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PHOTO- AND THERMOCHROMIC SPIRANES.
25*. NEW INDOLINOSPIROPYRANS CONTAINING A CONDENSED FURAN FRAGMENT

B. S. Lukyanov[1], A. N. Utenyshev[2], E. N. Shepelenko[1], V. V. Tkachev[2], M. B. Lukyanova[1], A. V. Metelitsa[1], S. O. Bezuglyi[1], S. M. Aldoshin[2], and V. I. Minkin[1]

New indolinospirochromenes have been synthesized containing a condensed furan fragment, an analog of the methoxyl substituent in position 6' of the chromene part of the molecule. Solutions of the obtained compounds display photochromic properties. For one of the compounds, depending on the temperature of irradiation, the cis-cisoid or trans-transoid isomer is fixed spectrally. An X-ray structural investigation has been carried out on crystals of spiropyrans possessing various forms of photoactivity in the solid phase.

Keywords: indolinospiropyrans, furan, X-ray structural analysis, photochromism.

The effect of substituents on the photochromic properties of spiropyrans has been studied previously, mainly on compounds containing various π-acceptor substituents [1-3]. The investigation of the effect of π-donor substituents is limited by their lack of variety, however it seems of particular interest since 6'-hydroxy-1,3,3-trimethylspiro(indolino-2',2'-[2H]-benzopyran) [4], and also indolinospiropyrans containing a methoxyl group in the 2H-chromene fragment [5], display photochromic properties in the solid phase.

A condensed furan fragment is an original replacement for the π-donor methoxyl group in position 6' of the spiropyran.

Spiropyrans **3b-d** (Table 1) were obtained by a method analogous to that described previously for spiropyran **3a** [6] by brief boiling in alcohol of equimolar quantities of 1,2,3,3-tetramethylindolenylium perchlorate **1**, the appropriate aldehyde **2**, and piperidine.

The IR spectra of compounds **3** contain absorption bands characteristic of the vibrations of the C=C bond of a pyran ring at 1600-1650 cm^{-1} (Table 2).

In the ^{1}H NMR ($CDCl_3$) spectra of spirochromenes **3** signals were observed separately for the *gem*-dimethyl groups of the indoline fragment as a result of the acoplanarity of the molecule, but the doublet signal of the proton in position 3' of the pyran ring is characterized by a coupling constant of 8.6-10.6 Hz, which corresponds to a *cis* configuration for the vinyl fragment (Table 2).

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* For Part 24 see [1].

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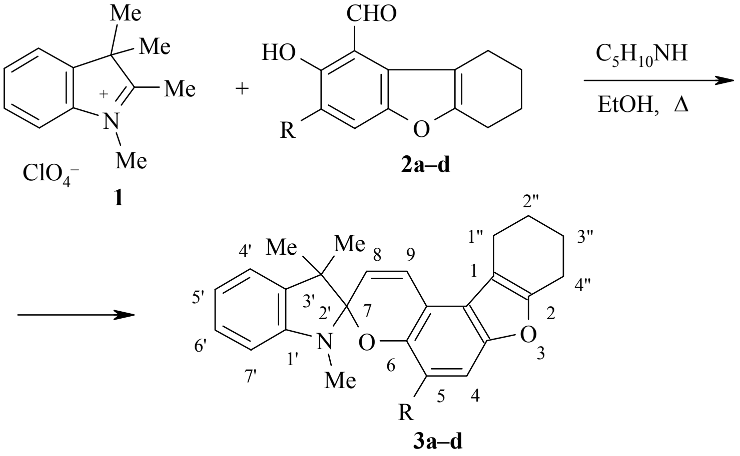
[1] Institute of Physical and Organic Chemistry, Rostov State University, Rostov-on-Don 344090, Russia; e-mail: bluk@ipoc.rsu.ru. [2] Institute of Problems of Chemical Physics, Russian Academy of Sciences, Chernogolovka 142432, Moscow Region. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 7, pp. 995-1005, July, 2006. Original article submitted September 9, 2004.



2,3 a R = H, **b** R = Br, **c** R = NO_2, **d** R = *t*-Bu

Compounds **3** display photochromic properties in solution. However, as a result of the short life time of the noncyclic isomers **4** under stationary conditions of irradiation, photocoloration is observed at temperatures below 250 K.

The cyclic isomers **3** are characterized by a long-wave structure-linked absorption at 330-380 nm (Table 3). At temperatures of 150-250 K, as a result of photoirradiation of solutions of compound **3**, the appearance was observed in their absorption spectra of bands with a maximum at 585-625 nm, characteristic of the absorption of the noncyclic transoid isomers **4** (see Fig. 1 for the example of compound **3b**). At lower temperatures (77 K) in the case of compound **3d** the appearance of an absorption band is observed initially with a maximum at 471 nm, which on prolonged irradiation is transformed into a band with maximum at 585 nm (Fig. 2). The observed spectral changes are linked with the formation at the first stage of the *cis*-cisoid X-isomer 3', formed directly after fission of the C_{spiro}–O bond and is stabilized as a result of the steric manifestations of the bulky *tert*-butyl group and the viscosity barriers at low temperature, subsequent *cis-trans* photoisomerization leads to the appearance of transoid isomers **4**.

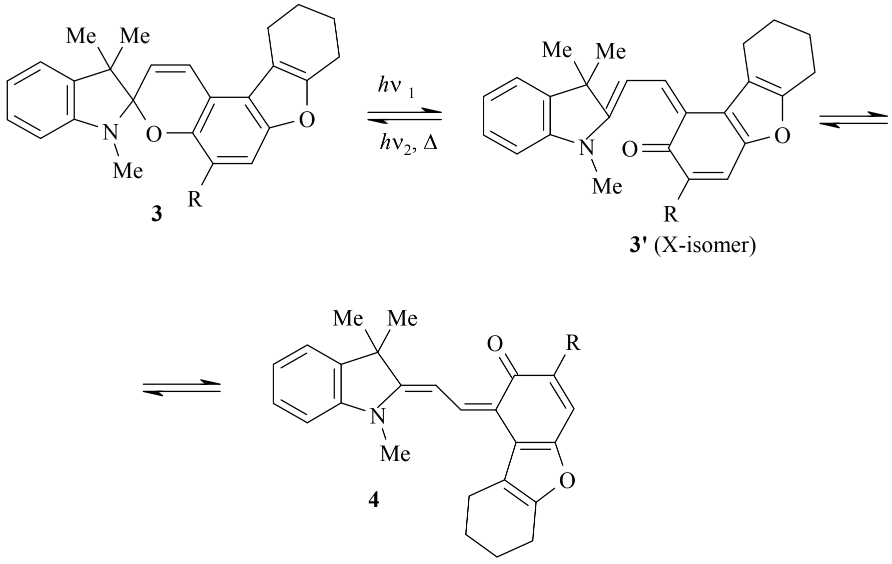


TABLE 1. Spiropyrans **3**

Compound	Empirical formula	Found, % / Calculated, %			mp, °C (from ethanol)	Yield, %
		C	H	N		
3a	$C_{25}H_{25}NO_2$	80.71 80.83	6.82 6.73	3.68 3.77	164-165 (176-178 [6])	72
3b*	$C_{25}H_{24}BrNO_2$	66.50 66.67	5.46 5.33	2.94 3.11	158	83
3c	$C_{25}H_{24}N_2O_4$	72.18 72.14	5.62 5.77	6.87 6.73	139-140	68
3d	$C_{29}H_{33}NO_2$	81.35 81.52	7.65 7.72	3.39 3.28	132	57

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* Found, %: Br 17.43; calculated, %: Br17.74.

TABLE 2. Spectral Characteristics of Spiropyrans **3**

Compound	IR spectrum, $\nu_{C=C}$ of pyrane ring, cm^{-1}	UV spectrum, λ_{max}, nm ($\log \varepsilon$)	^1H NMR spectrum, δ, ppm (*J*,Hz)
3a	1647, 1627, 1600	304 (4.26), 352 (3.50)	1.18 (3H, s, CH_3), 1.33 (3H, s, CH_3), 1.88 (4H, m, H-2",3"), 2.71 (2H, m, H-1"), 2.73 (1H, s, N–CH_3), 2.97 (2H, m, H-4"), 5.68 (1H, d, *J* = 8.6, H-8), 6.50 (1H, d, *J* = 9.00, H-7'), 6.55 (1H, d, *J* = 8.6, H-5), 6.82 (1H, td, *J* = 8.3, H-5'), 7.06 (1H, d, *J* = 8.6, H-4'), 7.08 (1H, d, *J* = 9.75, H-4), 7.16 (1H, td, *J* = 8.3, H-6'), 7.27 (1H, d, *J* = 10.6, H-4)
3b	1644, 1616, 1600	301 (4.26), 385 (3.50)	1.19 (3H, s, CH_3), 1.33 (3H, s, CH_3), 1.90 (4H, m, H-2",3"), 2.67 (2H, m, H-1"), 2.67 (1H, s, N–CH_3), 2.84 (2H, m, H-4"), 5.76 (1H, d, *J* = 10.6, H-8), 6.52 (1H, d, *J* = 8.0, H-7'), 6.83 (1H, td, *J* = 8.8, H-5'), 7.06 (1H, d, *J* = 8.0, H-4'), 7.15 (1H, td, *J* = 8.8, H-6'), 7.22 (1H, d, *J* = 10.6, H-9), 7.37 (1H, s, H-4)
3c	1637, 1611, 1590	305 (4.32), 355 (3.66)	1.18 (3H, s, CH_3), 1.25 (3H, s, CH_3), 1.94 (4H, m, H-2",3"), 2.72 (2H, m, H-1"), 2.62 (1H, s, N–CH_3), 2.84 (2H, m, H-4"), 5.83 (1H, d, *J* = 10.5, H-8), 6.50 (1H, d, *J* = 8.3, H-7'), 6.83 (1H, td, *J* = 8.2, H-5'), 7.14 (1H, td, *J* = 8.3, H-6'), 7.27 (1H, d, *J* = 10.5, H-4), 7.28 (1H, d, *J* = 8.8, H-4'), 7.74 (1H, s, H-4)
3d	1649, 1620, 1605	312 (4.14), 380 (3.95)	1.10 (9H, s, t-C_4H_9), 1.23 (3H, s, CH_3), 1.39 (3H, s, CH_3), 1.88 (4H, m, H-2",3"), 2.65 (1H, s, NCH_3), 2.70 (2H, m, H-1"), 2.81 (2H, m, H-4"), 5.77 (1H, d, *J* = 10.2, H-8), 6.44 (1H, d, *J* = 7.6, H-7'), 6.82 (1H, t, *J* = 7.4, H-5'), 7.10 (1H, d, *J* = 7.6, H-4'), 7.13 (1H, t, *J* = 7.6, H-6'), 7.21 (1H, s, H-4), 7.34 (1H, d, *J* =10.2, H-9)

After the end of UV irradiation of solutions of spiropyrans **3** relaxation processes were observed, leading to their decoloration. The rate of these processes at room temperature was large which does not permit a quantitative description of them. The exception was compound **3c**, for solutions in ethanol the life time of the photoinduced form was 66 sec at 296 K which makes it possible to investigate photoinduced processes under normal conditions (to determine rate constants of the direct and reverse photoreactions and their quantum

TABLE 3. Spectral-absorption and Spectral-luminescent Characteristics of Spiropyrans (mixtures of **3** in IPP at 77 K)

Compound	Form	Absorption, λ_{max}, nm	Fluorescence		Phosphorescence	
			λ_{max}^{ex}, mn	λ_{max}^{flu}, nm	λ_{max}^{ex}, nm	λ_{max}^{ph}, nm
3a	**3**	S_2-301				
		S_1-332	—	—	346	540
		346 sh				585
	4	S_2-468	S_2-470			
		S_1-546	S_1-610	648	—	—
		587		695		
		627				
3b	**3**	S_2-298				
		S_1-380 sh	—	—	382	512
						553
						598
	4	S_2-483	S_2-468			
		S_1-592	S_1-620	656	—	—
		625		710		
3c	**3**	S_2-302				
		S_1-338	S_1-348	374	343	548
		348 sh		394 sh		577
	4	S_2-458	473			
		482				
		S_1-550 sh	550	659	—	—
		585	585	718		
		625 sh	626			
3d	**3**, 290 K	S_2-312	S_2-328			
		S_1-379	S_1-379	547		
	3, 77 K	S_2-312	S_2-328			
		S_1-379	S_1-379	520	—	—
				550 sh		
	3', 77 K	471	468	543		
				572	—	—
	4, 178 K	S_2-470	S_2-468		—	—
		S_1-585	S_1-582	653		
		625 sh	625	698 sh		

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* IPP is a mixture of isopentane—2-propanol, 4 : 1.

yields). The quantum yields were determined on the basis of a photochemical analysis of the experimental dependencies of the optical densities on the time of irradiation according to [6], and amounted to 0.052 and 0.046 respectively for the forward and reverse photoreactions.

The overall rate constant for the dark reaction ($k = k_{BA} + k_{AB}$) was determined from the expression $D = D_0\exp(-kt)$, k_{BA} and k_{AB} were determined at a known value of k within the framework of a photokinetic analysis of the experimental dependencies of the optical densities: $k = 1.6 \times 10^{-2}$, $k_{BA} = 1.5 \times 10^{-2}$, and $k_{AB} = 2.4 \times 10^{-4}\text{sec}^{-1}$.

Compound **3c,** containing a π-acceptor nitro group, displays photochromic properties not only in solution but also in solid polydisperse films obtained by vacuum thermal spray coating on glass or quartz supports (Fig. 3).

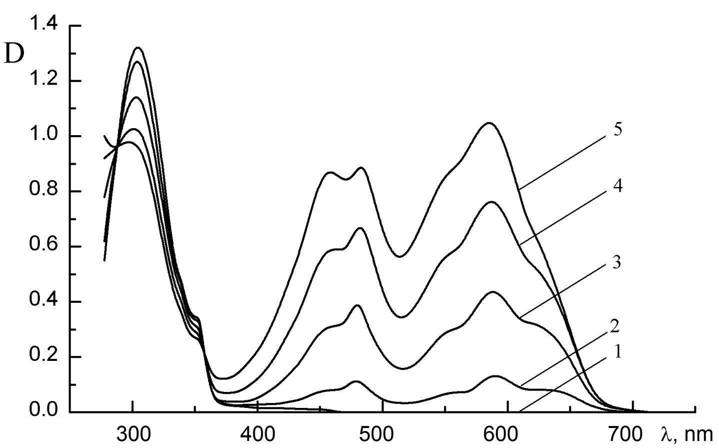

Fig. 1. Dependence of the absorption spectrum of spiropyrans **3b** in IPP at 77 K on the time of irradiation with UV light of λ_{max} 365 nm in the stationary state: 1) 0; 2) 60; 3) 180; 4) 360; 5) 630 sec irradiation.

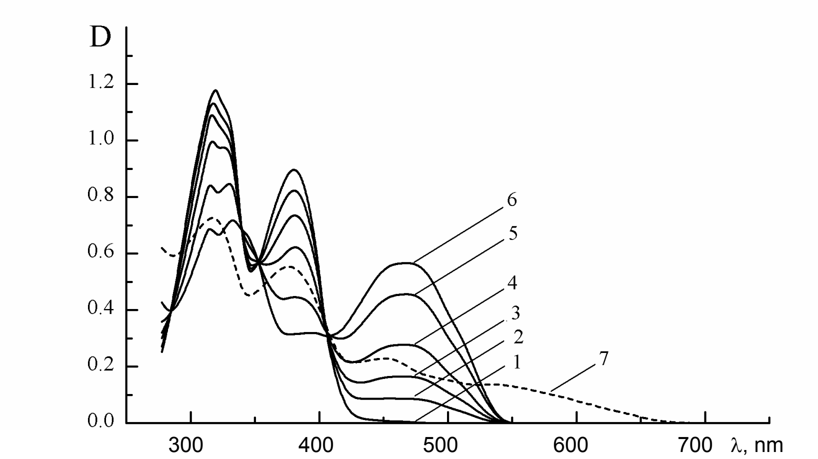

Fig. 2. Dependence of the absorption spectrum of spiropyran **3d** in IPP at 77 K on the time of irradiation with UV light of λ_{max} 365 nm in the stationary state: 1) 0; 2) 60; 3) 120; 4) 420; 5) 600; 6) 900 sec irradiation; 7) subsequent irradiation with unfiltered light for 1800 sec.

With the aim of refining the structure of the obtained compounds and to establish the connection between their structure and photochemical properties, crystals of compounds **3a-c** were grown from ethanol and were investigated by X-ray structural analysis. The overall form of the molecules with numbering of the atoms is given in Figs. 4-6. The main crystallographic characteristics are given in Table 4, values of bond lengths and angles of spiropyrans **3a-c** are given in Tables 5 and 6.

Analysis of the details of the structure afforded the conclusion that the benzopyran and indoline fragments in the **3a-c** molecules, as in the previously investigated indoline spiropyrans and benzoxazinone derivatives of spiropyrans [2, 3, 8], are disposed approximately orthogonally to one another and individually are not planar. In compound **3a** the indoline fragment has a bend along the line $N_{(1')}\cdots C_{(3')}$ of angle Φ = 25.0, in compound **3b** of 29.6, and in **3c** of 25.7°. The atoms $N_{(1')}$ and $C_{(3')}$ are located in the plane of the benzene ring

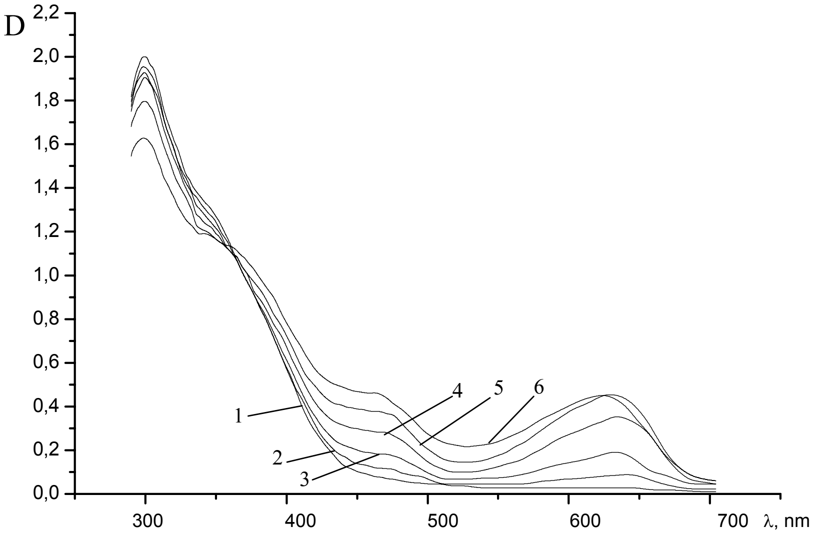

Fig. 3. Dependence of the absorption spectrum of spiropyran **3a**, in a thin polydisperse film obtained by vacuum thermal coating on a quartz plate, on the time of irradiation by UV light of λ_{max} 365 nm in the stationary state: 1) 0; 2) 60; 3) 180; 4) 240; 5) 360; 6) 630 sec irradiation.

TABLE 4. Basic Crystallographic Data and Characteristics of the Analysis of Compounds **3a-c**

Compound	**3a**	**3b**	**3c**
Empirical formula	$C_{25}H_{23}NO_2$	$C_{25}H_{24}BrNO_2$	$C_{26}H_{26}N_2O_4$
M	369.44	450.36	430.49
Space group	*C* 2/*c*	*P*-1	*P*-1
a, Å	23.274(5)	17.935(4)	6.970(1)
b, Å	6.881(1)	8.025(2)	11.063(2)
c, Å	25.066(5)	7.835(2)	14.153(3)
α°	90	73.61(3)	75.89(3)
β°	102.00(3)	86.50(3)	89.41(3)
γ°	90	78.02(3)	79.90(3)
V, $Å^3$	3927(1)	1058.2(4)	1041.4(3)
Z	8	2	2
D, g/cm^3	1.250	1.413	1.373
μ, mm^{-1}	0.620	1.963	0.930
θ	3.61-80.08	1.16-23.05	1.48-27.07
Total number of reflections	3159	2965	3902
Number of reflections $I > 2\sigma(I)$	1926	1907	2155
Number of reflections	254	262	299
R	0.063	0.056	0.069
wR_2	0.115	0.10	0.141
Radiation	CuKα	MoKα	MoKα
GOF	1.094	1.067	0.997

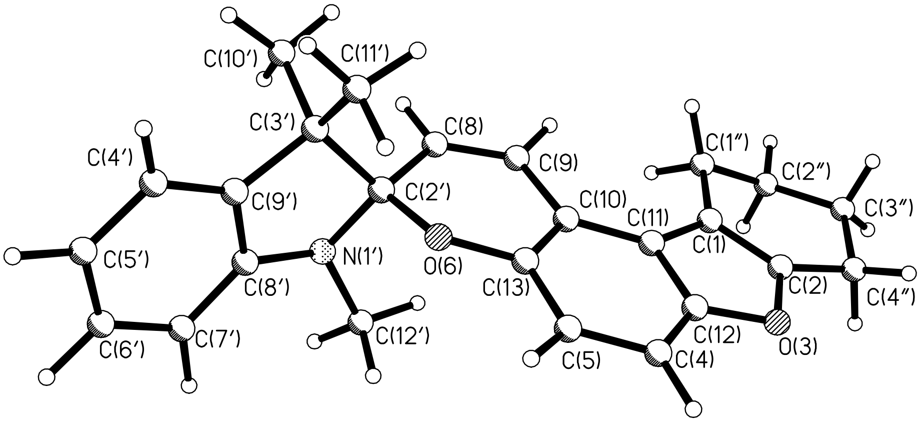

Fig. 4. Structure of spiropyran **3a** according to X-ray structural analysis.

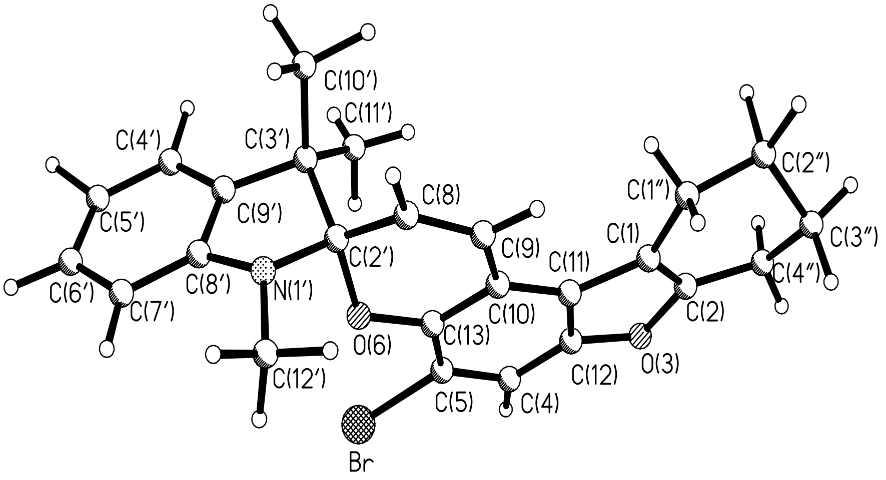

Fig. 5. Structure of spiropyran **3b** according to X-ray structural analysis.

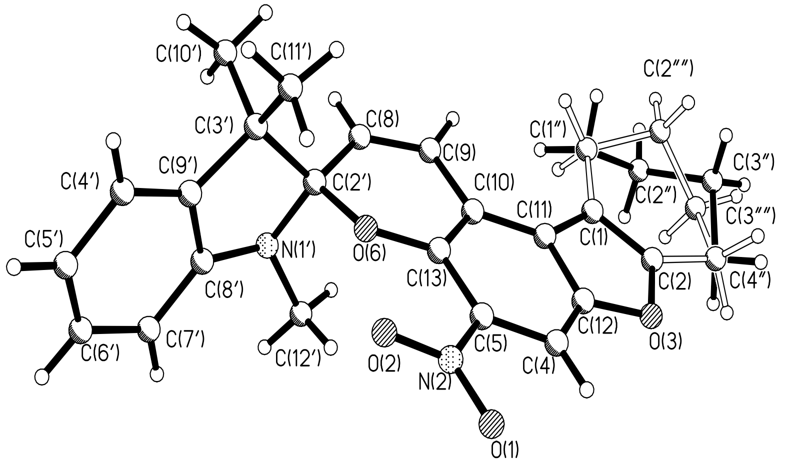

Fig. 6. Structure of spiropyran **3c** according to X-ray structural analysis.

TABLE 5. Bond lengths (*l*) of Spiropyrans **3a-c** according to X-ray Data

Bond	*l*, Å		
	3a	**3b**	**3c**
$C_{(2')}–O_{(6)}$	1.462(3)	1.482(8)	1.485(5)
$C_{(2')}–C_{(8)}$	1.499(4)	1.486(9)	1.489(7)
$C_{(8)}–C_{(9)}$	1.317(4)	1.330(9)	1.315(7)
$C_{(9)}–C_{(10)}$	1.451(4)	1.464(9)	1.444(6)
$C_{(10)}–C_{(13)}$	1.392(4)	1.410(8)	1.397(5)
$O_{(6)}–C_{(13)}$	1.383(4)	1.354(7)	1.350(4)
$C_{(2')}–C_{(3')}$	1.569(4)	1.553(10)	1.561(6)
$N_{(1')}–C_{(2')}$	1.463(4)	1.452(8)	1.460(6)
$N_{(1')}–C_{(12')}$	1.451(4)	1.413(10)	1.443(6)
$N_{(1')}–C_{(8')}$	1.406(4)	1.419(8)	1.385(7)

TABLE 6. Valence Angles (ω) of Spiropyrans 3a-c according to X-ray Data

Angle	ω, deg.		
	3a	**3b**	**3c**
$C_{(9)}N_{(1)}C_{(2'2)}$	108.1(2)	106.1(6)	108.7(3)
$C_{(9)}N_{(1)}C_{(10)}$	120.9(3)	122.3(6)	118.9(4)
$C_{(10)}N_{(1)}C_{(2'2)}$	118.3(3)	120.3(6)	119.7(3)
$N_{(1)}C_{(2'2)}O_{(1')}$	108.8(2)	104.9(5)	108.2(4)
$N_{(1)}C_{(2'2)}C_{(3')}$	111.6(3)	112.5(5)	110.7(3)
$N_{(1)}C_{(2'2)}C_{(3)}$	103.0(2)	103.9(5)	103.4(3)
$C_{(3)}C_{(2'2)}C_{(3')}$	115.2(3)	116.4(5)	117.9(3)
$C_{(3)}C_{(2'2)}O_{(1')}$	106.6(2)	108.8(5)	105.5(3)

$C_{(4')}C_{(5')}C_{(6')}C_{(7')}C_{(8')}C_{(9')}$ with a precision of 0.02, 0.016, and 0.013 Å in the crystals of **3a,b** and **c** respectively (all the geometric details of the molecular structures will be given and discussed in this sequence). While analyzing the structure of the benzospiran fragment we first noticed that the $O_{(6)}C_{(9)}C_{(10)}C_{(13)}C_{(5)}C_{(4)}C_{(12)}C_{(11)}O_{(3)}C_{(1)}C_{(2)}$ atoms lie in one plane with a precision of 0.016, 0.04, and 0.016 Å in the crystals of compounds **3a,b** and **c**. The bends at the lines passing through the $O_{(6)}C_{(9)}$ and $O_{(6)}C_{(8)}$ atoms are equal to 7.5 and 11.5, 11.0 and 33.0, and 8.1 and 13.3° respectively. Attention is attracted by the bend of 33.0° at the $O_{(6)}C_{(8)}$ line in **3b**, formally equal to 147°. In other words the mutual orientation of the two fragments relative to the $O_{(6)}C_{(8)}$ line in the investigated molecules **3a** and **3c**, in which the benzospirane fragment is bent back to the side of the N-methyl substituents, is different from that in **3b**, where a bend to the side of the geminal carbon atoms occurs. Altogether the geometric structures of the C_{spiro} unit and the benzopyran fragment in compounds **3a-c** are analogous to the structure of the indoline and benzoxazine spiropyrans studied previously. The emergence of the $N_{(1')}$ atom from the plane of the coordinating atoms $C_{(2'2)}C_{(8')}C_{(12')}$ was -0.30, 0.27, and -0.30 Å, the total valence angles at $N_{(1')}$ were 347.3, 349.3, and 347.3° respectively. The marked sp^3-character of the hybridization of the orbitals of the $N_{(1')}$ atom leads, apart from the bend at the $C_{(3')}\cdots N_{(1')}$ line of the indoline ring, to the torsion turn around the $C_{(8')}–N_{(1')}$ bond of the $C_{(2'2)}$ atom (angle $C_{(9')}C_{(8')}N_{(1')}C_{(2'2)}$ is equal to 18.8, -20.6, and 17.4) and to the simultaneous turn of the methylene group (atom $C_{(12')}$) around this bond (angle $C_{(9')}C_{(8')}N_{(1')}C_{(12)}$ is equal to 159.7, -165.0, and 159.1°). In difference to the benzoxazine analogs [2, 7], the absence of amide conjugation assists the interaction of the unshared electron pair of the $N_{(1')}$ atom with the σ*-orbital of the $C_{(2'2)}–O_{(6)}$ bond, and furthermore the geometric disposition of the $C_{(2'2)}–O_{(6)}$ bond relative to the coordinated plane $C_{(9')}–C_{(8')}–C_{(2'2)}$ of the $N_{(1')}$ atom remains more due to the *n*-σ*-orbital interactions. It is characteristic of spiropyrans of this type that under conditions of domination at the spiro center of the molecule of *n*-σ*-orbital interactions of the lone electron pair of the N atom with the σ*-orbitals of the $C_{(2'2)}$–$O_{(6)}$ bond, there will be noticeable shortening of the bond of N with the C_{spiro} atom and lengthening of the $O_{(6)}$–C_{spiro} bond compared with standard values for similar bond lengths. Thus the bond lengths for $C_{(2'2)}$–$O_{(6)}$ = 1.462(4), 1.483(8), and 1.476(4), and for $N_{(1')}$–$C_{(2'2)}$ = 1.463(4), 1.451(8), and 1.444(5) Å are within the range of bond lengths for previously studied indoline spiropyrans displaying photochromic properties (1.438-1.497 and 1.434-1.453 Å respectively) [8].

It should be noted that replacement of a π-donor by a condensed furan fragment did not change the picture of electronic interactions characteristic of indoline spiropyran molecules. Thus the degree of conjugation of the $O_{(6)}$ of the molecules with the π-orbital of the benzene ring is analogous to the previously investigated spiropyran and the $O_{(6)}$–$C_{(13)}$ bond length is 1.382(4) Å [8]. The presence in position 5 of the molecules of **3b,c** of a strong electron-withdrawing substituent, as in the benzoxazine spiropyrans, leads to a change in the electron density on the oxygen atom of the spirocenter and a strengthening of the degree of coordination of $O_{(6)}$ with the benzene fragment. As a result, in the crystals of **3b,c** the $O_{(6)}$–$C_{(13)}$ bond length is reduced and is 1,354(7) and 1.352(3) Å, which accompanies the lengthening of the C_{spiro}–$O_{(6)}$ bond to 1.483(8) and 1.476(4) Å.

EXPERIMENTAL

The IR spectra were recorded on a Specord prism double beam IR 75 spectrometer in nujol, the ^{1}H NMR spectra on a Varian Unity 300 (300 MHz) instrument in $CDCl_3$. Assignment of signals was carried out relative to the $CDCl_3$ signal (δ 7.26 ppm). The electronic spectra of the studied compounds were recorded at room temperature on a Specord M 40 spectrophotometer, and the absorption spectra of the spiropyrans before and after irradiation on a Specord UV-vis spectrophotometer with a cryostat at 77 K for low temperature measurements in a mixture of isopentane–2-propanol, 5:2.

The X-ray structural analysis of compounds **3a-c** was carried out on a four-circle KUMA automatic diffractometer at T = 293 K. Structures were found by the direct method and were refined by the least squares method in an anisotropic approximation with the set of programs SHELX-97 [9]. The main crystallographic data are given in Table 4. Some of the H atoms were localized in Fourier syntheses by the difference of electron density, the positions of the remaining H atoms were calculated. The rider model was used for structure **3c** when refining the positions of the hydrogen atoms, since randomization of the aliphatic atoms $C_{(2'')}$ and $C_{(3'')}$ at equipotential positions relative to the $C_{(1'')}$ and $C_{(4'')}$ atoms in a ratio of 1:1 was observed in it.

Spiropyrans 3. Piperidine (0.85 g, ~1 ml, 0.01 mol) was added to a boiling mixture of 1,2,3,3-tetramethylindolenium perchlorate (**1**) (2.74 g, 0.01 mol) and the appropriate aldehyde **2** (0.01 mol) in 2-propanol (20 ml). The reaction mixture was heated for a short time, cooled, poured into water (60 ml), and extracted with benzene. The organic layer was washed with water, dried over Na_2SO_4, evaporated in vacuum to a volume of 10-15 ml, and chromatographed on a column of Al_2O_3 (eluent benzene). The residue was chromatographed on neutral Al_2O_3 (Brockmann activity grade 2, eluent chloroform) and recrystallized from ethanol. The characteristics of the compounds obtained are given in Table 1.

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