

112 mass spectrometer (70 eV) with direct introduction of the samples into the ion source. The individuality of the substances was monitored on Silufol UV-254 plates in chloroform with development in UV light.

2-Methyl-3-ethoxycarbonyl-4,5-dihydroxy-6-chloro-7-(1'-ethoxycarbonyl-2'-oxopropyl)benzofuran (IX). A mixture of 13.4 g (0.05 mole) of 2-methyl-3-ethoxycarbonyl-4,5-dioxo-4,5-dihydro-6-chlorobenzofuran (I), 28g (0.1 mole) of acetoacetic ester (VII), and 26 g (0.1 mole) of anhydrous zinc chloride in 500 ml of absolute ethanol was refluxed with stirring for 15 min, after which the solution was cooled to 40°C and treated with 20 ml of a solution of Na₂S₂O₄ and 250 ml of water to decolorize the colored impurities. The resulting precipitate was removed by filtration, washed with water, and dried to give 16 g (80%) of product. Compounds X-XVII (Table 1) were obtained by a similar method.

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SYNTHESIS OF INDENOINDOLES ON THE BASIS OF

2-AMINOFLUORENE AND 2-AMINOFLUORENONE

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UDC 547.759.3'665.07:543.422

Ethyl esters of 2-carboxylic acids of 3H,10H-indeno[2,1-e]-1H,9H-indeno[1,2-f]indoles and their keto derivatives were obtained on the basis of 2-aminofluorene and its 9-keto derivative via the Japp-Klingemann and Fischer reactions. Saponification with subsequent decarboxylation of these products leads to unsubstituted cyclic systems and their keto derivatives.

Despite the large volume of research devoted to the synthesis and study of the properties of new heterocyclic systems that contain an indole fragment, only several derivatives of 3H,10H-indeno[2,1-e]- and 1H,9H-indeno[1,2-f]indoles have been described in the literature [1, 2]. In the present research we made an attempt to synthesize these compounds on the basis of the readily accessible fluorene derivatives 2-aminofluorene and 2-aminofluorenone via the scheme presented below.

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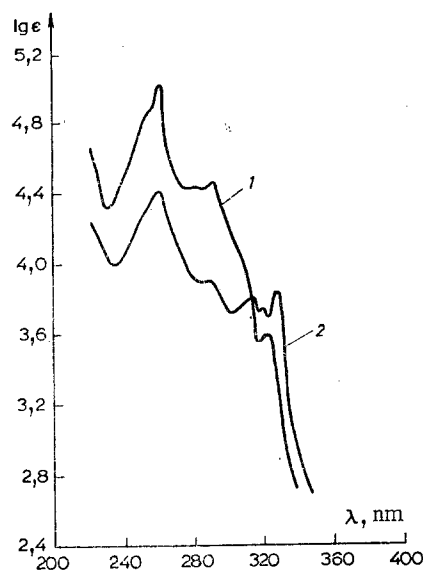


Fig. 1

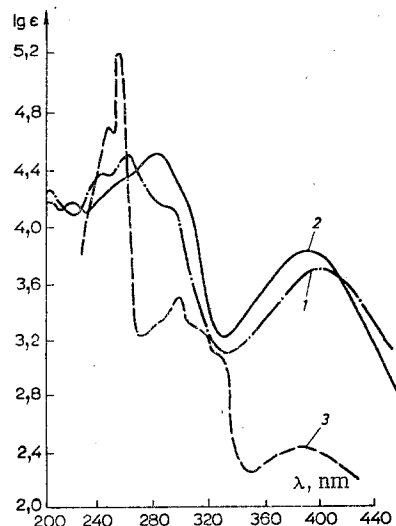
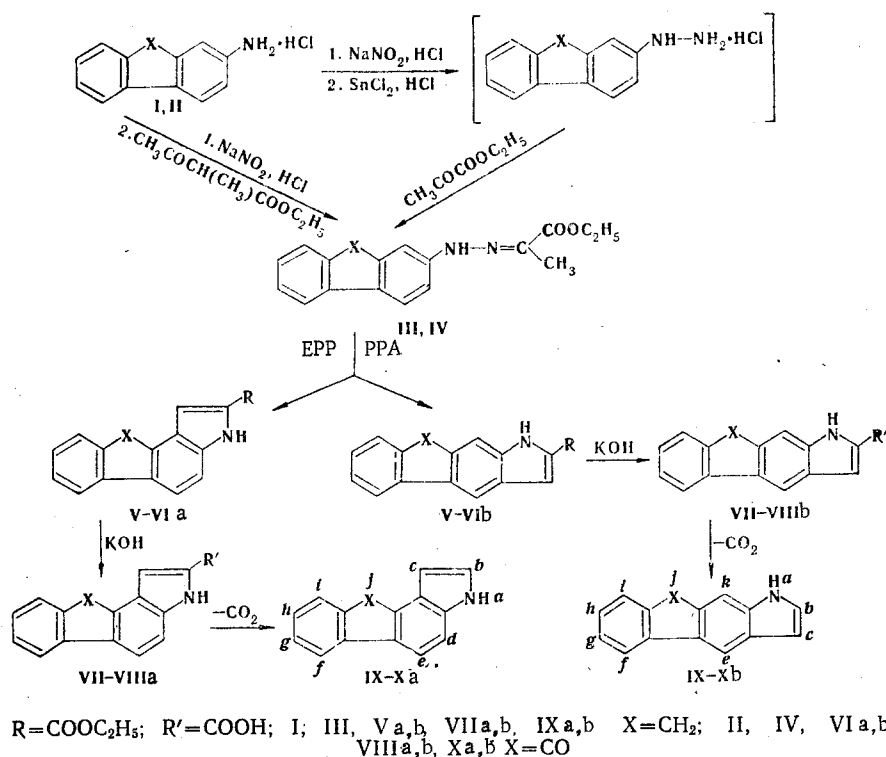


Fig. 2

Fig. 1. UV absorption spectra in alcohol: 1) 3H,10H-indeno[2,1-e]indole (IXa); 2) 1H,9H-indeno[1,2-f]indole (IXb).

Fig. 2. UV absorption spectra in alcohol: 1) 3H,10H-indeno[2,1-e]indol-10-one (Xa); 2) 1H,9H-indeno[1,2-f]-indol-9-one (Xb); 3) fluorenone.



Starting aminofluorenes I and II were obtained from fluorene via known methods [3, 4]. Diazotization of hydrochloride I and subsequent reduction of the reaction product with stannous chloride gave 2-fluorenylhydrazine hydrochloride, which reacts with ethyl pyruvate to give ethyl pyruvate 2-fluorenylhydrazone (III) in the form of a mixture of geometrical isomers.

In view of the presence of a carbonyl group in amine II, it did not seem possible to obtain hydrazone IV via a similar route. It was therefore synthesized by the reaction of 9-oxo-

TABLE 1. Chemical Shifts (δ , ppm) and SSCC (J, Hz) of Cyclic Compounds Obtained on the Basis of 2-Aminofluorene*

Compound	Chemical shifts, δ , ppm									J, Hz
	a-H	b-H	c-H	d-H	e-H	f-H	g-i-H	j-H	k-H	
Va	11,8 br s	—	7,2 [†]	7,43 dd	7,73 dd	7,72 m	7,1—7,3	4,00 s	—	$J_{CH_2, CH_3}=7,1$; $J_{d,e}\approx 8,8$;
Vb	11,6 br s	—	7,1 [†]	—	7,57 br s	7,79 m	7,2—7,5	3,94 s	8,01 br s	$J_{f,g}=7,7$; $J_{e,f}\approx 1$
VIIa	11,6 br s	—	7,10 br s	7,42 dd	7,67 br s	7,60 m	7,2—7,4	3,98 s	—	$J_{CH_2, CH_3}=7,1$ $J_{d,e}=8,4$; $J_{f,g}=7,9$
VIIb	11,2 br s	—	6,87 br s	—	7,79 br s	7,58 m	7,0—7,3	3,74 s	7,36 br s	$J_{f,g}=7,2$; $J_{f,h}\approx 1$
IXa	11,0 br s	7,30 dd	6,48 m	7,36 dd	7,55 dd	—	7,1—7,8	3,94 s	—	$J_{d,e}=8,7$; $J_{a,b}=2,7$; $J_{b,c}=2,9$; $J_{a,c}=2,1$; $J_{c,d}=0,8$
IXb	11,0 br s	7,30 dd	6,49 dd	—	7,50 br s	—	7,0—7,9	3,94 s	7,43 br s	$J_{a,b}=2,6$; $J_{b,c}=3,0$; $J_{a,c}=2,0$

*For convenience, the letter designations of the protons are introduced here (see the reaction scheme).

†The signal is found in the aromatic proton region.

TABLE 2. Chemical Shifts (δ , ppm) and SSCC (J, Hz) of Cyclic Compounds Obtained on the Basis of 2-Aminofluorenone*

Compound	Chemical shifts, δ , ppm										J, Hz
	a-H	b-H	c-H	d-H	e-H	f-H	g-H	h-H	i-H	kj-H	
VIa [†]	12,15 br s	—	7,23 d	7,61 s	7,61 s	7,42 m	7,18 br s	7,43 t	7,57 m	—	$J_{CH_2, CH_3}=7,3$; $J_{a,c}=2,0$; $J_{f,g}=J_{g,h}=J_{h,i}=7,8$
VIIb [†]	12,34 br s	—	7,11 d	—	7,61 br s	7,50 m	7,23 br s	7,48 br s	7,71 m	7,89 s	$J_{CH_2, CH_3}=7,3$; $J_{a,c}=2,0$; $J_{e,i}\approx 0,6$; $J_{f,g}=J_{g,h}=7,3$; $J_{g,i}\approx 0,5$; $J_{h,i}=7,8$; $J_{a,c}=1$
VIIIa	11,9 br s	—	7,23 br s	7,35 dd	7,54 dd	—	7,0—7,6	—	—	—	$J_{d,e}=8,5$
VIIIb	12,0 br s	—	7,06 br s	—	7,64 br s	—	7,0—7,8	—	7,87 s	—	
Xa	11,2 br s	7,37 dd	6,70 m	7,35 dd	7,54 dd	—	7,0—7,6	—	—	—	$J_{d,e}=8,5$; $J_{a,b}=2,5$; $J_{b,c}=3,1$; $J_{a,c}=2,0$
Xb	11,3 br s	7,42 dd	6,46 dd	—	7,58 br s	—	7,1—7,7	—	7,75 s	—	$J_{a,b}=2,6$; $J_{b,c}=3,4$; $J_{a,c}=2,5$; $J_{e,i}\approx 0,5$

*For convenience the letter designations of the protons (see the reaction scheme) are introduced here.

†The spectra were recorded with a Bruker WM-250 spectrometer.

fluorenediazonium chloride with methylacetoacetic ester via the Japp-Klingemann reaction [5].

A study of the cyclization reactions of hydrazones III and IV showed that the best results in the case of hydrazone III are obtained by cyclization of the latter in the presence of ethyl polyphosphate (EPP) at 75–90°C; a mixture of two isomeric ethyl 3H,10H-indeno[2,3-e] and 1H,9H-indeno[1,2-f]indole-2-carboxylates (Va,b) in a ratio of 1:1 is formed. The overall yield is 33%.

As a consequence of the inactivating effect of the keto group, hydrazone IV displays a certain inertness and does not undergo cyclization under similar conditions. However, the use of a more active cyclizing agent [polyphosphoric acid (PPA)] and raising the temperature (105–120°C) made it possible to obtain a mixture (1:2) of ethyl 10-oxo-3H,10H-indeno[2,1-e] and 9-oxo-1H,9H-indeno[1,2-f]indole-2-carboxylates (VIIa,b) in 68% overall yield. The ratios of the angular Va and VIa and linear Vb and VIIb isomers of the ethyl esters of the 2-carboxylic acids of the indenoindoles were determined by PMR spectroscopy.

Saponification of individual esters Va,b and VIIa,b with aqueous alcoholic alkali gave the corresponding 2-carboxylic acids VIIIa,b and VIIIa,b, the decarboxylation of which led to the formation of cyclic compounds that do not contain substituents in the pyrrole ring (IXa,b and Xa,b).

The UV spectra of the angular and linear isomers of these compounds differ (Figs. 1 and 2). Thus the presence of a whole series of maxima in the long-wave region that are absent in the spectrum of its angular analog IXa is characteristic for IXb (Fig. 1). In addition, for all of the structures obtained on the basis of 2-aminofluorenone (for example, Xa,b; Fig. 2) there is a long-wave maximum at 390-400 nm, which is characteristic for the spectrum of fluorenone.

The spectrum of an AB system of protons with spin-spin coupling constant (SSCC) $J = 8.5$ - 8.8 Hz, which is characteristic for ortho-oriented protons, is observed in the PMR spectra of indenoindoles IXa and Xa, and this makes it possible to classify these compounds as isomers of the angular type, whereas the very weak spin-spin coupling of the para-oriented protons with SSCC $J \approx 1$ Hz indicates the linear structure of IXb and Xb. A spectrum of an A_2 system of ortho-oriented protons is observed for Va, and this does not make it possible to unambiguously determine the direction of closing of the pyrrole ring; however, the following derivatives of this series, VIIa and IXa, have a clearly expressed AB system of these protons with SSCC $J = 8.5$ Hz, which once again confirms the angular makeup of these chemical structures.

EXPERIMENTAL

The course of the reactions and the purity of the compounds obtained were monitored by thin-layer chromatography (TLC) (Silufol UV-254). The IR spectra were recorded with a UR-20 spectrometer with NaCl and LiF prisms (for mineral oil suspensions); the scanning rate was 160 cm^{-1} vis-à-vis a spectral slit width of 4 cm^{-1} . The UV spectra of solutions in alcohol were recorded with a Specord UV-vis spectrophotometer. The PMR spectra* were obtained with a Varian CFT-20 spectrometer (80 MHz) with tetramethylsilane as the internal standard. The mass spectra were recorded with an MKh-1303 spectrometer with direct introduction of the samples into the ion source; the cathode emission current was $1.25\text{ }\mu\text{A}$ at an ionizing-electron energy of 50 eV.

Ethyl 3H,10H-Indeno[2,1-e]- and 1H,9H-Indeno[1,2-f]indole-2-carboxylates (Va,b). A solution of 2.21 g (0.032 mole) of NaNO_2 in 10 ml of water was added dropwise in the course of 15 min to a cooled (to 0 - 5°C) mixture of 6.96 g (0.032 mole) of 2-aminofluorene hydrochloride, 75 ml of water, and 20 ml of concentrated HCl, and the reaction mixture was maintained at this temperature for 1 h. A solution of 12.8 g (0.064 mole) of stannous chloride in 60 ml of concentrated HCl was then added dropwise at the same temperature to the resulting suspension of the diazonium salt, and the mixture was stirred for 3 h. The precipitated hydrazine hydrochloride was removed by filtration and dissolved in 1 liter of boiling water. The solution was filtered and treated with a saturated solution of CH_3COONa to bring the pH up to three, after which a solution of 3.52 ml (0.032 mole) of ethyl pyruvate in 4 ml of alcohol was added gradually with stirring at room temperature, and the mixture was allowed to stand for 5-6 h. The precipitated yellow hydrazone was removed by filtration, washed with water, and dried to give 4.37 g (46%) of crude ethyl pyruvate 2-fluorenylhydrazone (III).

The resulting hydrazone III was stirred at 75 - 90°C with 45 g of EPP, after which the mixture was cooled and poured over ice. The resulting precipitate was removed by filtration, washed with water, and dried. The mixture of esters was separated with a column filled with 100/160 μ silica gel by elution with benzene-petroleum ether (1:1).

Chromatography yielded 0.35 g (8%) of ester Va, 1.2 g (29%) of a mixture of esters Va,b, and 0.4 g (10%) of ester Vb. Ester Va had mp 206 - 208°C and R_f 0.50 [ether-petroleum ether (1:1)]. IR spectrum: 3348 (NH) and 1659 cm^{-1} (C=O). Ester Vb had mp 245 - 247°C and R_f 0.45 [ether-petroleum ether (1:1)]. IR spectrum: 3330 (NH) and 1695 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 208 (4.84), 267 (4.75), 277 (4.78), and 312 nm (4.35). The m/z value for the M peak was 277. Found for the Va,b mixture: C 77.8; H 5.5; N 5.0%. $\text{C}_{18}\text{H}_{15}\text{NO}_2$. Calculated: C 77.9; H 5.5; N 5.1%; M 277.

Ethyl 10-Oxo-3H,10H-indeno[2,1-e]- and 9-Oxo-1H,9H-indeno[1,2-f]indole-2-carboxylates (VIa,b). A solution of 2.21 g of NaNO_2 (0.032 mole) in 10 ml of water was added in the course of 15 min to a cooled (to 5 - 10°C) mixture of 7.41 g (0.032 mole) of 2-aminofluorenone hydrochloride (II), 32 ml of water, 12.8 ml of concentrated HCl, and 64 ml of acetic acid, and the reaction mixture was maintained at this temperature for ~ 1 h (reagent No. 1). A solution of 4.61 ml (0.032 mole) of methylacetoacetic ester in 32 ml of isopropyl alcohol was cooled to 0 - 5°C , a solution of 1.92 g of KOH in 12 ml of water cooled to this temperature was added,

*We thank L. N. Kurkovskaya for her assistance in interpreting the PMR spectra.

and reagent No. 1 was then poured into this mixture rapidly with vigorous stirring. The reaction mixture was stirred at 0-5°C for ~2 h and allowed to stand at room temperature for 24 h. It was then diluted with 1 liter of water, and the resulting ethyl pyruvate 9-oxo-2-fluorenylhydrazine was removed by filtration, washed with water, and dried to give 8.83 g (90%) of brick-red crude hydrazone IV.

Hydrazone IV was stirred with 90 g of PPA at 105-120°C for 45 min, after which the mixture was cooled and poured over ice. The precipitate was removed by filtration, washed with water, and dried. The mixture of esters was separated with a column filled with 100/160 μ silica gel by elution with benzene. This procedure gave 2.32 g (28%) of ester VIa with mp 271-272°C and R_f 0.59 (ether). IR spectrum: 3312 (NH); 1705, 1712 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 204 (4.26), 2.63 (4.52), 308 (4.70), 357 (3.95), and 385 nm (4.04). Found: C 74.6; H 4.9; N 4.9%; M^+ 291. $\text{C}_{18}\text{H}_{13}\text{NO}_3$. Calculated: C 74.2; H 4.5; N 4.8%. Also obtained was 4.21 g (50%) of ester VIb with mp 246-247°C and R_f 0.52 (ether). IR spectrum: 3300 (NH); 1700, 1718 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 204 (4.34), 256 (4.80), 265 (4.92), and 385 nm (4.04). Found: C 73.9; H 4.8; N 4.8%; M^+ 291. $\text{C}_{18}\text{H}_{13}\text{NO}_3$. Calculated: C 74.2; H 4.5; N 4.8%; M 291.

3H,10H-Indeno[2,1-e]indole-2-carboxylic Acid (VIIa). A 0.7-g (2.5 mmole) sample of ester Va was dissolved by heating in 50 ml of propanol, a solution of 0.42 g (7.5 mmole) of KOH in 5 ml of water was added at 40°C, and the mixture was stirred at this temperature for 2 h. It was then diluted with 200 ml of water, the mixture was cooled to 0-5°C, and a 10% aqueous solution of HCl was added slowly until crystalline acid VIIa began to precipitate. The mixture was allowed to stand in the cold for 2 h, after which the precipitated crystals were removed by filtration, washed with water, and dried to give 0.55 g (87%) of VIIa with mp 278-280°C. IR spectrum: 3350 (NH) and 1711 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 206 (4.55), 254 (4.45), 256 (4.79), and 296 nm (4.46). Found: C 77.2; H 4.6; N 5.6%; M^+ 249. $\text{C}_{16}\text{H}_{11}\text{NO}_2$. Calculated: C 77.1; H 4.5; N 5.6%; M 249.

1H,9H-Indeno[1,2-f]indole-2-carboxylic Acid (VIIf). This compound was obtained from ester Vb by a method similar to that used to prepare VIIa. The yield of acid VIIf, which melted above 270°C (with decomposition), was 0.48 g (76%). IR spectrum: 3348 (NH) and 1711 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 209 (4.44), 217 (4.30), 265 (4.42), 274 (4.66), 328 (4.15), and 342 nm (4.18). Found: C 77.3; H 4.6; N 5.7%; M^+ 249. $\text{C}_{16}\text{H}_{11}\text{NO}_2$. Calculated: C 77.1; H 4.5; N 5.6%; M 249.

10-Oxo-3H,10H-indeno[2,1-e]indole-2-carboxylic Acid (VIIIa). A hot solution of 3 g of KOH in 10 ml of water was added with stirring to a refluxing suspension of 2.32 g (0.008 mole) of ester VIa in 50 ml of propanol, after which the mixture was cooled to 0-5°C, and a 10% aqueous solution of HCl was added slowly until crystalline acid VIIIa began to precipitate. The mixture was maintained at this temperature for 2 h, after which it was filtered, and the precipitate was washed with water until the wash waters were neutral. It was then dried to give 1.98 g (94%) of acid VIIIa with mp 322-324°C. IR spectrum: 3360 (NH); 1698, 1711 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 204 (4.37), 256 (4.80), 297 (4.19), 322 (4.27), and 400 nm (3.95). Found: C 73.2; H 3.6; N 6.2%; M^+ 263. $\text{C}_{16}\text{H}_9\text{NO}_3$. Calculated: C 73.0; H 3.5; N 5.3%; M 263.

9-Oxo-1H,9H-indeno[1,2-f]indole-2-carboxylic Acid (VIIf). This compound was obtained from ester VIb by a method similar to that used to prepare VIIIa. The yield of acid VIIf, which melted above 300°C (with decomposition), was 2.02 g (96%). IR spectrum: 3380 (NH); 1682, 1700 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 204 (4.29), 260 (4.47), 303 (4.71), and 395 nm (4.02). Found: C 72.4; H 3.1; N 5.1%; M^+ 263. $\text{C}_{16}\text{H}_9\text{NO}_3$. Calculated: C 73.0; H 3.5; N 5.3%; M 263.

3H,10H-Indeno[2,1-e]indole (IXa). A 0.55-g (2.1 mmole) sample of acid VIIa was decarboxylated thermally at 290°C in the course of 20 min. After the mixture was cooled, IXa was purified with a column filled with 100/160 μ silica gel by elution with benzene to give 0.16 g (38%) of a product with mp 133-135°C. IR spectrum: 3408 cm^{-1} (NH). UV spectrum, λ_{max} (log ϵ): 261 (4.91); 282 (4.43); 292 (4.46); 323 nm (3.59). Found: C 87.8; H 5.4; N 6.9%; M^+ 205. $\text{C}_{15}\text{H}_{11}\text{N}$. Calculated: C 87.8; H 5.4; N 6.8%; M 205.

1H,9H-Indeno[1,2-f]indole (IXb). This compound was obtained from acid VIIf by a method similar to that used to prepare IXa. The yield of IXb, with mp 147-149°C, was 0.28 g (65%). IR spectrum: 3401 cm^{-1} (NH). UV spectrum, λ_{max} (log ϵ): 262 (4.42); 292 (3.89), 314 (3.77), 322 (3.73), and 329 nm (3.84). Found: C 87.9; H 5.5; N 6.8%; M^+ 205. $\text{C}_{15}\text{H}_{11}\text{N}$. Calculated: C 87.8; H 5.4; N 6.8%; M 205.

3H,10H-Indeno[1,2-f]indol-10-one (Xa). This compound was obtained from acid VIIIA by a method similar to that used to prepare IXa. The yield of Xa, with mp 231-232°C, was 0.22 g (49%). IR spectrum: 3341, 3270 (NH); 1692 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 206 (4.28), 243 (4.41), 263 (4.53), and 398 nm (3.71). Found: C 82.9; H 4.2; N 6.4%; M^+ 219. $\text{C}_{15}\text{H}_9\text{NO}$. Calculated: C 82.2; H 4.1; N 6.4%; M 219.

1H,9H-Indeno[1,2-f]indol-9-one (Xb). This compound was obtained from acid VIIIB by a method similar to that used to prepare IXa. The yield of Xb, with mp 249-250°C, was 0.15 g (33%). IR spectrum: 3270, 3340 (NH); 1700 cm^{-1} (C=O). UV spectrum, λ_{max} (log ϵ): 204 (4.20), 224 (4.17), 254 (4.35), 283 (4.52), and 392 nm (3.84). Found: C 82.2; H 4.2; N 6.3%; M^+ 219. $\text{C}_{15}\text{H}_9\text{NO}$. Calculated: C 82.2; H 4.1; N 6.4%; M 219.

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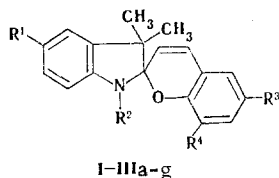
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SYNTHESIS AND PHOTOCHROMIC PROPERTIES OF INDOLINOSPIROCHROMENES WITH BENZYL, ETHYL, AND ACETONYL GROUPS ATTACHED TO THE NITROGEN ATOM

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A number of photochromic indoline spirochromenes with benzyl, acetonyl, and ethyl substituents attached to the nitrogen atom in the indoline part of the molecule were synthesized. The introduction of a benzyl group at the nitrogen atom in place of a methyl group does not change the rate constants of the dark decolorization of the photomerocyanines, whereas an acetonyl group increases them, and an ethyl group decreases them. The introduction of the indicated substituents in the 1 position gives rise to a bathochromic shift (up to 18 nm) of the long-wave absorption band of the merocyanine form of the spirochromene.

Substituents attached to the nitrogen atom have a substantial effect on the photochromic properties of indolinospirochromenes [1]. In the present research we studied this effect for a series of new compounds with benzyl, ethyl, and acetonyl substituents attached to the nitrogen atom.



I $R^1=\text{H}$, $R^2=\text{CH}_2\text{C}_6\text{H}_5$; II $R^1=\text{H}$, $R^2=\text{CH}_2\text{COCH}_3$; III $R^1=\text{C}_6\text{H}_5$, $R^2=\text{C}_2\text{H}_5$; I-III
a $R^3=\text{NO}_2$, $R^4=\text{H}$; b $R^3=\text{NO}_2$, $R^4=\text{OCH}_3$; c $R^3=\text{NO}_2$, $R^4=\text{Br}$; d $R^3=R^4=\text{NO}_2$; e $R^3=\text{H}$,
 $R^4=\text{NO}_2$; f $R^3=\text{OCH}_3$, $R^4=\text{NO}_2$; g $R^3=\text{Br}$, $R^4=\text{NO}_2$

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