid of SHELX 97 software package [10].

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