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PHOTOCHROMIC AND THERMOCHROMIC SPIRANES. 34.* SYNTHESIS OF PHOTOCHROMIC 5-(4,5-DIPHENYL-1,3-OXAZOL-2-YL)-SUBSTITUTED SPIROBENZOCHROMENEINDOLINES

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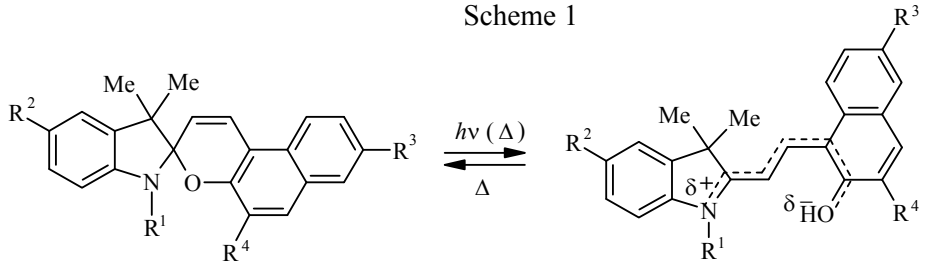
A simple and effective method is proposed for the synthesis of 4,5-diphenyloxazolyl-substituted o-hydroxynaphthaldehydes. Photochromic spirobenzochromeneindolines containing a 4,5-diphenyloxazole group at position 5 of the benzochromene fragment were obtained. In the cyclic form the new (diphenyloxazolyl)-substituted spiropyrans exhibit luminescent properties.

Keywords: merocyanines, spiropyrans, triaryloxazole, photochromism.

The synthesis and investigation of new effective photochromic systems with the aim of creating polyfunctional materials for molecular electronics and chemical sensors is a currently important problem [2, 3]. A special place among known types of photochromic compounds is occupied by relatively easily synthesized spiropyrans, the spectral and kinetic characteristics of which vary over a large range depending on the molecular structure [2–4].

Scheme 1



1A **1B**

*For Communication 33, see [1].

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The mechanism of the photochromic transformations of spiropyrans (Scheme 1) involves the thermally and photochemically reversible heterolytic cleavage of the C_{spiro}–O bond of the cyclic isomer **1A** followed by *cis–trans* isomerization to the metastable merocyanine form **1B** [2–4].

The insertion of various functional fragments into the spiropyran molecule opens up the possibility of obtaining a wide range of polyfunctional photochromic molecular systems exhibiting magnetic [5], fluorescent [6–9], and complexing [9–12] properties switched on by optical radiation. Earlier we reported on the synthesis of photochromic 5-(4,5-diphenyl-1,3-oxazol-2-yl)-substituted spiropyrans, the acyclic isomer of which forms reversibly complexes with the divalent cations of heavy metals [13]. The present work is a continuation of these investigations and is devoted to the synthesis of new spiropyrans containing a diphenyloxazolyl group at position 5 of the benzochromene fragment and their spectral and photochromic characteristics.

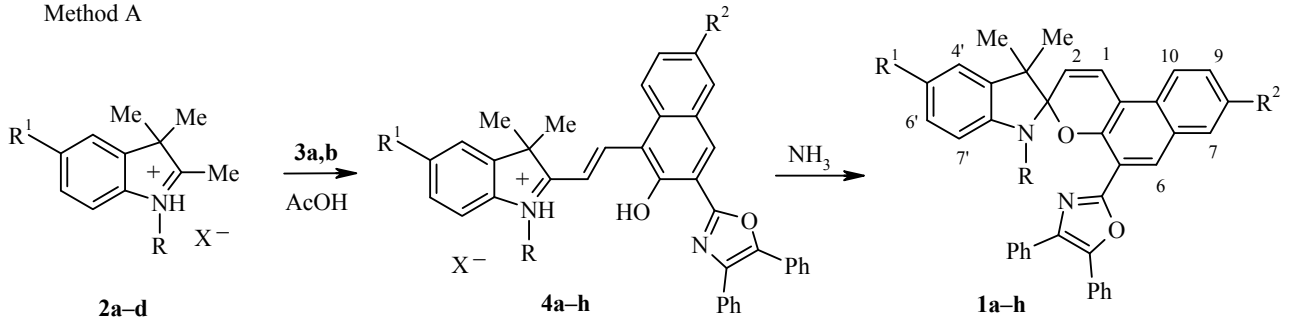
A convenient method for the production of 2,4,5-triaryl-1,3-oxazoles is cyclization of the esters of benzoin and aromatic acids (desyl esters), which can be obtained by the acylation of benzoin by Davidson's method [14]. In the case of 2-(hydroxyaryl)-4,5-diphenyl-1,3-oxazoles, the method of production of the desyl esters by the alkylation of the salts of the corresponding carboxylic acids with desyl chloride under the conditions of phase-transfer catalysis is preferred [15, 16].

5-(Diphenyloxazolyl)-substituted spiropyrans **1a**–**h** were obtained in two stages by the reaction of 3*H*-indolium salts **2a**–**d** with diphenyloxazolyl-substituted hydroxynaphthaldehydes **3a**,**b** in acetic acid with isolation of the obtained salts of *o*-hydroxynaphthylvinyl derivatives **4a**–**h** and treatment of the latter with ammonia (method A). The spiropyrans **1i**–**o** were obtained in one stage by the condensation of the 3*H*-indolium salts **2e**–**h** with hydroxynaphthaldehydes **3a**,**b** in the presence of triethylamine as base (method B) (Scheme 2). The diphenyloxazolyl-substituted aldehydes **3a**,**b** were obtained by the formylation of diphenyloxazolyl-substituted naphthols **5a**,**b**.

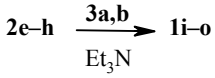
Instead of the previously described multistage method for the synthesis of the diphenyloxazolyl-substituted aldehyde **3a** [17] it is possible to propose a simple and effective method for the synthesis of the aldehydes **3a**,**b** (Scheme 3).

The starting compounds for the synthesis of the aldehydes **3a**,**b** were the sodium salts of the acids **6a**,**b**, the alkylation of which with 2-chloro-1,2-diphenylethanone (desyl chloride, **7**) under the conditions of phase-transfer catalysis in a solid phase–liquid system in the presence of 15-crown-5 gave the desyl esters **8a**,**b**. Reaction of the desyl esters **8a**,**b** with ammonium acetate in acetic acid by Davidson's method [14] gave the diphenyloxazolyl-substituted naphthols **5a**,**b**, the formylation of which by the Duff method in acetic acid gave the *o*-hydroxynaphthaldehydes **3a**,**b**.

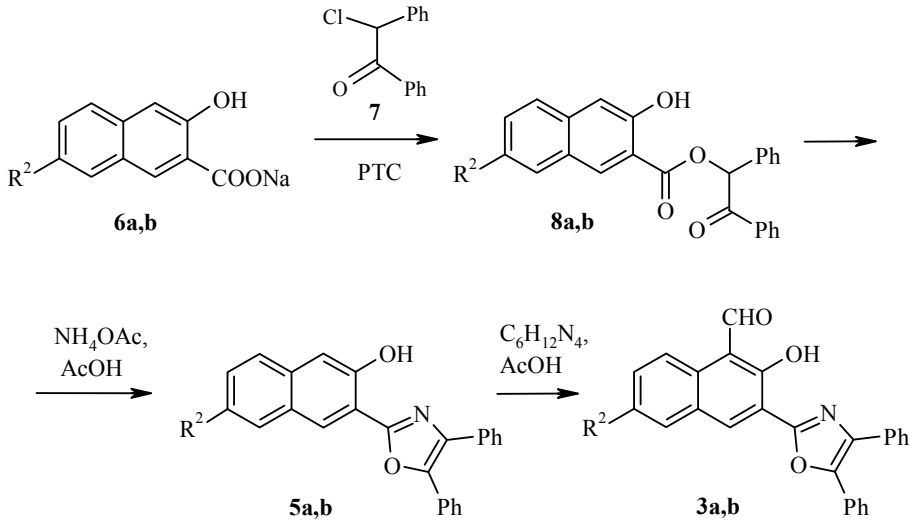
Scheme 2



Method B



Scheme 3



1a–c,e–g,k, 2a–c,e, 4a–c,e–g R = Me, **1d,h, 2d, 4d,h** R = Pr, **1i,j,m,n, 2f,g** R = All, **1l,o, 2h** R = *i*-Bu, **1a,e, 2a, 4a,e** R^1 = Cl, **1b,f, 2b, 4b,f** R^1 = Me, **1c,g,j,n, 2c,g, 4c,g** R^1 = OMe, **1d,h,i,l,m,o, 2d,f,h, 4d,h** R^1 = H, **1k, 2e** R^1 = OAll, **1a–d,i–l, 3a, 4a–d, 5,6,8a** R^2 = H, **1e–h,m–o, 3b, 4e–i, 5,6,8b** R^2 = OMe, **2a,g, 4a,e** X = ClO_4, **2b–f,h, 4b–d,f–i** X = I

The colorless or weakly colored 5-diphenyloxazolyl-substituted spiropyrans **1a**–**o** were purified by chromatography and recrystallized. The structure of compounds **1a**–**o**, **3a**,**b**, **5a**,**b**, and **8a**,**b** was established by ^{1}H NMR spectroscopy and confirmed by elemental analysis.

By ^{1}H NMR spectroscopy it is possible to establish quickly and accurately the structure of spiropyrans of the indoline series from the characteristic shifts, spin–spin coupling constants, and the number of different types of protons. The signals of such characteristic (indicator) groups as a *gem*-dimethyl group, an *N*-alkyl substituent, and the protons of the C(3)=C(4) double bond are usually determined easily and have different chemical shifts for the open and closed forms [18-20].

The upfield region of the spectrum of the spiropyrans contains two easily identified signals from the magnetically nonequivalent geminal methyl groups, the signal of the *N*-alkyl substituent (Me, Pr, All, *i*-Bu), and signals of the corresponding indicator groups of the substituents (Me, OMe, OAll) in the spiropyran.

The prochirality of the methylene group of the *N*-allyl substituent (spiropyrans **1i**,**j**,**m**,**n**) and the methyl groups and the protons of the methylene group of the *N*-isobutyl substituent (spiropyrans **1l**,**o**) leads to diastereotopic splitting of the signals of these groups, which appear in the form of two double doublets at 3.00 and 3.06 and two doublets at 0.87 and 0.91 ppm respectively.

The downfield part of the spectrum of the spiropyrans **1a**–**o** contains several groups of interdependent signals of protons belonging to the indoline and pyran fragments of the molecule and the signals of two groups, each of five interacting nuclei, belonging to the phenyls of the diphenyloxazolyl substituent. Unlike the diphenyloxazolyl-substituted naphthols **5** and aldehydes **3**, in the ^{1}H NMR spectra of which the signals of the ten protons of the phenyl groups appear in the form of four-proton and six-proton multiplets, in the ^{1}H NMR spectra of the spiropyrans **1** the signals of the protons of the two phenyl rings in the oxazole group form a complex picture of four multiplets with integral intensities of 2:3:3:2.

Thus, the structure of the obtained spiropyrans is confirmed unambiguously by the data from ^{1}H NMR spectroscopy (two signals from the magnetically nonequivalent geminal methyl groups, signals from the protons of the phenyl rings of the diphenyloxazolyl group, diastereotopic splitting of the signals for the protons of the *N*-alkyl substituent, the values of the chemical shifts of the protons and the spin–spin coupling constants of the diastereotopic protons of the *N*-isobutyl and *N*-allyl substituents, the protons of the double bond in the pyran

fragment, and the protons of the indoline and pyran fragments). The absence of signals for the *N*-methyl and *gem*-dimethyl groups, the *trans*-vinyl protons, and other protons of the indoline and benzochromene fragments in the regions of the spectra characteristic of the open merocyanine form indicates that the obtained compounds exist in solution in $CDCl_3$ mainly in the spirocyclic form.

The electronic absorption spectra of the cyclic forms **1A** of the spiropyrans **1a–o** in toluene solution are characterized by the presence of several bands. The less intense long-wave band with a molar extinction coefficient at the maximum of $4.5–9.0\times10^3$ $l\cdot mol^{-1}\cdot cm^{-1}$ is characterized by two poorly resolved maxima in the region of 380–395 nm (compounds **1a–c**,**i–l**, Table 1). The introduction of a methoxyl substituent at position 8 of the benzochromene fragment of the molecule gives rise to a bathochromic shift of this band by 10–15 nm (compounds **1e–h**,**m–o**). The position of the maxima of the more intense short-wave absorption bands of compounds **1a–o** (ε = $36.4–20.4\times10^3$ $l\cdot mol^{-1}\cdot cm^{-1}$) does not depend on the nature of the substituents and is located in the region of 310 and 340 nm.

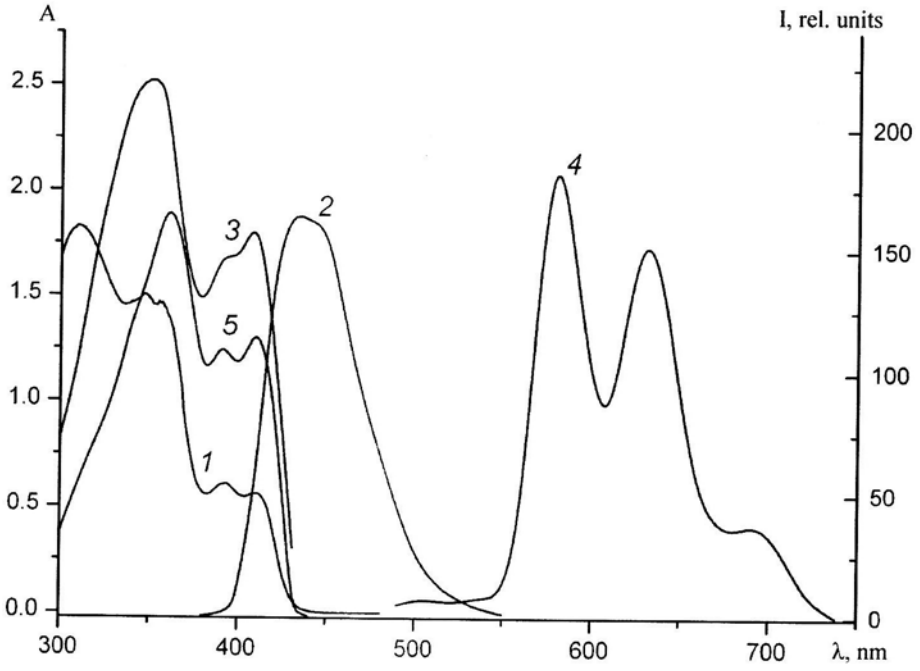


Fig. 1. The absorption spectrum (*1*) of a solution of **1g** and the fluorescence emission (*2*) and excitation (*3*) spectra of the spirocyclic isomer **1g**, T = 293 K, solvent toluene. The phosphorescence emission (*4*) and excitation (5) spectra of the spirocyclic isomer **1g**, T = 77 K, solvent toluene–ethanol–diethyl ether.

It was established that the cyclic forms of the spiropyrans at 293 K have fluorescent characteristics. The fluorescence emission spectrum represents a broad band in the region of 400–600 nm with poorly resolved maxima at 410 and 430 nm. As also in the case of the absorption spectra, the introduction of a methoxyl substituent at position 8 of the benzochromene part of the molecule leads to a bathochromic shift of the maxima of the fluorescence band by 20–25 nm.

At 77 K the solutions of compounds **1a–o** exhibit strong fluorescence, which is characterized by a structured band with maxima at 580, 630, and 690 nm; their position hardly depends at all on the nature of the substituents in the indoline and pyran parts of the molecule. The fluorescence and phosphorescence excitation spectra practically coincide with each other, and the position of their maxima correlates well with the position of the maxima in the absorption spectrum, providing grounds for attributing the observed luminescent characteristics to the spirocyclic isomers of the synthesized compounds (Fig. 1, Table 1).

TABLE 1. The Spectral Characteristics of Compounds **1a–o***

Compound	Isomer	λ_{abs}, nm ($\varepsilon \cdot 10^3$ l·mol^{-1}·cm^{-1})	$\lambda_{ex\,flu}$, nm	λ_{flu}, nm	$\lambda_{ex\,ph}$, nm	λ_{ph}, nm	λ_{abs}, nm ($\varepsilon \cdot 10^3$, l·mol^{-1}·cm^{-1})
		Toluene					Acetone
1a	**A**	306 (30.67), 342 (23.13), 380 (8.63), 396 (6.12)	385, 393	410, 430, 450	385, 393	580, 630, 685	380 (6.38), 400 (3.84)
	B	—	—	—	—	—	595
1b	**A**	309 (28.40), 342 (20.90), 380 (8.40), 396 (5.80)	385, 400	420, 433, 455	380, 395	575, 625, 680	380 (6.70), 395 (4.78)
	B	—	—	—	—	—	597
1c	**A**	309 (33.70), 341 (24.40), 380 (9.40), 397 (6.50)	385, 400	410, 430, 465	370, 400	577, 630, 690	378 (6.77), 394 (5.02)
	B	—	—	—	—	—	602
1d	**A**	377 (7.78), 395 (5.30)	357, 396	410, 430	393, 360	580, 630, 690	380 (6.72), 400 (4.20)
	B	594	—	—	—	—	593
1e	**A**	305 (36.14), 343 (30.83), 391 (9.91), 409 (9.09)	360, 393, 418	427, 447	360, 390, 410	578, 628, 690	390 (6.00), 410 (5.44)
	B	—	—	—	—	—	612
1f	**A**	305 (30.61), 343 (27.00), 392 (8.23), 410 (7.49)	393, 410	430, 448	396, 410	570, 625, 680	392 (4.65), 410 (4.20)
	B	—	—	—	—	—	613
1g	**A**	310 (32.10), 343 (27.12), 392 (8.38), 411 (7.75)	410, 395, 355	430, 455	412, 393, 362	580, 631, 690	393 (6.45), 409 (5.96)
	B	—	—	—	—	—	612
1h	**A**	301 (21.20), 343 (18.83), 393 (7.16), 410 (6.54)	394, 410	430, 450	410	580, 630, 690	394 (6.05), 410 (5.50)
	B	—	—	—	—	—	614

TABLE 1 (continued)

1	2	3	4	5	6	7	8
1h	**A**	301 (21.20), 343 (18.83), 393 (7.16), 410 (6.54)	394, 410	430, 450	410	580, 630, 690	394 (6.05), 410 (5.50)
	B	—	—	—	—	—	614
1i	A	304 (20.39), 342 (16.55), 380 (6.31), 395 (4.40)	360, 393	410, 432	360, 395	582, 630, 690	379 (6.77), 400 (4.03)
	B	588	—	—	—	—	587
1j	A	310 (36.44), 343 (26.10), 382 (9.58), 396 (7.00)	370, 390	410, 430, 465	363, 397	577, 630, 690	380 (4.62), 399 (4.01)
	B	601	—	—	—	—	600
1k	A	309 (33.40), 342 (24.17), 380 (9.16), 396 (6.53)	396	430, 450	398	578, 630, 690	380 (6.78), 395 (4.69)
	B	—	—	—	—	—	602
1l	A	382 (7.85), 396 (6.00)	395, 380	414, 430	395, 375	578, 628, 690	380 (6.78), 394 (4.95)
	B	595	—	—	—	—	591
1m	A	341 (17.23), 361 (10.73), 392 (5.17), 409 (4.55)	370, 412	438, 452	378, 410	580, 630	391 (5.69), 409 (4.94)
	B	—	—	—	—	—	613
1n	A	310 (20.74), 343 (16.90), 393 (6.78), 410 (6.18)	390, 410	428, 447	390, 408	578, 630, 685	392 (4.33), 408 (3.96)
	B	—	—	—	—	—	620
1o	A	393 (6.02), 410 (5.40)	390, 410	440	390, 410	580, 630, 690	393 (4.82), 410 (4.51)
	B		—	—	—	—	616

* λ_{max}^{abs}, $\lambda_{max}^{ex\_flu}$, λ_{max}^{flu}, $\lambda_{max}^{ex\_ph}$, λ_{max}^{ph} – The wave lengths of the maxima in the absorption, fluorescence excitation, fluorescence, phosphorescence excitation, and phosphorescence spectra respectively.

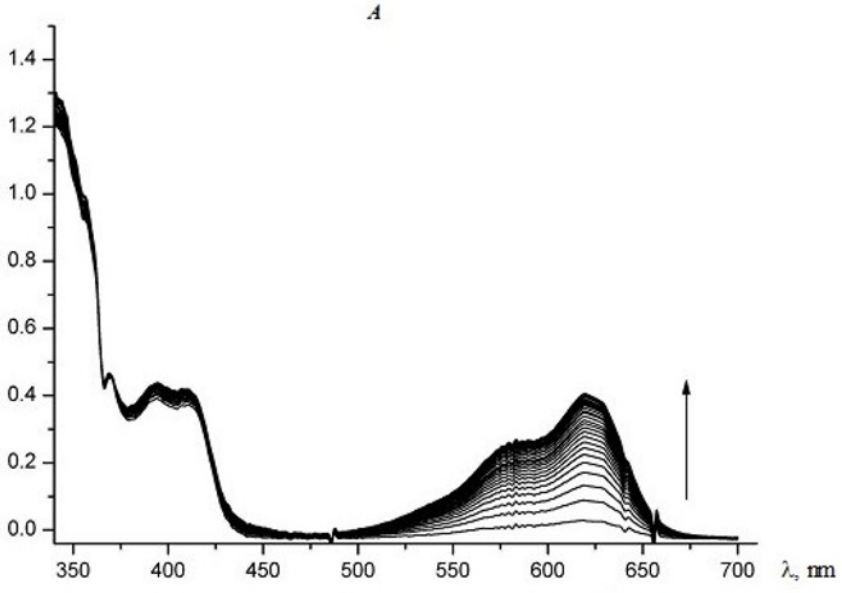


Fig. 2. The variation of the absorption spectra of a solution of **1g** in acetone during irradiation with light at 365 nm, spectrum recording interval 5 s.

Compounds **1a**–**o** exhibit photochromic properties at temperatures below room temperature, and this is due to the high rates of thermal recyclization of the isomers **1B** → **1A**. Thus, during the exposure of acetone solutions of these compounds in the region of the long-wave absorption of the cyclic forms **1A** at *T* 270 K their colorization, accompanied by the appearance of bands in the electronic absorption spectra in the region of 550-650 nm (Fig. 2, Table 1) characteristic of the noncyclic merocyanine isomers **1B** of the spiropyrans, is observed [2–4]. The position of the maximum of the long-wave absorption band of the merocyanine form **1B** in the series of compounds **1a**–**o** is shifted appreciably into the long-wave region of the spectrum with the introduction of the methoxy group at position 5 of the benzochromene part of the spiropyran.

Thus, the obtained new 5-(4,5-diphenyl-1,3-oxazol-2-yl)-substituted spirobenzochromeneindolines possess fluorescence and phosphorescence in the spirocyclic form and exhibit photochromic properties in solutions.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Varian Unity-300 spectrometer (300 MHz) in $CDCl_3$. The signals were assigned with reference to the signal of the residual protons of the deuterated solvent (δ 7.26 ppm). The electronic absorption spectra of the investigated compounds were recorded on an Agilent 8453 spectrophotometer with a thermostat attachment for the samples, and the electronic spectra were recorded on a Varian Cary Eclipse spectrofluorimeter. Photolysis of the solutions was realized with a DRSh-250 lamp with a set of interference filters. Toluene and acetone (Aldrich) of spectral purity were used to prepare the solutions. The sodium salt of the acid **6b** was the commercial product (Fluka).

The sodium salt of the acid **6a**, the desyl chloride **7**, and the 3*H*-indolium salts **2a-d** were obtained by the previously described methods [16, 21–25].

2-Oxo-1,2-diphenyl Esters of 3-Hydroxy-2-naphthoic Acids 8a,b (General Method). A mixture of the sodium salt **6a**,**b** (33 mol), 15-crown-5 (1 ml, 5 mmol), and acetonitrile (90 ml) was stirred at 70°C for 30 min, and desyl chloride **7** (6.93 g, 30 mmol) was added. The mixture was stirred with boiling for 7 h and poured into 200 ml of water and ice. The precipitate was filtered off, washed with water, and dried. The obtained esters were recrystallized from a mixture of 2-propanol and toluene.

2-Oxo-1,2-diphenylethyl Ester of 3-Hydroxy-2-naphthoic Acid (8a). Yield 81%; mp 152–153.5°C (2-propanol–toluene, 3:1). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.17 (1H, s, 2-COOCH); 7.29–7.34 (2H, m, H-4,7); 7.42–7.64 (9H, m, H-6, H Ph); 7.68 (1H, d, *J* = 8.4, H-5); 7.82 (1H, d, *J* = 8.4, H-8); 8.00–8.03 (2H, m, H Ph); 8.64 (1H, s, H-1); 10.10 (1H, s, 3-OH). Found, %: C 78.67; H 4.65. $C_{25}H_{18}O_4$. Calculated, %: C 78.52; H 4.74.

2-Oxo-1,2-diphenylethyl Ester of 3-Hydroxy-7-methoxy-2-naphthoic Acid (8b). Yield 74%; mp 159–160°C (2-propanol–toluene, 2:1). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.86 (3H, s, 7-OCH_3); 7.08 (1H, d, *J* = 2.4, H-8); 7.15 (1H, s, 2-COOCH); 7.17 (1H, dd, *J* = 8.8, *J* = 2.4, H-6); 7.25 (1H, s, H-4); 7.40–7.46 (5H, m, H Ph); 7.52–7.62 (4H, m, H-5, H Ph); 7.98–8.01 (2H, m, H Ph); 8.51 (1H, s, H-1); 9.92 (1H, s, 3-OH). Found, %: C 75.90; H 4.95. $C_{26}H_{20}O_5$. Calculated, %: C 75.72; H 4.89.

3-(4,5-Diphenyl-1,3-oxazol-2-yl)-2-naphthols 5a,b (General Method). A mixture of desyl naphthoate **8a**,**b** (20 mmol), ammonium acetate (9.24 g, 120 mmol), and acetic acid (40 ml) was boiled for 4 h. The mixture was poured into ice (500 g), and the precipitate was filtered off, washed with water, and dried. The obtained naphthols **5a**,**b** were recrystallized from a mixture of 2-propanol and toluene.

3-(4,5-Diphenyl-1,3-oxazol-2-yl)-2-naphthol (5a). Yield 69%; mp 154–155°C (2-propanol–toluene, 3:1). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.35 (1H, m, H-7); 7.40–7.45 (7H, m, H-1, H Ph); 7.48 (1H, m, H-6); 7.71–7.77 (5H, m, H-5, H Ph); 7.86 (1H, d, *J* = 8.4, H-8); 8.49 (1H, s, H-4); 11.15 (1H, s, 2-OH). Found, %: C 82.75; H 4.79; N 3.76. $C_{25}H_{17}NO_2$. Calculated, %: C 82.63; H 4.71; N 3.85.

3-(4,5-Diphenyl-1,3-oxazol-2-yl)-6-methoxy-2-naphthol (5b). Yield 72%; mp 194–195.5°C (2-propanol–toluene, 1:1). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.90 (3H, s, 6-OCH_3); 7.14 (1H, d, *J* = 2.6, H-5); 7.15 (1H, dd, *J* = 9.7, *J* = 2.6, H-7); 7.37 (1H, s, H-1); 7.39–7.46 (6H, m, H Ph); 7.61 (1H, d, *J* = 9.7, H-8); 7.70-7.75 (4H, m, H Ph); 8.37 (1H, s, H-4); 10.95 (1H, s, 2-OH). Found, %: C 79.48; H 4.95; N 3.50. $C_{26}H_{19}NO_3$. Calculated, %: C 79.37; H 4.87; N 3.56.

2-Hydroxy-3-(4,5-diphenyl-1,3-oxazol-2-yl)-1-naphthaldehydes 3a,b (General Method). A mixture of the naphthol **5a**,**b** (10 mmol), hexamethylenetetramine (2.80 g, 20 mmol), and acetic acid (40 ml) was stirred at 95–100°C for 5 h 30 min. A mixture of conc. HCl (15 ml) and water (18 ml) was added, and the reaction mixture was stirred at 95–100°C for 1 h. It was poured into 175 ml of water, and the precipitate was filtered off, washed with water, and dried. The obtained aldehydes **3a**,**b** were purified by column chromatography on Al_2O_3 (eluent chloroform) and recrystallized from a 2:1 mixture of benzene and acetonitrile.

2-Hydroxy-3-(4,5-diphenyl-1,3-oxazol-2-yl)-1-naphthaldehyde (3a). Yield 51%; mp 213–214°C. The ^{1}H NMR spectrum of the obtained compound was identical with the spectrum of the compound obtained earlier [17]. Found, %: C 79.67; H 4.24; N 3.70. $C_{26}H_{17}NO_3$. Calculated, %: C 79.78; H 4.38; N 3.58.

2-Hydroxy-3-(4,5-diphenyl-1,3-oxazol-2-yl)-6-methoxy-1-naphthaldehyde (3b). Yield 38%; mp 217.5–218.5°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.93 (3H, s, 6-OCH_3); 7.18 (1H, d, *J* = 2.7, H-5); 7.34 (1H, dd, *J* = 9.5, *J* = 2.7, H-7); 7.41–7.49 (6H, m, H Ph); 7.71–7.76 (4H, m, H Ph); 8.58 (1H, s, H-4); 9.19 (1H, d, *J* = 9.5, H-8); 11.02 (1H, s, 1-CHO); 12.22 (1H, s, 2-OH). Found, %: C 76.80; H 4.65; N 3.43. $C_{27}H_{19}NO_4$. Calculated, %: C 76.95; H 4.54; N 3.32.

3',3'-Dimethyl-5-(4,5-diphenyl-1,3-oxazol-2-yl)spiro[benzo[*f*]chromene-3,2'-indolines] 1a–h (General Method). A. A mixture of the 3H-indolium salt **2a**–**d** (1 mmol), aldehyde **3a**,**b** (1 mmol), and glacial acetic acid (8 ml) was boiled for 5 h 30 min and kept at ~20°C for 12 h. The precipitate was filtered off, washed with ether, dried, and then used without further purification. Dry ammonia was passed into a suspension of the obtained salt **4a**–**h** in benzene (20 ml), the solvent was evaporated, and the residue was purified by column chromatography on Al_2O_3 (eluent benzene). The spiropyrans **1a**–**h** were recrystallized from a 2:1 mixture of heptane and toluene.

3',3'-Dimethyl-5-(4,5-diphenyl-1,3-oxazol-2-yl)spiro[benzo[*f*]chromene-3,2'-indolines] 1i–o (General Method). B. A mixture of the 3*H*-indolium salt **2e**–**h** (1 mmol), triethylamine (0.14 ml, 1 mmol), and aldehyde **3a**,**b** (1 mmol) in benzene (8 ml) and 2-propanol (2 ml) was boiled for 10 h and evaporated. The residue was purified by column chromatography on Al_2O_3 (eluent benzene). The spiropyrans **1i**–**o** were recrystallized from a 3:1 mixture of isooctane and toluene.

5'-Chloro-5-(4,5-diphenyl-1,3-oxazol-2-yl)-1',3',3'-trimethylspiro[benzo[*f*]chromene-3,2'-indoline] (1a). Yield 50%; mp 219.5–221°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.26 (3H, s, 3'-CH_3); 1.43 (3H, s, 3'-CH_3); 2.77 (3H, s, 1'-CH_3); 5.88 (1H, d, *J* = 10.5, H-2); 6.45 (1H, d, *J* = 8.2, H-7'); 7.08 (1H, d, *J* = 2.1, H-4'); 7.13 (1H, dd, *J* = 8.2, *J* = 2.1, H-6'); 7.15–7.19 (2H, m, H Ph); 7.30–7.42 (7H, m, H-8, H Ph); 7.57 (1H, dd, *J* = 8.5, *J* = 6.9, *J* = 1.4, H-9); 7.59–7.62 (2H, m, H Ph); 7.68 (1H, d, *J* = 10.5, H-1); 7.87 (1H, d, *J* = 8.1, H-7); 8.05 (1H, d, *J* = 8.5, H-10); 8.62 (1H, s, H-6). Found, %: C 78.68; H 5.15; N 4.89. $C_{38}H_{29}ClN_2O_2$. Calculated, %: C 78.54; H 5.03; N 4.82.

5-(4,5-Diphenyl-1,3-oxazol-2-yl)-1',3',3',5'-tetramethylspiro[benzo[*f*]chromene-3,2'-indoline] (1b). Yield 43%; mp 222–223°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.25 (3H, s, 3'-CH_3); 1.41 (3H, s, 3'-CH_3); 2.34 (3H, s, 5'-CH_3); 2.78 (3H, s, 1'-CH_3); 5.90 (1H, d, *J* = 10.5, H-2); 6.47 (1H, d, *J* = 7.8, H-7'); 6.93 (1H, m, H-4'); 7.00 (1H, m, H-6'); 7.13–7.16 (2H, m, H Ph); 7.21–7.29 (3H, m, H Ph); 7.31–7.41 (4H, m, H-8, H Ph); 7.56 (1H, ddd, *J* = 8.4, *J* = 6.9, *J* = 1.4, H-9); 7.61–7.64 (2H, m, H Ph); 7.65 (1H, d, *J* = 10.5, H-1); 7.86 (1H, d, *J* = 8.1, H-7); 8.05 (1H, d, *J* = 8.5, H-10); 8.60 (1H, s, H-6). Found, %: C 83.37; H 5.83; N 5.09. $C_{39}H_{32}N_2O_2$. Calculated, %: C 83.54; H 5.75; N 5.00.

5-(4,5-Diphenyl-1,3-oxazol-2-yl)-1',3',3'-trimethyl-5'-methoxyspiro[benzo[*f*]chromene-3,2'-indoline] (1c). Yield 42%; mp 192.5–193.5°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.27 (3H, s, 3'-CH_3); 1.43 (3H, s, 3'-CH_3); 2.74 (3H, s, 1'-CH_3); 3.79 (3H, s, 5-OCH_3); 5.90 (1H, d, *J* = 10.5, H-2); 6.46 (1H, d, *J* = 8.3, H-7'); 6.71 (1H, dd, *J* = 8.3, *J* = 2.4, H-6'); 6.78 (1H, d, *J* = 2.4, H-4'); 7.12–7.16 (2H, m, H Ph); 7.26–7.40 (7H, m, H-8, H Ph); 7.56 (1H, ddd, *J* = 8.4, *J* = 6.9, *J* = 1.4, H-9); 7.61–7.64 (2H, m, H Ph); 7.66 (1H, d, *J* = 10.5, H-1); 7.86 (1H, d, *J* = 8.1, H-7); 8.05 (1H, d, *J* = 8.5, H-10); 8.62 (1H, s, H-6). Found, %: C 81.37; H 5.46; N 4.75. $C_{39}H_{32}N_2O_3$. Calculated, %: C 81.23; H 5.59; N 4.86.

5-(4,5-Diphenyl-1,3-oxazol-2-yl)-3',3'-dimethyl-1'-propylspiro[benzo[*f*]chromene-3,2'-indoline] (1d). Yield 45%; mp 159.5–161°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.84 (3H, t, *J* = 7.4, 1'-$CH_2CH_2C\underline{H}_3$); 1.25 (3H, s, 3'-CH_3); 1.42 (3H, s, 3'-CH_3); 1.65 (2H, m, 1'-$CH_2C\underline{H}_2CH_3$); 3.25 (2H, m, 1-$C\underline{H}_2CH_2CH_3$); 5.89 (1H, d, *J* = 10.5, H-2); 6.58 (1H, d, *J* = 7.7, H-7'); 6.87 (1H, td, *J* = 7.4, *J* = 0.9, H-5'); 7.09–7.13 (3H, m, H-4', H Ph); 7.21 (1H, dt, *J* = 7.6, *J* = 1.2, H-6'); 7.23–7.27 (3H, m, H Ph); 7.31–7.40 (4H, m, H-8, H Ph); 7.56 (1H, ddd, *J* = 8.3, *J* = 6.9, *J* = 1.3, H-9); 7.60–7.64 (3H, m, H-1, H Ph); 7.86 (1H, d, *J* = 8.1, H-7); 8.05 (1H, d, *J* = 8.5, H-10); 8.61 (1H, s, H-6). Found, %: C 83.45; H 6.03; N 4.98. $C_{40}H_{34}N_2O_2$. Calculated, %: C 83.60; H 5.96; N 4.87.

5'-Chloro-5-(4,5-diphenyl-1,3-oxazol-2-yl)-8-methoxy-1',3',3'-trimethylspiro[benzo[*f*]chromene-3,2'-indoline] (1e). Yield 46%; mp 225–226°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.26 (3H, s, 3'-CH_3); 1.42 (3H, s, 3'-CH_3); 2.76 (3H, s, 1'-CH_3); 3.92 (3H, s, 8-OCH_3); 5.87 (1H, d, *J* = 10.5, H-2); 6.44 (1H, d, *J* = 8.2, H-7'); 7.08 (1H, d, *J* = 2.1, H-4'); 7.14 (1H, dd, *J* = 8.1, *J* = 2.1, H-6'); 7.15–7.18 (3H, m, H-7, H Ph); 7.25 (1H, dd, *J* = 9.2, *J* = 2.7, H-9); 7.29–7.41 (6H, m, H Ph); 7.60–7.64 (3H, m, H-1, H Ph); 7.96 (1H, d, *J* = 9.2, H-10); 8.53 (1H, s, H-6). Found, %: C 76.54; H 5.25; N 4.66. $C_{39}H_{31}ClN_2O_3$. Calculated, %: C 76.65; H 5.11; N 4.58.

5-(4,5-Diphenyl-1,3-oxazol-2-yl)-8-methoxy-1',3',3',5'-tetramethylspiro[benzo[*f*]chromene-3,2'-indoline] (1f). Yield 46%; mp 241–242°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.25 (3H, s, 3'-CH_3); 1.41 (3H, s, 3'-CH_3); 2.34 (3H, s, 5'-CH_3); 2.77 (3H, s, 1'-CH_3); 3.91 (3H, s, 8-OCH_3); 5.89 (1H, d, *J* = 10.5, H-2); 6.46 (1H, d, *J* = 7.8, H-7'); 6.93 (1H, d, *J* = 1.6, H-4'); 7.00 (1H, m, H-6'); 7.13–7.18 (3H, m, H-7, H Ph); 7.20–7.28 (4H, m, H-9, H Ph); 7.30–7.39 (3H, m, H Ph); 7.61–7.64 (2H, m, H Ph); 7.59 (1H, d, *J* = 10.5, H-1); 7.96 (1H, d, *J* = 9.3, H-10); 8.51 (1H, s, H-6). Found, %: C 81.17; H 5.69; N 4.85. $C_{40}H_{34}N_2O_3$. Calculated, %: C 81.33; H 5.80; N 4.74.

5-(4,5-Diphenyl-1,3-oxazol-2-yl)-5',8-dimethoxy-1',3',3'-trimethylspiro[benzo[*f*]chromene-3,2'-indoline] (1g). Yield 47%; mp 231–232°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.27 (3H, s, 3'-CH_3); 1.42 (3H, s, 3'-CH_3); 2.73 (3H, s, 1'-CH_3); 3.79 (3H, s, 5'-OCH_3); 3.92 (3H, s, 8-OCH_3); 5.90 (1H, d, *J* = 10.5, H-2); 6.45 (1H, d, *J* = 8.3, H-7'); 6.72 (1H, dd, *J* = 8.3, *J* = 2.6, H-6'); 6.78 (1H, d, *J* = 2.6, H-4'); 7.12–7.18 (3H, m, H-7,

H Ph); 7.21–7.29 (4H, m, H-9, H Ph); 7.31–7.40 (3H, m, H Ph); 7.58–7.65 (3H, m, H-1, H Ph); 7.96 (1H, d, *J* = 9.2, H-10); 8.53 (1H, s, H-6). Found, %: C 79.05; H 5.74; N 4.73. $C_{40}H_{34}N_2O_4$. Calculated, %: C 79.19; H 5.65; N 4.62.

5-(4,5-Diphenyl-1,3-oxazol-2-yl)-8-methoxy-3',3'-dimethyl-1'-propylspiro[benzo[f]chromene-3,2'-indoline] (1h). Yield 45%; mp 229–230°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.84 (3H, t, *J* = 7.4, 1'-$CH_2CH_2C\underline{H}_3$); 1.24 (3H, s, 3'-CH_3); 1.41 (3H, s, 3'-CH_3); 1.64 (2H, m, 1'-$CH_2C\underline{H}_2CH_3$); 3.24 (2H, m, 1'-$C\underline{H}_2CH_2CH_3$); 3.92 (3H, s, 8-OCH_3); 5.89 (1H, d, *J* = 10.5, H-2); 6.58 (1H, d, *J* = 7.7, H-7'); 6.87 (1H, td, *J* = 7.4, *J* = 0.9, H-5'); 7.08–7.18 (4H, m, H-4',7, H Ph); 7.19–7.27 (5H, m, H-6',9, H Ph); 7.31–7.39 (3H, m, H Ph); 7.57 (1H, d, *J* = 10.5, H-1); 7.61–7.64 (2H, m, H Ph); 7.95 (1H, d, *J* = 9.2, H-10); 8.52 (1H, s, H-6). Found, %: C 81.25; H 5.91; N 4.76. $C_{41}H_{36}N_2O_3$. Calculated, %: C 81.43; H 6.00; N 4.63.

1'-Allyl-5-(4,5-diphenyl-1,3-oxazol-2-yl)-3',3'-dimethylspiro[benzo[*f*]chromene-3,2'-indoline] (1i). Yield 44%; mp 179–180°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.29 (3H, s, 3'-CH_3); 1.44 (3H, s, 3'-CH_3); 3.80 (1H, ddt, *J* = 17.3, *J* = 5.3, *J* = 1.6, 1'-$C\underline{H}_2CH=CH_2$); 4.04 (1H, ddt, *J* = 17.3, *J* = 4.2, *J* = 2.0, 1'-$C\underline{H}_2CH=CH_2$); 4.99 (1H, dq, *J* = 10.3, *J* = 1.7, 1'-$CH_2CH=C\underline{H}_2$); 5.15 (1H, dq, *J* = 17.2, *J* = 1.8, 1'-$CH_2CH=C\underline{H}_2$); 5.89 (1H, m, 1'-$CH_2C\underline{H}=CH_2$); 5.90 (1H, d, *J* = 10.5, H-2); 6.59 (1H, d, *J* = 7.7, H-7'); 6.90 (1H, td, *J* = 7.4, *J* = 0.9, H-5'); 7.09–7.16 (3H, m, H-4', H Ph); 7.20 (1H, td, *J* = 7.6, *J* = 1.3, H-6'); 7.23–7.26 (3H, m, H Ph); 7.31–7.41 (4H, m, H-8, H Ph); 7.56 (1H, ddd, *J* = 8.4, *J* = 6.9, *J* = 1.4, H-9); 7.60–7.65 (3H, m, H-1, H Ph); 7.86 (1H, d, *J* = 8.1, H-7); 8.04 (1H, d, *J* = 8.5, H-10); 8.61 (1H, s, H-6). Found, %: C 83.74; H 5.69; N 4.80. $C_{40}H_{32}N_2O_2$. Calculated, %: C 83.89; H 5.63; N 4.89.

1'-Allyl-5-(4,5-diphenyl-1,3-oxazol-2-yl)-5'-methoxy-3',3'-dimethylspiro[benzo[*f*]chromene-3,2'-indoline] (1j). Yield 60%; mp 195–196.5°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.29 (3H, s, 3'-CH_3); 1.43 (3H, s, 3'-CH_3); 3.72 (1H, ddt, *J* = 17.2, *J* = 5.4, *J* = 1.6, 1'-$C\underline{H}_2CH=CH_2$); 3.78 (3H, s, 5'-OCH_3); 3.98 (1H, ddt, *J* = 17.2, *J* = 4.5, *J* = 2.1, 1'-$C\underline{H}_2CH=CH_2$); 4.98 (1H, dq, *J* = 10.3, *J* = 1.7, 1'-$CH_2CH=C\underline{H}_2$); 5.14 (1H, dq, *J* = 17.2, *J* = 1.7, 1'-$CH_2CH=C\underline{H}_2$); 5.88 (1H, m, 1'-$CH_2C\underline{H}=CH_2$); 5.90 (1H, d, *J* = 10.5, H-2); 6.48 (1H, d, *J* = 8.4, H-7'); 6.69 (1H, dd, *J* = 8.4, *J* = 2.6, H-6'); 6.78 (1H, d, *J* = 2.6, H-4'); 7.12–7.17 (2H, m, H Ph); 7.24-7.28 (3H, m, H Ph); 7.32–7.40 (4H, m, H-8, H Ph); 7.55 (1H, ddd, *J* = 8.4, *J* = 6.9, *J* = 1.4, H-9); 7.60–7.65 (3H, m, H-1, H Ph); 7.86 (1H, dd, *J* = 8.2, *J* = 1.3, H-7); 8.03 (1H, d, *J* = 8.5, H-10); 8.61 (1H, s, H-6). Found, %: C 81.54; H 5.60; N 4.56. $C_{41}H_{34}N_2O_3$. Calculated, %: C 81.70; H 5.69; N 4.65.

5'-Allyloxy-5-(4,5-diphenyl-1,3-oxazol-2-yl)-1',3',3'-trimethylspiro[benzo[f]chromene-3,2'-indoline] (1k). Yield 50%; mp 188–189.5°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.27 (3H, s, 3'-CH_3); 1.42 (3H, s, 3'-CH_3); 2.74 (3H, s, 1'-CH_3); 4.46 (2H, ddd, *J* = 5.4, *J* = 2.6, *J* = 1.3, 5'-$OC\underline{H}_2CH=CH_2$); 5.28 (1H, dq, *J* = 10.4, *J* = 1.4, 5'-$OCH_2CH=C\underline{H}_2$); 5.44 (1H, dq, *J* = 17.2, *J* = 1.6, 5'-$OCH_2CH=C\underline{H}_2$); 5.90 (1H, d, *J* = 10.5, H-2); 6.10 (1H, ddt, *J* = 17.2, *J* = 10.6, *J* = 5.3, 5'-$OCH_2C\underline{H}=CH_2$); 6.44 (1H, d, *J* = 8.3, H-7'); 6.73 (1H, dd, *J* = 8.3, *J* = 2.5, H-6'); 6.80 (1H, d, *J* = 2.5, H-4'); 7.14–7.18 (2H, m, H Ph); 7.26–7.40 (7H, m, H-8, H Ph); 7.56 (1H, ddd, *J* = 1.4, *J* = 6.8, *J* = 8.4, H-9); 7.61–7.64 (2H, m, H Ph); 7.66 (1H, d, *J* = 10.5, H-1); 7.86 (1H, d, *J* = 8.1, H-7); 8.05 (1H, d, *J* = 8.5, H-10); 8.62 (1H, s, H-6). Found, %: C 81.59; H 5.81; N 4.59. $C_{41}H_{34}N_2O_3$. Calculated, %: C 81.70; H 5.69; N 4.65.

5-(4,5-Diphenyl-1,3-oxazol-2-yl)-1'-isobutyl-3,3-dimethylspiro[benzo[*f*]chromene-3,2'-indoline] (1l). Yield 38%; mp 180–181°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.87 (3H, d, *J* = 6.7, 1'-$CH_2CH(C\underline{H}_3)_2$); 0.91 (3H, d, *J* = 6.6, 1'-$CH_2CH(C\underline{H}_3)_2$); 1.27 (3H, s, 3'-$CH_3$); 1.42 (3H, s, 3'-$CH_3$); 2.06 (1H, m, 1'-$CH_2C\underline{H}(CH_3)_2$); 3.00 (1H, dd, *J* = 14.4, *J* = 8.8, 1'-$C\underline{H}_2CH(CH_3)_2$); 3.06 (1H, dd, *J* = 14.4, *J* = 6.3, 1'-$C\underline{H}_2CH(CH_3)_2$); 5.92 (1H, d, *J* = 10.6, H-2); 6.57 (1H, d, *J* = 7.8, H-7'); 6.87 (1H, td, *J* = 7.4, *J* = 0.9, H-5'); 7.09–7.14 (3H, m, H-4', H Ph); 7.18 (1H, td, *J* = 7.6, *J* = 1.3, H-6'); 7.24–7.29 (3H, m, H Ph); 7.31–7.40 (4H, m, H-8, H Ph); 7.56 (1H, ddd, *J* = 8.4, *J* = 6.9, *J* = 1.4, H-9); 7.59–7.63 (3H, m, H-1, H Ph); 7.85 (1H, d, *J* = 8.1, H-7); 8.04 (1H, d, *J* = 8.5, H-10); 8.58 (1H, s, H-6). Found, %: C 83.77; H 6.09; N 4.64. $C_{41}H_{36}N_2O_2$. Calculated, %: C 83.64; H 6.16; N 4.76.

1'-Allyl-5-(4,5-diphenyl-1,3-oxazol-2-yl)-3',3'-dimethyl-8-methoxyspiro[benzo[*f*]chromene-3,2'-indoline] (1m). Yield 40%; mp 212.5–214°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.28 (3H, s, 3'-CH_3); 1.43 (3H, s, 3'-CH_3); 3.79 (1H, ddt, *J* = 17.3, *J* = 5.2, *J* = 1.7, 1'-C$\underline{H}_2$CH=CH_2); 3.92 (3H, s, 8-OCH_3); 4.04 (1H, ddt, *J* = 17.3, *J* = 4.2, *J* = 2.0, 1'-C$\underline{H}_2$CH=CH_2); 4.98 (1H, dq, *J* = 10.3, *J* = 1.7, 1'-CH_2CH=C$\underline{H}_2$); 5.14 (1H, dq, *J* = 17.2, *J* = 1.8, 1'-CH_2CH=C$\underline{H}_2$); 5.89 (1H, m, 1'-CH_2C$\underline{H}$=CH_2); 5.90 (1H, d, *J* = 10.5, H-2); 6.58 (1H, d, *J* = 7.7, H-7'); 6.89 (1H, td, *J* = 7.4, *J* = 0.9, H-5'); 7.09–7.18 (4H, m, H-4',7, H Ph); 7.19–7.26 (5H, m, H-6',9, H Ph); 7.31–7.39 (3H, m, H Ph); 7.57 (1H, d, *J* = 10.5, H-1); 7.60–7.64 (2H, m, H Ph); 7.94 (1H, d, *J* = 9.3, H-10); 8.52 (1H, s, H-6). Found, %: C 81.56; H 5.77; N 4.76. $C_{41}H_{34}N_2O_3$. Calculated, %: C 81.70; H 5.69; N 4.65.

1'-Allyl-5-(4,5-diphenyl-1,3-oxazol-2-yl)-5',8-dimethoxy-3',3'-dimethylspiro[benzo[*f*]chromene-3,2'-indoline] (1n). Yield 41%; mp 221–222.5°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.28 (3H, s, 3'-CH_3); 1.42 (3H, s, 3'-CH_3); 3.71 (1H, ddt, *J* = 17.2, *J* = 5.4, *J* = 1.6, 1'-C$\underline{H}_2$CH=CH_2); 3.78 (3H, s, 5'-OCH_3); 3.92 (3H, s, 8-OCH_3); 3.97 (1H, ddt, *J* = 17.2, *J* = 4.4, *J* = 2.1, 1'-C$\underline{H}_2$CH=CH_2); 4.98 (1H, dq, *J* = 10.2, *J* = 1.7, 1'-CH_2CH=C$\underline{H}_2$); 5.14 (1H, dq, *J* = 17.2, *J* = 1.7, 1'-CH_2CH=C$\underline{H}_2$); 5.88 (1H, m, 1'-CH_2C$\underline{H}$=CH_2); 5.89 (1H, d, *J* = 10.5, H-2); 6.48 (1H, d, *J* = 8.4, H-7'); 6.69 (1H, dd, *J* = 2.6, *J* = 8.4, H-6'); 6.78 (1H, d, *J* = 2.5, H-4'); 7.12-7.18 (3H, m, H-7, H Ph); 7.21–7.40 (7H, m, H-9, H Ph); 7.57 (1H, d, *J* = 10.5, H-1); 7.62–7.65 (2H, m, H Ph); 7.94 (1H, d, *J* = 9.3, H-10); 8.52 (1H, s, H-6). Found, %: C 79.65; H 5.90; N 4.47. $C_{42}H_{36}N_2O_4$. Calculated, %: C 79.72; H 5.73; N 4.43.

5-(4,5-Diphenyl-1,3-oxazol-2-yl)-1'-isobutyl-8-methoxy-3',3'-dimethylspiro[benzo[*f*]chromene-3,2'-indoline] (1o). Yield 39%; mp 204–205°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.88 (3H, d, *J* = 6.7, 1'-CH_2CH(C$\underline{H}_3$)$_2$); 0.91 (3H, d, *J* = 6.6, 1'-CH_2CH(C$\underline{H}_3$)$_2$); 1.27 (3H, s, 3'-CH_3); 1.42 (3H, s, 3'-CH_3); 2.06 (1H, m, 1'-CH_2C$\underline{H}$(CH_3)$_2$); 3.00 (1H, dd, *J* = 14.5, *J* = 8.8, 1'-C$\underline{H}_2$CH(CH_3)$_2$); 3.06 (1H, dd, *J* = 14.5, *J* = 6.4, 1'-C$\underline{H}_2$CH(CH_3)$_2$); 3.92 (3H, s, 8-OCH_3); 5.92 (1H, d, *J* = 10.5, H-2); 6.58 (1H, d, *J* = 7.8, H-7'); 6.87 (1H, dt, *J* = 7.4, *J* = 0.9, H-5'); 7.10–7.18 (4H, m, H-4',7, H Ph); 7.19–7.28 (5H, m, H-6',9, H Ph); 7.31–7.39 (3H, m, H Ph); 7.56 (1H, d, *J* = 10.5, H-1); 7.60–7.63 (2H, m, H Ph); 7.95 (1H, d, *J* = 9.2, H-10); 8.50 (1H, s, H-6). Found, %: C 81.68; H 6.11; N 4.41. $C_{42}H_{38}N_2O_3$. Calculated, %: C 81.53; H 6.19; N 4.53.

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