

Synthesis and study of some novel chelating photochromic spironaphthoxazines

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This paper reviews a series of some novel spironaphthoxazines containing chelating functional groups, which have been synthesized and investigated by the authors. Physical and spectroscopic characteristics (UV, ¹H-NMR, and elemental analysis) of these compounds have been determined. Photochromic reactions of the synthesized compounds were studied under continuous UV irradiation in solvents of different polarity. Solvent and structural effects on absorption properties and thermal bleaching kinetics of the photomerocyanines of synthesized spironaphthoxazines were investigated. The photomerocyanines obtained under steady irradiation of spiroindolinonaphthoxazines, containing a hydroxyl substituent in the naphthoxazine moiety, form complexes with Al(III), Cu(II), and Fe(II) in polar solvents, while those containing a benzothiazolyl substituent interact with Co(II), Ni(II), and Zn(II). Complexation of metal ions with the merocyanines induces a slight hypsochromic shift of its visible absorption band and drastically slows down its thermal bleaching in the dark.

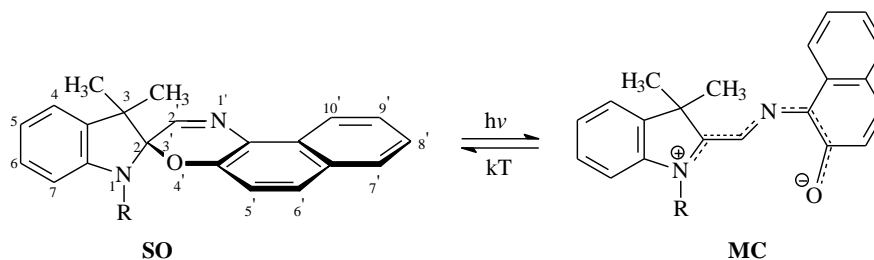
Key words: synthesis, photochromism, complexation, spectral properties, kinetics.

INTRODUCTION

Spirooxazines are a particularly interesting family of photochromic molecules [1, 2] due to their remarkable properties, such as photo-fatigue resistance, strong photocolouration, and fast thermal relaxation [3, 4]. These properties give spiro-oxazines the ability to be applied in many new technologies, for example light filters and optical devices, photochromic liquid crystals, photochromic plastics, photochromic substances useful in lenses, dynamic biosensors and bioelectronic materials, in catalysis, and as metal complex agents [5–14]. The photochromism of these molecules is owing to a photocleavage of the C–O spirobond according to Scheme 1 under UV irradiation and subsequent rotation about a C–C bond to give an open merocyanine (MC) structure (photomerocyanine), which absorbs in the visible region and the molecule is intensively

coloured. The spirooxazines are comprised of two heterocyclic nearly planar moieties (indoline and naphthoxazine) joined by a tetrahedral spiro-carbon which prevents the two π electron systems from conjugation. As a result, the spiro-compounds are almost colourless or pale yellow since the lowest electronic transition of the molecule occurs in the near UV region.

There is a technical interest in photochromic materials based on spiro-compounds [1–14] in which the coloured photomerocyanine once produced cannot revert to the colourless spiro-form, or at least reversion rate is greatly retarded [15, 16]. One way of achieving this is to try to trap the photomerocyanine in a metal-chelated form [17–21] by having suitable substituents in the spiro-compound and metal ions in the solution. This approach has been reported for spiropyranes [17–20] and in single cases for spirooxazines [21, 22].



Scheme 1

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EXPERIMENTAL

All solvents and starting materials, not synthesized in this laboratory, were commercial products (Sigma Aldrich, TCI Europe) and were used without further purification. Synthesis of spirooxazine compounds **0-6** was carried out in a one- to three-step sequence as depicted in Schemes 2–4 [23]. ^1H NMR spectra of the dyes were recorded in CDCl_3 on a Bruker 500 MHz spectro-meter.

A 250-W medium-pressure mercury lamp housed in a light box was used for producing coloured forms and absorption spectra were recorded on an Ocean Optics PC2000 fast scanning spectrometer capable of recording the whole absorption spectrum in the visible region in 10 ms using a 1-cm quartz cell. Irradiation was performed in the spectrophotometer holder at right angles to the monitoring beam. Light was homogeneously spread on cell window to avoid stirring [11].

A general procedure for determining photomerocyanine fading kinetics was as follows. A solution of spiro-compound in a suitable solvent, with a concentration sufficient to give an absorbance of ca. 0.8–1.0 at a corresponding λ_{max} , was exposed to UV light in a quartz cell until the colour was fully developed. SO thermal ring-opening kinetics was measured in the presence of metal ions at a different temperature in the range of 20–50°C about 30 min after having set the temperature control in order to allow initial solution to reach the appropriate temperature in a separate vessel. The ring-closure reaction after photocoloration was monitored directly after switching off light source and scanning the complex between the MC form and the metal ion at the wavelength of maximum absorption (λ_{compl}) at 15-s intervals over a period of 20 min at room temperature. First-order rate constants were obtained from linear $\ln A$ vs. time descending curves. By extrapolation of the obtained $\ln A/t$ plots to zero time, complex absorbance A_0 at $t = 0$ was related to

‘colourability’ [11] using the expression $A_0/c_{\text{SO}} \cdot b$ in which c_{SO} is the initial SO concentration and b is the optical path length.

RESULTS AND DISCUSSION

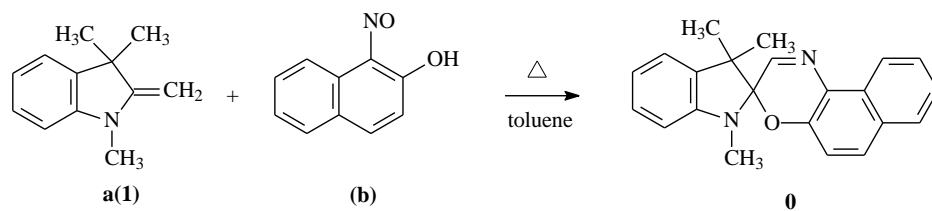
The most common method employed to synthesize spironaphthoxazines is condensation of an alkylidene heterocycle with *o*-nitrosonaphthols in polar organic solvents, such as methanol [8] or chlorinated lower aliphatic hydrocarbons. The present photochromic spironaphthoxazines were prepared by the reaction of 1,3,3-trimethyl-2-methylene indoline, or 1-butyl-3,3-dimethyl-2-methylene indoline, or the corresponding benz[c]indolium perchlorate compounds 1,3,3-trimethyl-benz[c]indolium perchlorate or 1-butyl-3,3-dimethyl benz[c]indolium perchlorate with the matching *o*-nitrosonaphthols [23].

The visible spectra of photomerocyanines **0-6**, acquired by UV irradiation in solvents of different polarity, are characterised by a strong absorption band in the range of 560–660 nm (Table 1). According to data in Table 1, addition of –OH at the 5' position in the naphthoxazine moiety has a minor effect on the visible band of MC derived from compounds **1** and **2** compared to the parent **0**. The effect of benzothiazolyl substituent at the same position reveals a moderate bathochromic shift of 30 to 65 nm in the spectrum of the MC derived from compound **3**. Replacing benzene in the indoline moiety by naphthalene additionally shifts the visible band of the MC originated from compounds **5** and **6** by 15–20 nm to longer wavelengths.

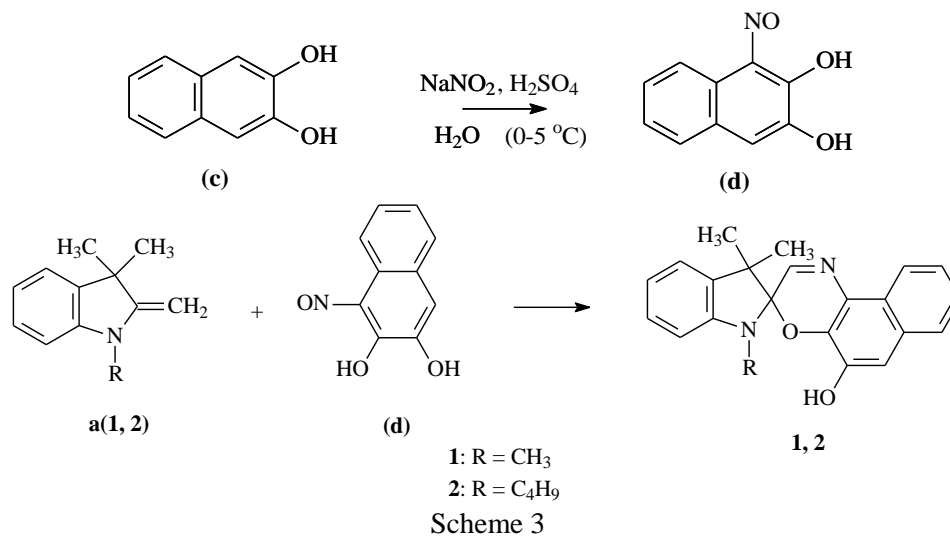
Visible spectra of photomerocyanines derived from compounds **0** and **3-6** are apparently not single absorptions, but show in each case a shorter wavelength shoulder of the main absorption band (Fig. 2) like the spectra of other photomerocyanines of this type [8, 10, 12]. Visible spectra of **1** and **2** reveal only a wide single absorption band (Fig. 3).

Table 1. Experimental values of λ_{max} (nm) and ‘colourability’ ($\times 10^{-4}$) of the visible absorption of photomerocyanines derived from compounds **0-6**

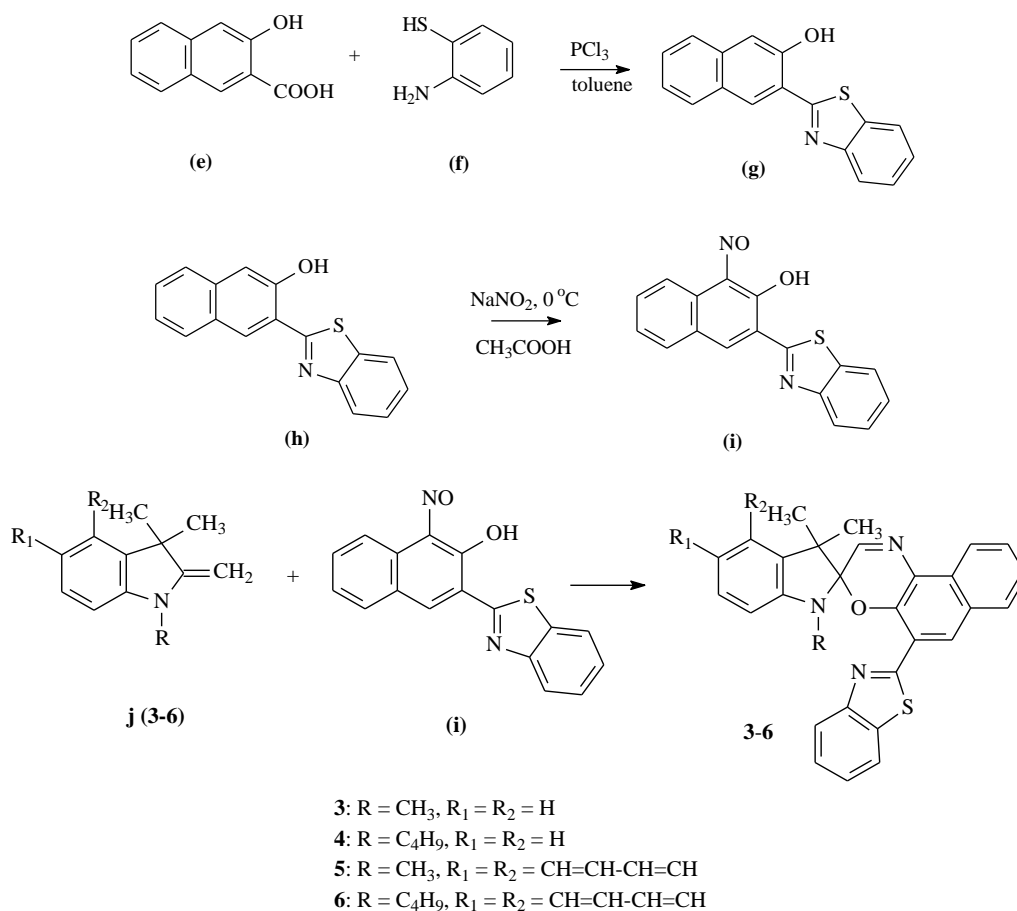
Solvent	λ_{max} (colourability)						
	0	1	2	3	4	5	6
hexane	560 (4.55)	565 (6.87)	570 (6.96)	625 (6.61)	630 (6.84)	642 (6.46)	648 (7.25)
acetone	600 (5.50)	590 (4.27)	595 (4.37)	640 (5.89)	642 (7.25)	655 (6.76)	656 (7.28)
acetonitrile	600 (6.92)	595 (5.79)	597 (5.88)	640 (6.19)	645 (6.98)	658 (7.08)	658 (7.24)
ethanol	610 (5.25)	600 (5.94)	605 (6.32)	642 (7.10)	648 (8.45)	660 (6.82)	660 (8.02)



Scheme 2



Scheme 3



Scheme 4

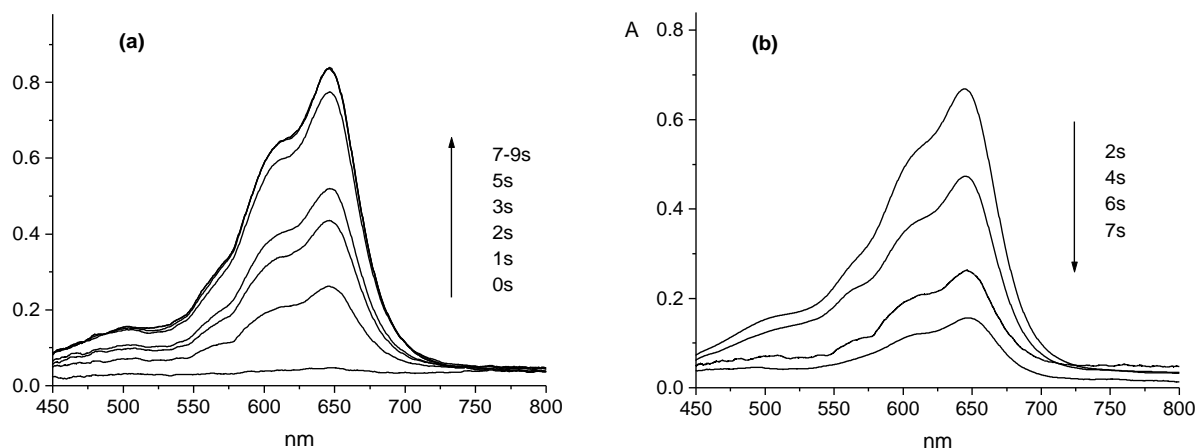


Fig. 2. Visible absorption spectra of 1×10^{-5} M hexane solution of **6** taken: upon 0, 1, 2, 3, 5 or 7-9 s UV irradiation (a) and on keeping the irradiated sample for 2, 4, 6, 7, 10, and 15 s in the dark (b).

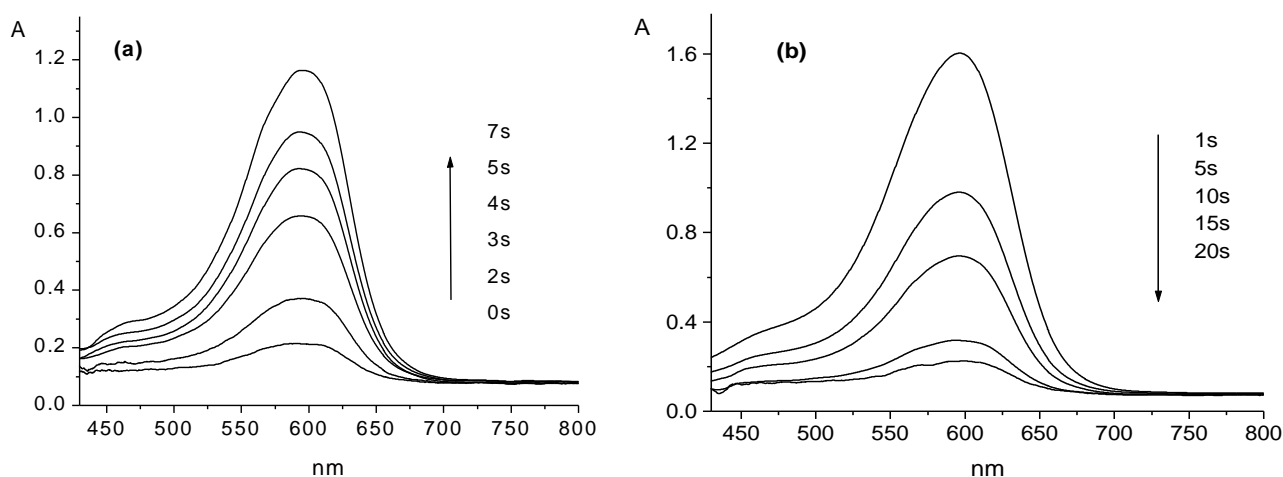


Fig. 3. Visible absorption spectra of 2.5×10^{-5} M acetonitrile solution of **1** taken: upon 0, 1, 3, 4, 5, and 7 s UV irradiation (a) and on keeping the irradiated sample for 1, 5, 10, 15, and 20 s in the dark (b).

'Colourability' [9, 11] of photomerocyanines was calculated from the absorbance A_0 obtained by extrapolation to $t = 0$ of the room temperature linearly descending kinetic curves $\ln A/t$ (Figs. 4 and 5) taken immediately after UV irradiation. Values of A_0/cl are displayed in Table 1. Bathochromic shifts given in Table 1 at a higher solvent polarity, positive solvatochromism, are indicative of a better stabilisation of photomerocyanine-excited state relative to the ground state. A high 'colourability' of the visible absorption bands and observed bathochromic shifts of the spectrum at a higher polarity of the medium are consistent with a $\pi-\pi^*$ character of the singlet excited state of the open forms [10].

Kinetic runs followed immediately after irradiation of **1** or **6** in a range of solvents of different polarity (Figs. 4a,b) show that the thermal ring closure reaction follows first-order kinetics in

concentration of the photomero-cyanine, as the $\ln A/t$ plots are linear. The slopes of the $\ln A/t$ curves gave the first-order rate constants, k . Photomerocyanine relaxation times (τ_{MC-SO}) given in Table 2 were obtained from the first-order rate constants using the expression $\tau = 1/k$. Thus, data in Fig. 4a do not indicate any noticeable dependence of the rate of cyclization of photomerocyanine derived from $-OH$ derivative **1** on solvent polarity and the same is observed about compound **2** and parent unsubstituted compound **0** in Table 2. On the contrary, data in Fig. 4b show that solvent nature has a significant effect on the rate of cyclization of photomerocyanine derived from benzothiazolyl-substituted compound **6** and similar results were obtained for compounds **3-5** in Table 2. In hexane, the merocyanine receives little solvent stabilisation and cyclization is rapid, whereas in acetone and aceto-

nitrile, a relatively higher stabilisation of the polar merocyanine compared to the non-polar spiro-compound is the reason of slower cyclization. The effect is quite pronounced in ethanol, where decolouration reaction is up to 30 times slower than in hexane most probably due to a combined effect of stabilisation of the polar merocyanine and its specific solute-solvent interaction like hydrogen bonding with ethanol. Kinetic runs illustrating the effect of different substituents on colour fading of photomerocyanines in ethanol are presented in Figs. 5a,b. Corresponding values of k and $\tau_{\text{MC-SO}}$ are presented in Table 2. According to these values addition of –OH at the 5' position in the naphthoxazine moiety only slightly increases the relaxation time of photomerocyanines derived from compounds **1** and **2**. Addition of benzothiazolyl substituent at the same position has a stronger effect on the rate of cyclization and the value of MC-SO of the photomerocyanine derived from compound **3** is up to 25 times higher than that acquired for parent unsubstituted compound **0**. These results definitively show that the $\tau_{\text{MC-SO}}$ values vary gradually for **0–6** depending on both structural characteristics and solvent polarity.

Colourless spiro compounds gave no complexes with metal ions. However, the merocyanine form of spirooxazines containing a hydroxyl substituent at the 5' position in the naphthoxazine moiety appeared to form complexes with Al(III), Cu(II), and Fe(II), while those containing a benzothiazolyl substituent interacted with Co(II), Ni(II), Zn(II) in polar solvents.

The visible spectra of photomerocyanines, obtained upon UV illumination of **1** or **2** in polar solvents [46], are characterized by absorption in the

range of 600–620 nm. Irradiation of **1** and **2** in the presence of 1 equivalent of Cu(II), Fe(II) or Al(III) generated a new photo-reversible absorption band in the range of 540–600 nm with band intensity dependent on the $[\text{M(II),(III)}]/[\text{SO}]$ ratio, the highest effectiveness being at 10 equivalents of added metal ion. The observed λ_{max} values (Table 3) are slightly blue-shifted and the same was observed at a 10-fold molar excess of M(II),(III). It is obvious that UV irradiation of SO in the presence of a suitable metal ion yields a MC-M(II),(III) complex and this process is considered a photoinduced complexation. Thermal coordination to the metal ion also takes place, giving a complex, which is spectroscopically identical to the photoinduced product. The colouration obtained in the dark becomes more intense upon UV illumination and reverses back to its initial level after switching off the light.

A strong absorption band in the range of 640–660 nm characterizes visible spectra of photomerocyanines, obtained by UV irradiation of **3–6** in acetone [46]. Insertion of Ni(II), Co(II) or Zn(II) generates a new intense photo-reversible absorption band in the range of 610–640 nm. According to the data reported in Table 4 the new absorption band obtained on adding 5×10^{-5} M acetone solution of Ni(II), Co(II) or Zn(II) to equimolar acetone, hexane (or cyclohexane) solutions of **3–6** is slightly blue-shifted, about 25–45 nm for **5** in acetone. The same was observed at a 10- to 250-fold molar excess of M(II). While λ_{max} depends only moderately on metal ion nature (15–45 nm), the time needed for re-establishing equilibria is sensitive to the metal ion and substantially prolonged compared to non-complexed merocyanine.

Table 2. Rate constants k ($\times 10^2 \cdot \text{s}^{-1}$) of thermal ring closure of photomerocyanines derived from compounds **0–6** and their lifetimes $\tau_{\text{MC-SO}}$ (s)

solvent	0	1	2	3	4	5	6
hexane	52.5 (2)	18.0 (5)	18.7 (5)	18.0 (5)	18.4 (5)	25.2 (4)	22.5 (4)
acetone	44.7 (2)	16.8 (6)	15.9 (6)	5.4 (19)	5.1 (20)	2.5 (40)	2.1 (48)
acetonitrile	54.6 (2)	15.3 (6)	14.2 (7)	5.2 (19)	5.7 (18)	3.3 (30)	2.8 (36)
ethanol	42.9 (2)	13.6 (7)	12.9 (8)	2.0 (50)	1.9 (53)	1.0 (100)	(125)

Table 3. Absorption maxima of complexes obtained upon UV illumination of 5×10^{-5} M solutions of **1** and **2** in the presence of 1 equiv. of metal ion

SO	Solvent	λ_{max} , nm			
		MC	MC-Cu(II)	MC-Al(III)	MC-Fe(II)
1	EtOH/H ₂ O	610	595	585	546
	CH ₃ COCH ₃ /H ₂ O	600	590	580	540
2	EtOH/H ₂ O	620	600	580	546
	CH ₃ COCH ₃ /H ₂ O	600	590	580	540

Table 4. Experimental values of λ_{max} (nm) and ϵ ($\text{M}^{-1} \cdot \text{cm}^{-1}$) of the visible absorption band of Ni(II), Co(II), and Zn(II) complexes with **3–6** in acetone

SO	λ_{max} (nm)		
	MC-Ni(II)	MC-Co(II)	MC-Zn(II)
3	620 (48500)	610 (44500)	600 (36500)
4	625 (51500)	615 (48000)	605 (38500)
5	630 (59000)	620 (54000)	610 (41000)
6	640 (54000)	630 (53500)	620 (40500)

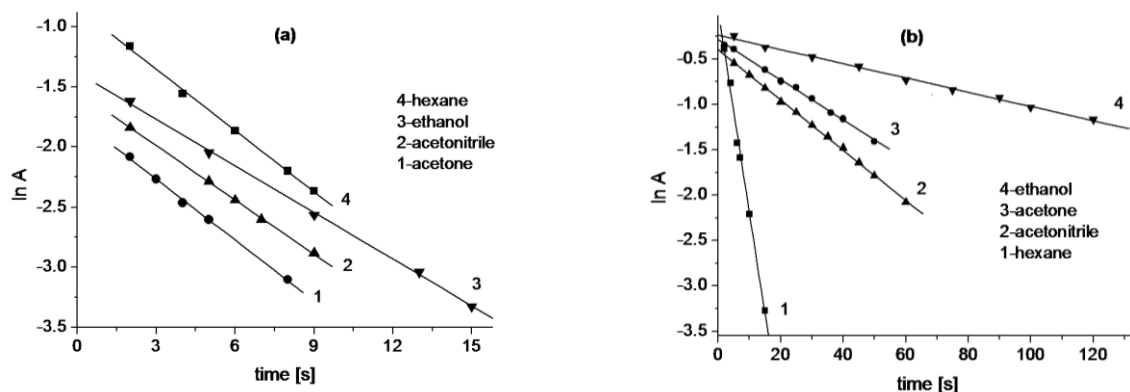


Fig. 4. Kinetic runs ($\ln A$ vs. time) at 600 nm after irradiation of 1×10^{-5} M solutions of **1** (a) and **6** (b) in solvents of different polarity.

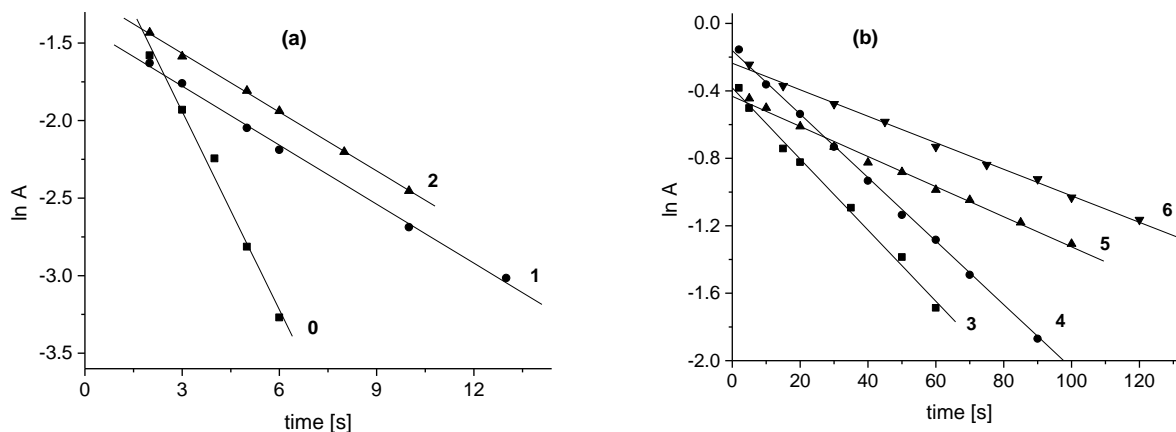


Fig. 5. Kinetics runs ($\ln A$ vs. time) at 600 nm after irradiation of 1×10^{-5} M solutions of **0–2** (a) and **3–6** (b) in ethanol.

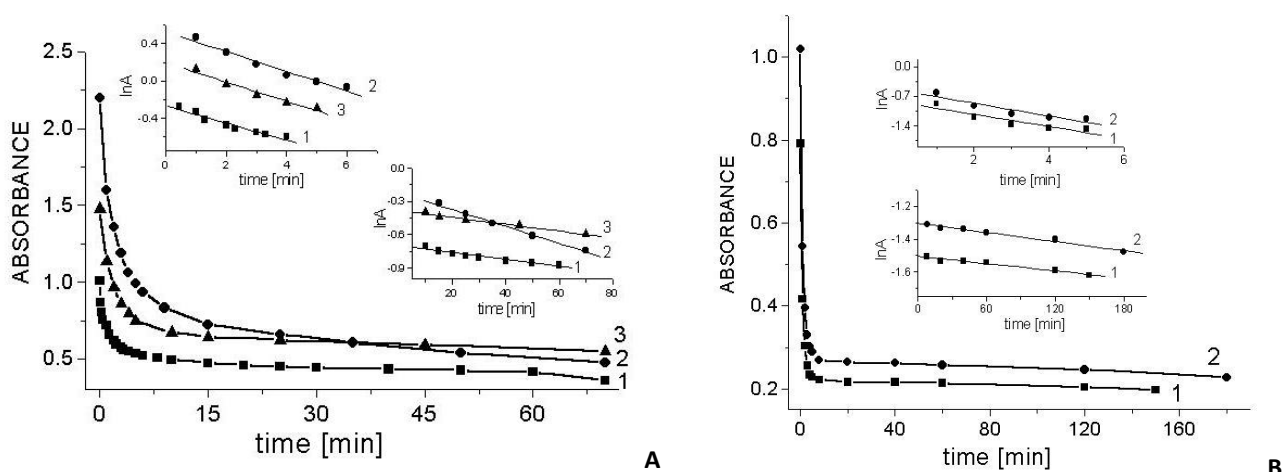


Fig. 6. A). Absorbance changes at 590 nm following 15-s UV illumination of an ethanol solution of **1** after insertion of: (1) 0.5 or (2) 10 equivalents of Cu(II) in ethanol and (3) 10 equivalents of aqueous Cu(II). Insets: $\ln A$ vs. time plots, initial (0.5–6 min) and final (10–70 min); B). Absorbance changes at 600 nm following 15-s UV illumination of an ethanol solution of **2** after insertion of: (1) 0.5 or (2) 1 equivalent of aqueous Cu(II). Insets: $\ln A$ vs. time plots, initial (1–5 min) and final (10–180 min).

Photoirradiation of the parent-unsubstituted spiroindolinonaphthoxazine (Scheme 1) in the presence of either metal salt [28] induces irreversible photo-degradation without any complexation indicating that the nitrogen atom at the 1' position in the naphthoxazine moiety takes no part in chelation.

In the absence of metal ion, the first-order decay of the photomerocyanines of **1** and **2** occurs with $k_r \sim 0.1 \text{ s}^{-1}$ [24] and the relaxation time in acetone and ethanol is $\tau_{\text{MC-SO}} \sim 10 \text{ s}$. Taking into account that colouring and fading times are important characteristics of the photochromic systems, we have studied the effect of chelation on the kinetics of spiroindolinoxazine photochromic transformations. A typical picture of bleaching after irradiation of **1** and **2** in the presence of Al(III), Cu(II) or Fe(II) is shown in figures 6A and 6B. Kinetic runs in figure 6A followed at k_{compl} immediately after illumination of ethanol (plots 1 and 2) or 1:1 aqueous/ethanol (plot 3) solutions indicate that the photoinduced MC-Cu(II) complex of **1** undergoes spontaneous thermal decolouration. It is first order in concentration of the starting complex as the $\ln A$ vs. time plots (initial inset in Fig. 6A) are linear ($q > 0.98$) at about 50% of reaction completion. Similar changes are observed in figure 6B for the MC-Cu(II) complex of **2**. A strongly descending part of the curves associated with thermal spiroindolinoxazine ring closure, analysed by the least squares method, give metal-dependent values of the re-equilibration rate constant k_r ranging from 1.5 to $2.6 \times 10^{-3} \text{ s}^{-1}$ at 20°C .

Relaxation times ($\tau_{\text{com-SO}}$) of 5–10 min for **1,2** and of 2–42 min for **3–6** were obtained using the expression $\tau = 1/k_r$. The rate constants calculated by the least squares method from the final $\ln A$ vs. time linear plots are ca. $5 \times 10^{-5} \text{ s}^{-1}$ for **1** and $1 \times 10^{-5} \text{ s}^{-1}$ for **2** in 1:1 aqueous/ethanol (up to $1 \times 10^{-4} \text{ s}^{-1}$ in ethanol).

CONCLUSION

We have successfully designed and synthesized six novel spiroindolinonaphthoxazines containing chelating functional groups. The photochromic properties of the parent spiroindolinonaphthoxazine were modulated by introduction of substituents at different positions of the molecular framework. A thermal ring closure reaction was strongly suppressed when a benzthiazolyl substituent was attached to the 5' position in the naphthoxazine moiety and benzene was replaced by naphthalene in the indoline fragment. A combined effect of both substituents could be assigned to a decreased rotational freedom upon cyclization. High 'colourability' values (up to 8.5×10^4) suggest high effectivity of the colour-forming reaction. As a rule,

substituents have stronger influence than solvents and provide an active method of tuning and designing new photochromic molecules. Coordination of Al(III), Fe(II), and Cu(II) with the open MC form of 5'-(hydroxy)spiroindolino(-)naphthoxazines and of Ni(II), Co(II) or Zn(II), with the open MC form of 5'-(benzthiazol-2-yl)spiroindolinonaphthoxazines takes place thermally (slowly) or photochemically in a suitable solvent yielding a reversibly photochromic complex. The thermal build-up reaction ($\sim 10^{-3} \text{ s}^{-1}$) is independent of $[\text{M(II)}]$, which implies that ring opening is the rate-determining step. Chelation induces a 10–45-nm hypsochromic shift of the visible absorption band of the photomerocyanine and drastically slows down thermal bleaching in the dark.

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СИНТЕЗ И ИЗСЛЕДВАНЕ НА НОВИ ХЕЛАТНИ ФОТОХРОМНИ СПИРОНАФТОКСАЗИНИ

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(Резюме)

Статията е обзор върху серия нови спирооксазини, съдържащи различни хелатни функционални групи, които са синтезирани и изследвани в нашата лаборатория. Определени са физичните и спектроскопски характеристики (UV, ¹H-ЯМР и елементарен анализ). Фотохимичната реакция на синтезираните съединения е изследвана при непрекъснато облъчване с УВ светлина в разтворители с различна полярност. Изследвани са ефектът на разтворителя и структурата на съединенията върху абсорбционните свойства и кинетиката на термично обезцветяване на мероцианиновата форма на синтезираните спиронафтоксазини. Фотомероцианините, получени при стационарно облъчване с УВ светлина на спиронафтоксазините, съдържащи хидроксилен заместител в нафт(о)ксазиновата част, образуват комплекси с Al(III), Cu(II) и Fe(II) в полярни разтворители, докато тези, съдържащи бензотиазолилов заместител взаимодействат с Co(II), Ni(II) и Zn(II). Комплексообразването между мероцианиновата форма и металните йони се характеризира със слаб хипсохромен ефект върху абсорбционната ивица във видимата област и значително понижение на скоростта на рециклизация.