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PHOTO- AND THERMOCHROMIC SPIRANS. 35.* SYNTHESIS AND PHOTOCHROMIC PROPERTIES OF SPIRO[INDOLINE-2,3'-PYRANO[3,2-*f*]QUINOLINES] AND THEIR CATIONIC DERIVATIVES

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New photochromic spiropyranoquinolines and their cationic derivatives have been synthesized. Quaternization of the quinoline fragment leads to significant enhancement of the thermal stability of the merocyanine isomers of these cationic spiropyrans.

Keywords: 6-hydroxyquinoline-5-carbaldehyde, merocyanines, spiropyran salts, spiropyrano-quinolines, photochromism.

There is considerable present interest in the design and study of new and efficient photochromic systems with the aim of creating polyfunctional materials for molecular electronics [2-4]. The relatively easily-synthesized spiropyrans have special significance among the reported classes of photochromic compounds. Depending on their molecular structures, these compounds display a broad range of spectrokinetic characteristics [2-5].

The synthesis, structure, and photochemical properties of crystals of spiropyran and spirooxazine salts have attracted special attention [6-9]. Cationic spiropyrans SP^+X^- hold promise for use in the preparation of hybrid polyfunctional materials combining two sublattices in a single crystal lattice: a photochromic sublattice of a spiropyran or spirooxazine cation [10] and a magnetic anionic sublattice of mono- or bimetallic (tris)oxalates [6, 11-17], in which the spiro compound acts as a photochemical molecular switch, perturbing the magnetic sublattice.

These findings have led to the study of new cationic spiropyran and spirooxazine derivatives [7,18-22] for the design of photochromic magnetic materials.

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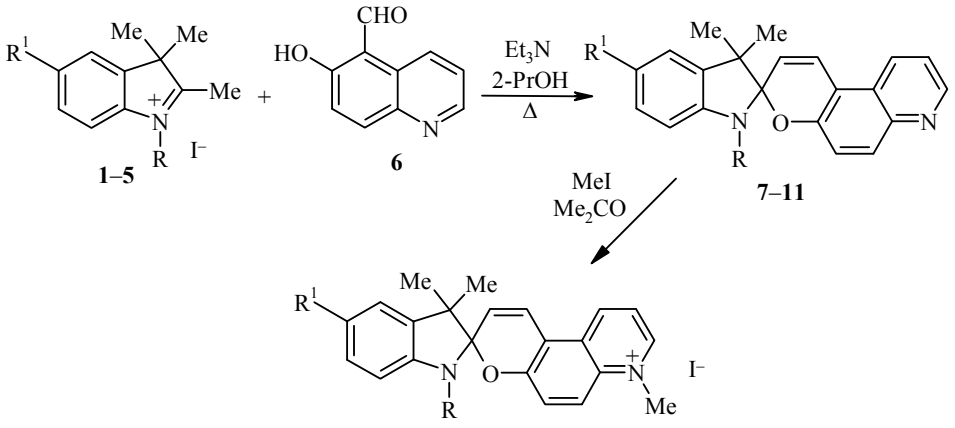
In a continuation of these studies, we have investigated the synthesis and photochromic properties of a series of quinolinespiropyranindolines and their cationic analogs.

The reaction of 3*H*-indolium iodides **1-5** with 5-formyl-6-hydroxyquinoline (**6**) in the presence of base and subsequent alkylation of the resultant spiro[indoline-2,3'-pyranoquinolines] **7-11** by methyl iodide gave cationic spiropyrans **12-16** containing a quaternized pyridine fragment in a conjugated ring system.



1–5 **6** **7–11** **12–16**

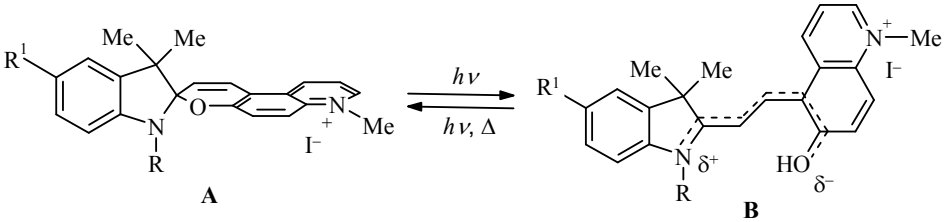
1, **7**, **12** R = Pr, R^1 = H; **2**, **8**, **13** R = All, R^1 = H; **3**, **9**, **14** R = Pr, R^1 = Br;
4, **10, 15** R = Me, R^1 = Me; **5, 11**, **16** R = Me, R^1 = OMe

The structures of compounds **7-16** were established by ^{1}H NMR spectroscopy and supported by elemental analysis.

The ^{1}H NMR spectra of spiropyrans **7-16** show two signals for magnetically non-equivalent geminal methyl groups, signals for the *N*-alkyl substituents (Me, Pr, All), upfield signals for the corresponding substituent indicator groups (Me, OMe), and several groups of interconnected downfield signals related to the indoline and pyranoquinoline fragments. The signals of the diastereotopic protons of the methylene group of *N*-allylspiropyrans **8** and **13** appear as two triplets of a doublet of doublets, while the signals for the terminal CH_2 group protons appear each as a quartet of doublets.

The ^{1}H NMR spectral data unequivocally support the structure of the spiropyran obtained. The lack of signals for protons of the indoline and pyranoquinoline fragments in the spectral regions characteristic for the open merocyanine form [23-25] indicates that these compounds exist in $CDCl_3$ solution in the spirocyclic form.

The electronic absorption spectra of solutions of spiro[indoline-2,3'-pyranoquinolines] **7-11** show long-wavelength bands with maxima at 347-349 nm and molar extinction coefficients 4280-4520 l/mol·cm (Table 1). In contrast, the absorption bands of cationic derivatives **12-16**, containing a quaternized pyridine fragment in a conjugated system, are shifted toward longer wavelengths and have lower intensity. The maxima of the long-wavelength absorption bands of the cationic spiropyrans are found at 398-401 nm, while the corresponding molar extinction coefficients are 3470-3710 l/mol·cm (Table 1). The spectral characteristics of the cationic and non-cationic spiropyrans hardly change upon variation of the substituents in the indoline fragment.



A **B**

The mechanism for the photochromic transformations of the spiropyrans involves the thermally and photochemically reversible heterolytic cleavage of the C_{spiro}–O bond in cyclic isomer **A** with subsequent *cis-trans* isomerization to metastable merocyanine form **B** [2-4].

Irradiation of solutions of both the cationic spiropyrans and their neutral analogs at the long-wavelength absorption bands leads to coloration related to the appearance of merocyanine isomers **B** [2-4].

Merocyanine isomers of spiro[indoline-2,3'-pyranoquinolines] **7-11** have a long-wavelength absorption band with maximum at 564-570 nm. The acyclic forms **B** of cationic derivatives **12-16** absorb at longer wavelengths and have long-wavelength band maxima at 570-579 nm. The introduction of the electron-donor methoxy group at C-5' position of the indoline fragment of analogs **11** and **16** has the greatest effect on the absorption spectra upon structural modification of the cationic and non-cationic compounds. This effect is expressed in the bathochromic shift of the long-wavelength absorption band maxima of colored forms **B** (Table 1) relative to the maxima of the derivatives having other substituents.

TABLE 1. Spectrokinetic Characteristics of Spiropyrans **7-11** and Their Cationic Derivatives **12-16** in Acetonitrile at 293 K

Compound	Form **A**		Form **B**	
	λ_{max}^{abs}, nm	$\varepsilon \cdot 10^3$, $l \cdot mol^{-1} \cdot cm^{-1}$	λ_{max}, nm	τ_{293}, s
7	291	10260	528, 564	0.2
	349	4460		
	364 (sh)	3710		
8	290	10330	526, 561	0.1
	349	4280		
	363 sh	3630		
9	289 sh	9940	530, 565	0.1
	301	9660		
	347	4430		
	362 sh	3670		
10	291	9970	529, 565	0.3
	298	9920		
	348	4510		
	363 sh	3840		
11	302	9010	535, 570	0.4
	348	4520		
12	271	46590	572	10.6
	320	4320		
	333	3750		
	401	3600		
13	269	45090	570	6.7
	320	4070		
	333	3720		
	399	3530		
14	269	51840	576	7.7
	333	3870		
	398	3470		
15	270	44550	573	15.4
	333	3980		
	400	3710		
16	242	29880	579	35.4
	270	45910		
	317 (sh)	8060		
	401	3670		

Thermal relaxation processes are observed in solutions of the cationic and non-cationic spiropyrans after the cessation of irradiation. These processes are related to recyclization of merocyanine isomers **B** to the starting spirocyclic forms **A** and, thus, indicate that all the spiropyrans obtained possess photochromic properties. Monoexponential kinetics is found for the relaxation processes, which may be correctly described with a lifetime constants for these species. For the acyclic spiro[indoline-2,3'-pyranoquinolines] **7-11**, the lifetime is 0.1-0.4 sec, reaching a maximum in case of the 5'-methoxyspiropyran **11**. The lifetime of the colored forms of cationic spiropyrans **12-16** is one or two orders of magnitude greater (6.7-35.4 sec, Table 1). Such a significant enhancement of the thermal stability for the acyclic isomers of the cationic spiropyrans may be attributed to the electron-withdrawing properties of the cationic fragment, which has a stabilizing effect on the dipolar merocyanine structure.

The cationic spiropyrans also show a reverse photoreaction, leading to efficient photodecoloration of the previously colored solutions of these compounds, in addition to the thermal recyclization reaction (Fig. 1).

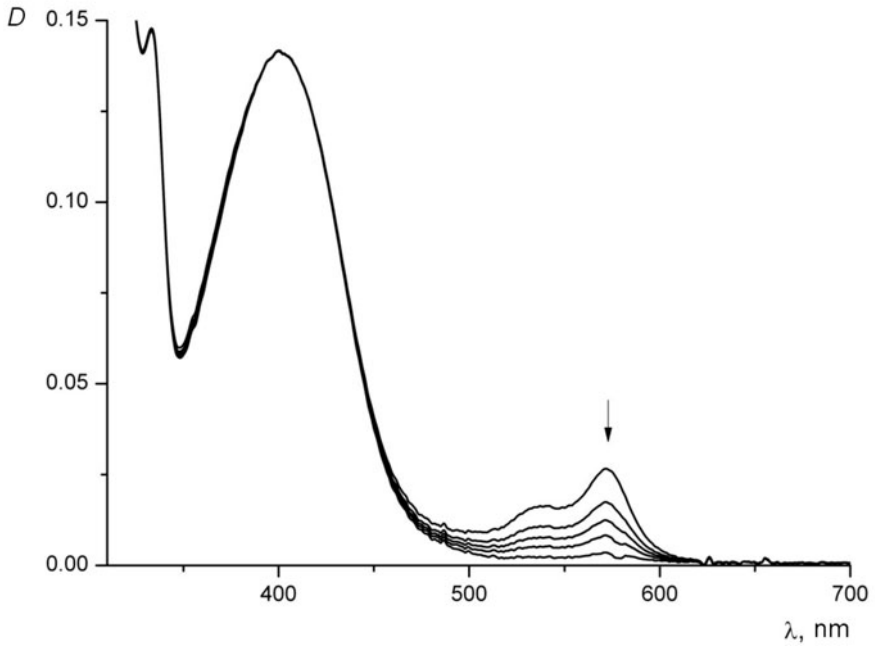


Fig. 1. Electronic absorption spectra of preliminary colored spiropyran **12** ($c = 3.93 \cdot 10^{-5}$ M, 293 K) during the photochemical decoloration reaction upon irradiation ($\lambda = 578$ nm, *dt* 0.6 s) in acetonitrile.

Thus, the new cationic spiropyrans obtained, containing a quaternized pyridine fragment in a conjugated ring system, demonstrate photochromic properties. The reverse channel of the photochromic cycle is encountered in both the ground and excited states.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Varian Unity-300 spectrometer at 300 MHz in $CDCl_3$ at 20°C. The signals were assigned relative to the signals of the residual solvent protons at 7.26 ppm. The electronic absorption spectra and kinetic curves of the thermal recyclization reactions were recorded on an Agilent 8453 spectrophotometer with a device for maintaining constant temperature. The photolysis of solutions with $c = 2 \cdot 10^{-5}$ M were recorded on a Newport system using a 200 W lamp and set of interference light filters. A sample of spectral-grade acetonitrile obtained from Aldrich was used for preparing the solutions.

3*H*-Indolium salts **1-5** and aldehyde **6** were obtained according to previously reported procedures [26-29].

3,3-Dimethylspiro[indoline-2,3'-pyrano[3,2-*f*]quinolines] 7-11 (General Method). A solution of triethylamine (0.28 ml, 2 mmol) in 2-propanol (2 ml) was added over 30 min to a mixture of 3*H*-indolium iodide **1-5** (2 mmol), formylquinolinol **6** (0.35 g, 2 mmol), and 2-propanol (28 ml) at reflux. The mixture was heated at reflux for 4 h and cooled. The solvent was evaporated in vacuum. The residue was subjected to chromatography on an alumina column using benzene as the eluent and the crude product was recrystallized.

3,3-Dimethyl-1-propylspiro[indoline-2,3'-pyrano[3,2-*f*]quinoline] (7). Yield 82%; mp 163-164°C (heptane). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.86 (3H, t, *J* = 7.4, $CH_2CH_2C\underline{H}_3$); 1.19 (3H, s, CH_3); 1.30 (3H, s, CH_3); 1.51-1.72 (2H, m, $CH_2C\underline{H}_2CH_3$); 3.04-3.23 (2H, m, $C\underline{H}_2CH_2CH_3$); 5.82 (1H, d, *J* = 10.5, H-2'); 6.54 (1H, d, *J* = 7.8, H-7); 6.83 (1H, dt, *J* = 7.4, *J* = 1.0, H-5); 7.08 (1H, dd, *J* = 7.3, *J* = 1.3, H-4); 7.15 (1H, d, *J* = 9.2, H-5'); 7.16 (1H, dt, *J* = 7.6, *J* = 1.4, H-6); 7.39 (1H, dd, *J* = 8.6, *J* = 4.2, H-9'); 7.46 (1H, d, *J* = 10.5, H-1'); 7.85 (1H, d, *J* = 9.2, H-6'); 8.32 (1H, d, *J* = 8.6, H-8'); 8.74 (1H, dd, *J* = 4.2, *J* = 1.6, H-10'). Found, %: C 81.01; H 6.68; N 7.93. $C_{24}H_{24}N_2O$. Calculated, %: C 80.87; H 6.79; N 7.86.

1-Allyl-3,3-dimethylspiro[indoline-2,3'-pyrano[3,2-*f*]quinoline] (8). Yield 68%; mp 156-157°C (heptane). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.22 (3H, s, CH_3); 1.32 (3H, s, CH_3); 3.67 (1H, tdd, *J* = 17.2, *J* = 5.2, *J* = 1.6) and 3.94 (1H, tdd, *J* = 17.2, *J* = 4.3, *J* = 2.1, $C\underline{H}_2CH{=}CH_2$); 5.05 (1H, qd, *J* = 10.2, *J* = 1.7) and 5.16 (1H, qd, *J* = 17.2, *J* = 1.8, $CH_2CH{=}C\underline{H}_2$); 5.83 (1H, d, *J* = 10.5, H-2'); 5.85 (1H, m, $CH_2C\underline{H}{=}CH_2$); 6.54 (1H, d, *J* = 7.7, H-7); 6.85 (1H, dt, *J* = 7.4, *J* = 1.0, H-5); 7.09 (1H, dd, *J* = 7.3, *J* = 1.3, H-4); 7.14 (1H, dt, *J* = 7.6, *J* = 1.3, H-6); 7.16 (1H, d, *J* = 9.2, H-5'); 7.38 (1H, dd, *J* = 8.6, *J* = 4.2, H-9'); 7.47 (1H, d, *J* = 10.5, H-1'); 7.85 (1H, d, *J* = 9.2, H-6'); 8.31 (1H, d, *J* = 8.6, H-8'); 8.74 (1H, dd, *J* = 4.2, *J* = 1.6, H-10'). Found, %: C 81.21; H 6.42; N 7.96. $C_{24}H_{22}N_2O$. Calculated, %: C 81.33; H 6.26; N 7.90.

5-Bromo-3,3-dimethyl-1-propylspiro[indoline-2,3'-pyrano[3,2-*f*]quinoline] (9). Yield 72%; mp 137-138°C (heptane). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.85 (3H, t, *J* = 7.3, $CH_2CH_2C\underline{H}_3$); 1.18 (3H, s, CH_3); 1.27 (3H, s, CH_3); 1.49-1.69 (2H, m, $CH_2C\underline{H}_2CH_3$); 2.99-3.21 (2H, m, $C\underline{H}_2CH_2CH_3$); 5.79 (1H, d, *J* = 10.5, H-2'); 6.40 (1H, *J* = 8.2, H-7); 7.14 (1H, d, *J* = 2.2, H-4); 7.15 (1H, d, *J* = 9.2, H-5'); 7.24 (1H, dd, *J* = 8.2, *J* = 2.2, H-6); 7.39 (1H, dd, *J* = 8.6, *J* = 4.2, H-9'); 7.47 (1H, d, *J* = 10.5, H-1'); 7.86 (1H, d, *J* = 9.2, H-6'); 8.32 (1H, d, *J* = 8.6, H-8'); 8.75 (1H, dd, *J* = 4.2, *J* = 1.6, H-10'). Found, %: C 66.36; H 5.25; N 6.37. $C_{24}H_{23}BrN_2O$. Calculated, %: C 66.21; H 5.32; N 6.43.

1,3,5,5-Tetramethylspiro[indoline-2,3'-pyrano[3,2-*f*]quinoline] (10). Yield 70%; mp 142-143°C (heptane). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.19 (3H, s, 3-CH_3); 1.32 (3H, s, 3-CH_3); 2.32 (3H, s, 5-CH_3); 2.69 (3H, s, 1-CH_3); 5.82 (1H, d, *J* = 10.5, H-2'); 6.44 (1H, d, *J* = 7.8, H-7); 6.91 (1H, m, H-4); 6.98 (1H, m, H-6); 7.19 (1H, d, *J* = 9.2, H-5'); 7.38 (1H, dd, *J* = 8.6, *J* = 4.2, H-9'); 7.49 (1H, d, *J* = 10.5, H-1'); 7.85 (1H, d, *J* = 9.2, H-6'); 8.33 (1H, d, *J* = 8.6, H-8'); 8.74 (1H, dd, *J* = 4.2, *J* = 1.6, H-10'). Found, %: C 80.47; H 6.61; N 8.25. $C_{23}H_{22}N_2O$. Calculated, %: C 80.67; H 6.48; N 8.18.

5-Methoxy-1,3,3-trimethylspiro[indoline-2,3'-pyrano[3,2-*f*]quinoline] (11). Yield 73%; mp 186-187°C (heptane–toluene, 2:1). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.22 (3H, s, 3-CH_3); 1.32 (3H, s, 3-CH_3); 2.70 (3H, s, 1-CH_3); 3.81 (3H, s, OCH_3); 5.84 (1H, d, *J* = 10.5, H-2'); 6.46 (1H, d, *J* = 7.8, H-7); 6.71-6.75 (2H, m, H-4,6); 7.21 (1H, d, *J* = 9.2, H-5'); 7.40 (1H, dd, *J* = 8.6, *J* = 4.3, H-9'); 7.51 (1H, d, *J* = 10.5, H-1'); 7.87 (1H, d, *J* = 9.2, H-6'); 8.35 (1H, d, *J* = 8.6, H-8'); 8.76 (1H, dd, *J* = 4.3, *J* = 1.5, H-10'). Found, %: C 77.16; H 6.08; N 7.67. $C_{23}H_{22}N_2O_2$. Calculated, %: C 77.07; H 6.19; N 7.82.

3,3,7'-Trimethylspiro[indoline-2,3'-pyrano[3,2-*f*]quinolinium] Iodides 12-16 (General Method). A mixture of spiropyran **7-11** (1 mmol), methyl iodide (0.426 g, ~0.2 ml, 3 mmol), and acetone (5.0 ml) was maintained for five days at room temperature. The precipitate formed was filtered off and washed with acetone.

3,3,7'-Trimethyl-1-propylspiro[indoline-2,3'-pyrano[3,2-*f*]quinolinium Iodide (12). Yield 85%; mp 224-226°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.87 (3H, t, *J* = 7.4, $CH_2CH_2C\underline{H}_3$); 1.21 (3H, s, 3-CH_3); 1.28 (3H, s, 3-CH_3); 1.51-1.72 (2H, m, $CH_2C\underline{H}_2CH_3$); 3.11 (2H, t, *J* = 7.4, $C\underline{H}_2CH_2CH_3$); 4.80 (3H, s, 7'-CH_3); 6.09 (1H, d, *J* = 10.6, H-2'); 6.57 (1H, d, *J* = 7.7, H-7); 6.87 (1H, dt, *J* = 7.4, *J* = 0.9, H-5); 7.08 (1H, dd, *J* = 7.3, *J* = 1.3, H-4);

7.18 (1H, dt, *J* = 7.6, *J* = 1.3, H-6); 7.57 (1H, d, *J* = 9.6, H-5'); 7.64 (1H, d, *J* = 10.6, H-1'); 8.05 (1H, d, *J* = 9.6, H-6'); 8.13 (1H, dd, *J* = 8.8, *J* = 5.7, H-9'); 9.29 (1H, d, *J* = 8.8, H-8'); 10.07 (1H, d, *J* = 5.7, H-10'). Found, %: C 60.36; H 5.27; N 5.56. $C_{25}H_{27}IN_2O$. Calculated, %: C 60.25; H 5.46; N 5.62.

1-Allyl-3,3,7'-trimethylspiro[indoline-2,3'-pyrano[3,2-*f*]quinolinium] Iodide (13). Yield 90%; mp 203-205°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.23 (3H, s, 3-CH_3); 1.30 (3H, s, 3-CH_3); 3.68 (1H, tdd, *J* = 17.2, *J* = 5.4, *J* = 1.5) and 3.90 (1H, tdd, *J* = 17.2, *J* = 4.2, *J* = 2.0, C$\underline{H}_2$CH=CH_2); 4.81 (3H, s, 7'-CH_3); 5.04 (1H, qd, *J* = 10.2, *J* = 1.7) and 5.11 (1H, qd, *J* = 17.2, *J* = 1.8, CH_2CH=C$\underline{H}_2$); 5.83 (1H, m, CH_2C$\underline{H}$=CH_2); 6.08 (1H, d, *J* = 10.6, H-2'); 6.57 (1H, d, *J* = 7.8, H-7); 6.89 (1H, dt, *J* = 7.5, *J* = 0.9, H-5); 7.09 (1H, dd, *J* = 7.3, *J* = 1.3, H-4); 7.16 (1H, dt, *J* = 7.6, *J* = 1.3, H-6); 7.56 (1H, d, *J* = 9.6, H-5'); 7.66 (1H, d, *J* = 10.6, H-1'); 8.07 (1H, d, *J* = 9.6, H-6'); 8.13 (1H, dd, *J* = 8.8, *J* = 5.7, H-9'); 9.30 (1H, d, *J* = 8.8, H-8'); 10.07 (1H, d, *J* = 5.7, H-10'). Found, %: C 60.37; H 4.98; N 5.58. $C_{25}H_{25}IN_2O$. Calculated, %: C 60.49; H 5.08; N 5.64.

5-Bromo-3,3,7'-trimethyl-1-propylspiro[indoline-2,3'-pyrano[3,2-*f*]quinolinium] Iodide (14). Yield 74%; mp 205-207°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.86 (3H, t, *J* = 7.4, $CH_2CH_2C\underline{H}_3$); 1.20 (3H, s, 3-CH_3); 1.25 (3H, s, 3-CH_3); 1.47-1.68 (2H, m, $CH_2C\underline{H}_2CH_3$); 3.05-3.11 (2H, m, C$\underline{H}_2CH_2CH_3$); 4.80 (3H, s, 7'-CH_3); 6.06 (1H, d, *J* = 10.6, H-2'); 6.43 (1H, d, *J* = 8.2, H-7); 7.15 (1H, d, *J* = 2.0, H-4); 7.26 (1H, dd, *J* = 8.2, *J* = 2.0, H-6); 7.56 (1H, d, *J* = 9.6, H-5'); 7.67 (1H, d, *J* = 10.6, H-1'); 8.07 (1H, d, *J* = 9.6, H-6'); 8.14 (1H, dd, *J* = 8.8, *J* = 5.7, H-9'); 9.31 (1H, d, *J* = 8.8, H-8'); 10.08 (1H, d, *J* = 5.7, H-10'). Found, %: C 51.83; H 4.70; N 4.94. $C_{25}H_{26}BrIN_2O$. Calculated, %: C 52.01; H 4.54; N 4.85.

1,3,3,5,7'-Pentamethylspiro[indoline-2,3'-pyrano[3,2-*f*]quinolinium] Iodide (15). Yield 77%; mp 230-232°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.21 (3H, s, 3-CH_3); 1.29 (3H, s, 3-CH_3); 2.32 (3H, s, 5-CH_3); 2.69 (3H, s, 1-CH_3); 4.80 (3H, s, 7'-CH_3); 6.08 (1H, d, *J* = 10.6, H-2'); 6.46 (1H, d, *J* = 7.8, H-7); 6.91 (1H, d, *J* = 1.7, H-4); 7.00 (1H, dd, *J* = 7.8, *J* = 1.7, H-6); 7.60 (1H, d, *J* = 9.6, H-5'); 7.66 (1H, d, *J* = 10.6, H-1'); 8.05 (1H, d, *J* = 9.6, H-6'); 8.12 (1H, dd, *J* = 8.8, *J* = 5.7, H-9'); 9.28 (1H, d, *J* = 8.8, H-8'); 10.12 (1H, d, *J* = 5.7, H-10'). Found, %: C 59.37; H 5.43; N 5.90. $C_{24}H_{25}IN_2O$. Calculated, %: C 59.51; H 5.20; N 5.78.

5-Methoxy-1,3,3,7'-tetramethylspiro[indoline-2,3'-pyrano[3,2-*f*]quinolinium] Iodide (16). Yield 89%; mp 219-221°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.24 (3H, s, 3-CH_3); 1.31 (3H, s, 3-CH_3); 2.69 (3H, s, 1-CH_3); 3.81 (3H, s, OCH_3); 4.83 (3H, s, 7'-CH_3); 6.11 (1H, d, *J* = 10.6, H-2'); 6.48 (1H, d, *J* = 9.1, H-7); 6.73-6.77 (2H, m, H-4,6); 7.63 (1H, d, *J* = 9.6, H-5'); 7.68 (1H, d, *J* = 10.6, H-1'); 8.09 (1H, d, *J* = 9.6, H-6'); 8.14 (1H, dd, *J* = 8.8, *J* = 5.7, H-9'); 9.29 (1H, d, *J* = 8.8, H-8'); 10.12 (1H, d, *J* = 5.7, H-10'). Found, %: C 57.78; H 5.10; N 5.75. $C_{24}H_{25}IN_2O_2$. Calculated, %: C 57.61; H 5.04; N 5.60.

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