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Journal of Photochemistry and Photobiology A: Chemistry 189 (2007) 161–166
Journal of Photochemistry and Photobiology A:Chemistry
www.elsevier.com/locate/jphotochem

Novel photochromic spirocyclic compounds of thienopyrroline series: 1 Spiropyrans

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Received 2 November 2006; received in revised form 27 December 2006; accepted 16 January 2007
Available online 30 January 2007

Abstract

Novel spiropyrans of the thienopyrroline series have been synthesized and their photochromic properties studied and compared with those of the indoline analogues. As compared with the indoline analogues, the absorption bands of the cyclic forms of the thienopyrroline spiropyrans are shifted to the blue spectral region, whereas those of the ring-opened forms are shifted to the red region. The merocyanine isomers of the thienopyrroline spiropyrans demonstrate greater thermal stability than their indoline analogues. At the same time thienopyrroline spiropyrans are more photodegradable and less fatigue resistant than the indoline congeners.

Keywords: Spiropyrans; Thienopyrrolenines; Photochromism; Fluorescence

1. Introduction

Spiropyrans (SPPs) are among the most amply studied families of photochromic compounds [1–3]. Exhibiting high quantum efficiency of their photoinitiated rearrangements governed by the reversible photochemical cleavage of the C_{spiro}–O bond in the 2*H*-chromene ring and possessing high two-photon absorption coefficients, the photochromic spiropyrans have a significant potential for diverse technical applications such as molecular switches and high-density photochemical erasable memories [4,5]. The majority of the currently studied spiropyrans relates to the indoline series [1–3]. At the same time, the replacement of a benzene ring in the indoline moiety by a π-electron isologue, e.g. thiophene ring, like in the spirocyclic thieno[3,2-*b*]pyrrole systems may result in the appearance of new useful properties of photochromic spiropyrans. Therefore, the goal of this work was to study the photochromic properties of spiropyrans based on thienopyrroline derivatives. Recently, we developed a convenient method for the synthesis of 3*H*-thieno[3,2-*b*]pyrroline and 3*H*-benzothieno[3,2-*b*]pyrrole derivatives under the Fischer reaction conditions and prepared the first spiro compounds and merocyanine dyes based on these heterocycles [6–9]. This work describes the synthesis of spiropyrans of the thienopyrroline series and investigation into their spectral and photochromic properties in comparison with those of indolinospiropyrans.

2. Experimental

2.1. Synthesis

2,5,6,6-Tetramethyl-6*H*-thieno[3,2-*b*]pyrrole **1a** [6], ethyl 2,5,6,6-tetramethyl-6*H*-thieno[3,2-*b*]pyrrole-3-carboxylate **1b** [7] were prepared following the previously developed procedures. Spiropyrans of indoline series **4a–c** were prepared by

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doi:10.1016/j.jphotochem.2007.01.026

the condensation of 1,3,3-trimethyl-2-methyleneindoline with corresponding hydroxyaldehyde [10]. Commercially available (Merck, Acros, Aldrich) samples of triethylamine, methyl triflate and anhydrous (99.9%) acetonitrile, methanol and ethanol were used.

2.2. Instruments and spectral measurements

The ^{1}H and ^{13}C NMR spectra were recorded on Bruker WM-250 and Bruker AM-300 $CDCl_3$ spectrometers and the EI mass spectra were run on a Kratos instrument (70 eV) with direct sample injection into the ion source. Melting points were measured on a Boetius hot stage and were not corrected. The completion of the reactions was detected using TLC (Silufol UV-254, elution with petroleum ether (60–80 °C)–ethyl acetate, 12:1). Column chromatography was performed using Acros silica gel (C.A.S.-7631-86-9) (0.060–0.200).

Absorption spectra were recorded with Agilent 8453 diode array spectrophotometer. Irradiation light was brought into the temperature-controlled cell compartment at 90° from a 250 W high-pressure mercury lamp equipped with glass filters for allocation of mercury lines. The monochromatic light intensity was determined directly in the stirred 1 cm × 1 cm quartz cell using either potassium ferrioxalate ($I_0^{365} = 3.3 \times 10^{-5}$ mol l^{-1} s^{-1}) or Aberchrome 540 ($I_0^{546} = 4.98 \times 10^{-5}$ mol l^{-1} s^{-1}) [11]. The solutions were stirred with a magnetic bar driven by a speed controlled motor. Low temperature absorption spectra were recorded using a homemade quartz cryostat with working temperature range of 77–293 K. Luminescence emission and excitation spectra were recorded with a Varian Cary Eclipse spectrofluorimeter.

The solvents were EtOH, i-PrOH, MeCN, DMF, acetone and toluene of the spectroscopic grade (Acros Organics). In the course of fatigue resistance property studies the initial absorbance of the solutions at the irradiation wavelength was the same in order to compare the efficiency of photoinduced degradation process.

2.3. Preparation of spiropyrans of thienopyrroline series (**3a–c**, general procedure)

A solution of the thienopyrrolenine (2.6 mmol) and the alkylating agent (2.6 mmol) in 10 ml of anhydrous acetonitrile was refluxed for 1–6 h under argon. The solvent was evaporated and anhydrous alcohol (10 ml), the corresponding salicylic aldehyde (2.2 mmol) and triethylamine (0.23 g, 2.2 mmol) were added. The reaction mixture was refluxed for 1 h, cooled and the solvent evaporated *in vacuo*.

2.3.1. 4′-Ethyl-2′,6′,6′-trimethylspiro[2H-1-benzopyran-2,5′-thieno[3,2-b]pyrroline] **3a**

The procedure above described was applied using 2,5,6,6-tetramethyl-6*H*-thieno[3,2-*b*]pyrrole **1a** (2.6 mmol, 0.47 g), 2-hydroxybenzaldehyde (2.2 mmol, 0.27 g) and ethyl iodide that was used as the alkylating agent. The solution was refluxed for 6 h. Column chromatography using 12:1 petroleum ether–ethyl acetate mixture as the eluent gave compound **3a** (0.3 g, 40%) as a white solid. Crystallization from hexane/ether (6:1) gave colorless crystals, m.p. 72–74 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.19 (3H, t, CH_3CH$_2$, J = 7.2 Hz); 1.19 (3H, s, C(CH_3)$_2$); 1.28 (3H, s, C(CH_3)$_2$); 2.49 (3H, s, CH_3, thiophene); 3.07 (1H, dq, ABX$_3$ CH_2CH$_3$, part A, J_{AX} = 7.2 Hz, J_{AB} = 12.5 Hz); 3.13 (1H, dq, ABX CH_2CH$_3$, part B, J_{BX} = 7.2 Hz, J_{AB} = 12.5 Hz); 5.71 (1H, d, CH = CH–Ph, J = 10.5 Hz); 6.35 (1H, s, CH, thiophene); 6.75 (1H, d, CH = CH–Ph, J = 10.5 Hz); 6.87–7.17 (4H, m, CH, benzene) ppm. MS (IE): m/z (%) = 311 (32) [M$^+$], 296 (9) [M$^+$−15], 282 [M$^+$−29]. $C_{19}H_{21}NOS$ (311.44): Calcd. C 73.27, H 6.80, N 4.50, S 10.30; found C 73.08, H 6.78, N 4.11, S 9.94.

2.3.2. 4′-Ethyl-6-nitro-2′,6′,6′-trimethylspiro[2H-1-benzopyran-2,5′-thieno[3,2-b]pyrroline] **3b**

The procedure above described was applied using 2,5,6,6-tetramethyl-6*H*-thieno[3,2-*b*]pyrrole **1a** (2.6 mmol, 0.47 g), 2-hydroxy-5-nitrobenzaldehyde (2.2 mmol, 0.27 g) and ethyl iodide as the alkylating agent. The solution was refluxed for 6 h. Column chromatography using 8:1 petroleum ether–ethyl acetate mixture as the eluent gave compound **3b** (0.23 g, 25%) as a white solid. Crystallization from petroleum ether gave colorless crystals, m.p. 112–114 °C (lit. [6], 110–112 °C).

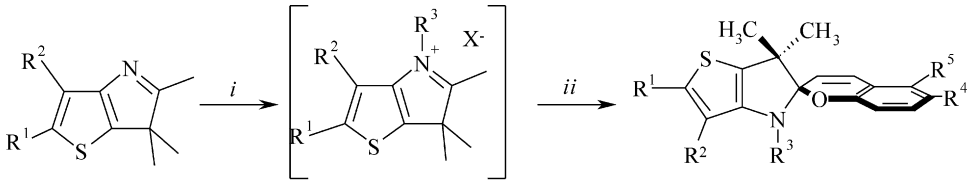
2.3.3. 3′-Carbomethoxy-2′,4′,6′,6′-tetramethylspiro[3H-naphtho[2,1-b]pyran-3,5′-thieno[3,2-b]pyrroline] **3c**

The procedure above described was applied using ethyl 2,5,6,6-tetramethyl-6*H*-thieno[3,2-*b*]pyrrole-3-carboxylate **1b** (2.6 mmol, 0.62 g), 2-hydroxy-1-naphthalenecarboxaldehyde (2.2 mmol, 0.38 g). Methyl triflate was used as the alkylating agent. The solution was refluxed for 1 h. Column chromatography using 3:1 petroleum ether–ethyl acetate mixture as the eluent gave compound **3c** (0.84 g, 79%) as a white solid. Crystallization from hexane gave colorless crystals, m.p. 126–128 °C. ^{1}H NMR (250 MHz, $CDCl_3$): δ 1.25 (3H, s, C(CH_3)$_2$); 1.30 (3H, s, C(CH_3)$_2$); 2.63 (3H, s, CH_3, thiophene); 2.83 (3H, s, N–CH_3); 3.86 (3H, s, CO$_2$CH_3); 5.85 (1H, d, CH = CH–Ph, J = 10.3 Hz); 7.08 (1H, d, CH, naphthalene, J = 8.8 Hz); 7.31–7.77 (4H, M, CH, naphthalene); 7.56 (1H, d, CH = CH–Ph, J = 10.3 Hz); 8.03 (1H, d, CH, naphthalene, J = 8.8 Hz). MS (IE): m/z (%) = 405 (100) [M$^+$], 390 (25) [M$^+$−15], 358 (58) [M$^+$−47]. $C_{24}H_{23}NO_3S$ (405.51): Calcd. C 71.08, H 5.72, N 3.45; found C 71.02, H 6.01, N 3.66.

3. Results and discussion

3.1. Synthesis

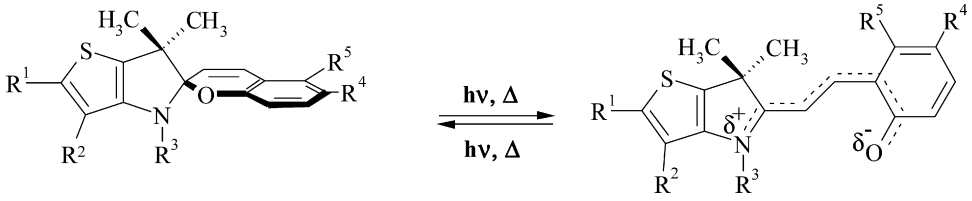
The alkylation of compounds **1a**,**b** by ethyliodide or methyl triflate in acetonitrile is the first stage of the synthesis. The ammonium salts **2a**,**b** were used in the next stage without preliminary separation. The spiropyrans **3a–c** were obtained by the reaction of **2a**,**b** with the corresponding salicylic aldehyde in ethanol in the presence of triethylamine. The total yields of the targeted spiropyrans were in the range of 25–79% depending on the substituents in the thiophene ring.



1a,b **2a,b** **3a-c**

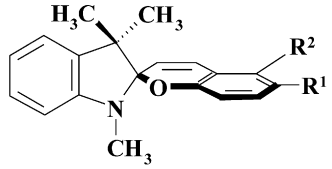
i, EtI or MeOTf, MeCN; *ii*, Et_3N, aldehyde, EtOH;

1a: $R^1 = CH_3$, $R^2 = H$; **1b:** $R^1 = CH_3$, $R^2 = COOCH_3$;
2,3a: $R^1 = CH_3$, $R^3 = C_2H_5$, $R^2 = R4 = R5 = H$;
2,3b: $R^1 = CH_3$, $R^3 = C_2H_5$; $R^4 = NO_2$, $R^2 = R^5 = H$;
2,3c: $R^1 = R^3 = CH_3$; $R^2 = COOCH_3$; $R^4 + R^5 = C_4H_4$



A **B**

3a: $R^1 = CH_3$, $R^3 = C_2H_5$, $R^2 = R4 = R5 = H$;
3b: $R^1 = CH_3$, $R^3 = C_2H_5$; $R^4 = NO_2$, $R^2 = R^5 = H$;
3c: $R^1 = R^3 = CH_3$; $R^2 = COOCH_3$; $R^4 + R^5 = C_4H_4$



4a: $R^1 = CH_3$, $R^2 = H$; **4b**: $R^1 = NO_2$, $R^2 = H$; **4c**: $R^1 + R^2 = C_4H_4$

3.2. Absorption spectra of the spiro forms

In solution, the longest wavelength absorption band of the ring-closed spiro forms **A** of spiropyrans **3a–c** has a maximum in the spectral region of 292–348 nm (molar absorption coefficients are 5410–7730 l mol^{-1} cm^{-1}). The absorption bands of the thienopyrroline spiropyrans **3a–c** are blue shifted with respect to those of the indoline analogues **4a–c**. Table 1 contains the data on spectral properties of spiropyrans **3**, **4**.

As similar to the indoline SPPs **4**, position of the absorption band maxima of SPPs **3** is slightly affected by the type of solvent. The molar absorption coefficients of thienopyrroline spirobenzopyrans **a** and **b** are less than those of the indoline ones, whereas for the spironaphthopyrans **c** the opposite is true.

3.3. Absorption and fluorescence spectra of the merocyanine form

UV-irradiation of solutions of spiropyrans **3a–c**, **4a–c** result in coloration due to the photoinitiated ring-opening reaction leading to the formation of the merocyanine isomers **B** (Table 2). Since the rate of the back thermal recyclization reaction **B** → **A** is too high at room temperature to observe intense coloration of solutions of spiropyrans they were cooled down to 203 K. Fig. 1 pictures the spectral changes of the photochromic system by example of spiropyran **3c**. The spectral pattern closely resembles that reported for other photochromic spiropyrans [1–3]. The long wavelength absorption maxima of the merocyanines **3a–c** appearing at 525–598 nm and 390–425 nm are assigned to

Table 1
Wavelengths of the absorption maxima (λ_{max}, nm) and corresponding absorption coefficients (ε, l mol^{-1} cm^{-1}) of the cyclic forms **A** of spiropyrans **3a–c** and **4a–c** in toluene and ethanol solutions at 293 K

Compounds	3a	4a	3b	4b	3c	4c
λ_{max} (toluene)	294	297	333	337	345	348
ε (toluene)	5410	6270	7730	8940	7370	5020
λ_{max} (ethanol)	292	295	335	338	343	346
ε (ethanol)	4970	5790	8110	9360	7230	4450

Table 2
Wavelengths (λ_{max}, nm) of the absorption maxima (abs), excitation fluorescence (ex) and fluorescence (flu) of the ring-opened forms **B** of SPPs **3**, **4** in ethanol solutions at 203 K

Compounds	3a	4a	3b	4b	3c	4c
λ_{max} (abs)	598	569	525	518	588	572
λ_{max} (ex)	–	–	522	516	585	568
λ_{max} (flu)	–	–	584	566	603	590

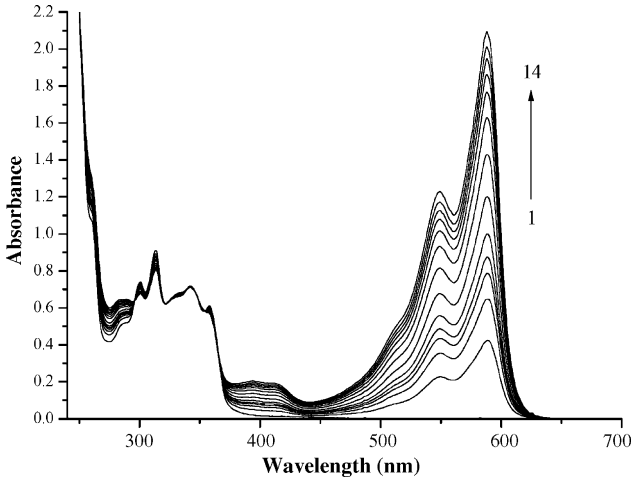


Fig. 1. Absorption spectra of the compound **3c** ([c] = 1.0×10^{-4} mol l^{-1}) in EtOH at $T = 203$ K under UV irradiation ($\lambda_{ir} = 365$ nm, $\Delta t = 30$ s).

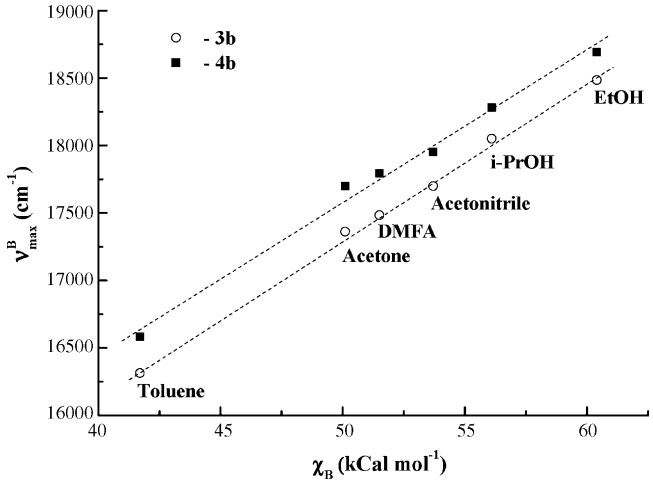


Fig. 2. The linear relationship between the wavenumber of the absorption maximum of the colored forms of spiropyrans **3b** and **4b** and the χ_B (blue shift) Brooker's solvatochromic parameters.

$S_0 \rightarrow S_1$ and $S_0 \rightarrow S_2$ electronic transitions in the most stable merocyanine conformer (Fig. 1).

The long wavelength absorption of merocyanine isomers of the thienopyrroline spiropyrans **3a–c** is red shifted as compared with that of the indoline derivatives **4a–c** (Table 2). Irradiation of the preliminarily UV-activated SPPs solutions in the longest wavelength spectral region of merocyanine forms **B** induces the back **B** → **A** photoreaction.

In ethanol solutions at $T = 203$ K, the merocyanine forms **B** of spiropyrans **3b** and **3c** exhibit fluorescence with maxima at 584 and 603 nm, respectively. The fluorescence excitation spectra conform to the absorption ones (Table 2). The fluorescent maxima of **3b** and **3c** are red shifted relative to those of **4b** and **4c**, respectively (Table 2).

3.4. Solvent effect on the spectra of the merocyanine forms

In order to know the polarity of each merocyanine form, we have measured the absorption spectra of the thienopyrroline derivatives **3b** and **3c** in several solvents and compared the wavenumbers of the absorption maxima against the Brooker's blue and red parameters [12,13]. As shown in Fig. 2, a linear correlation exists between the wavenumber ν_{max}(**B**) and the Brooker's blue parameter (χ_B) [14] for thienopyrroline nitro-substituted spirobenzopyran **3b**. Such a behavior associated with decrease in λ_{max} with increase in solvent polarity corresponds to the negative solvatochromism [15] (Table 3). The same type behavior is characteristic also of indoline nitro-substituted spirobenzopyran **4b** (Fig. 2). On the other hand, spirothienopyrrolinonaphthopyran **3c** exhibits positive solvatochromism as exemplified by a linear correlation of ν_{max}(**B**) with Brooker's red parameter (χ_R) (Fig. 3).

Table 3
Absorption band maxima of the ring-opened forms of SPPs in various solvents at 293 K

Compounds	λ_{max}					
	Toluene	Acetone	DMF	Acetonitrile	i-PrOH	EtOH
3b	613	576	572	565	554	541
4b	603	565	562	557	547	535
3c	564	–	–	572	580	583

The negative solvatochromism of nitro-substituted spirobenzopyrans **3b** and **4b** [1] may be considered as an evidence for pronounced charge delocalization in the ground state of photomerocyanines **B** resulted in stabilization of the bipolar resonance structure. On the contrary, positive solvatochromism of SPP **3c** is a consequence of relatively weak polarity of the ground state of the isomer **B**, for which predominant is quinoidal photomerocyanine structure.

3.5. Kinetics of the thermal bleaching reaction

Kinetics of the back ring-closing reaction of spiropyrans was studied as the relaxation kinetics of merocyanine forms induced upon irradiation of a solution of spirocyclic isomers.

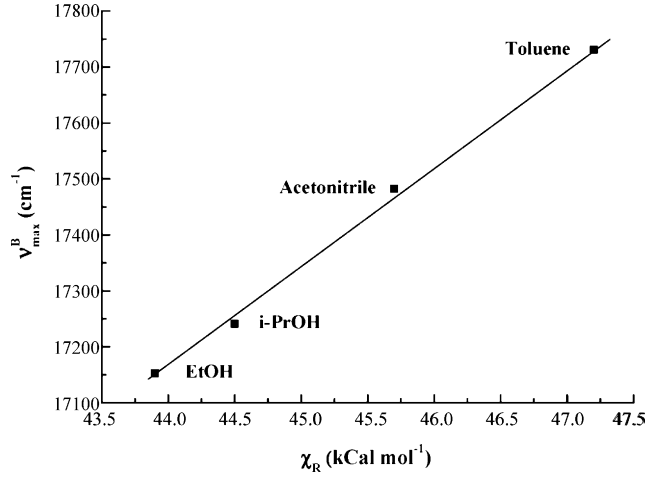


Fig. 3. The linear relationship between the wavenumber of the absorption maximum of the colored forms of spiropyran **3c** and the χ_R (red shift) Brooker's solvatochromic parameters.

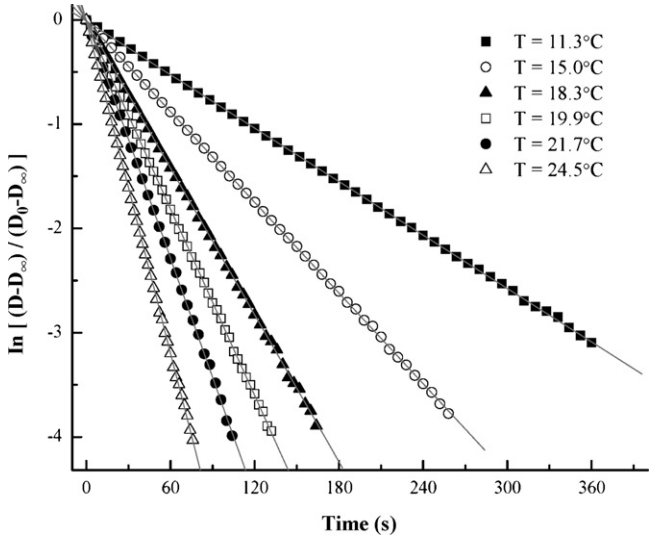


Fig. 4. First-order kinetic plot for the thermal decay of the colored **3b**(**B**) isomers in toluene at various temperatures.

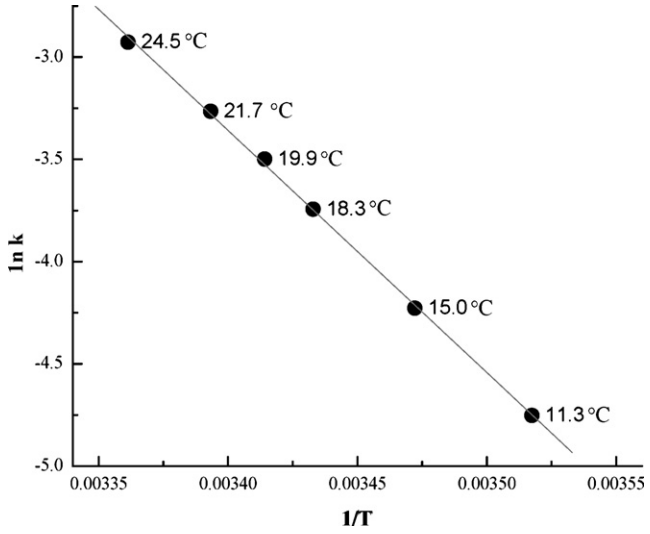


Fig. 5. Arrhenius plot for the thermal decay of the colored **3b**(**B**) isomers in toluene.

Data treatment of the thermal bleaching kinetics showed that in all cases, except for SPP **4b**, the decay followed a first-order reaction which is in good agreement with the data of earlier studies [16–19]. The mono-exponential character of the thermal bleaching kinetics is exemplified by the case of spiropyran **3b** (Fig. 4).

Table 4 contains the rate constants for dark reactions of SPPs. The back thermal processes for the thienopyrroline spiropyrans **3a–c** are slower than those for the indoline derivatives **4a–c**. The reactions in ethanol occur generally slower compared to those in toluene. This fact may be explained as the additional stabilization of merocyanines due to formation of intermolecular H-bonds with solvent molecules.

Activation energy ($E_a = 98.5$ kJ mol^{-1}) for the thermal decay of **3b**(**B**) was determined from the Arrhenius plot (Fig. 5).

3.6. Fatigue resistance

In order to compare the fatigue resistance properties of the thienopyrroline and indoline SPPs, their solutions were investigated under prolonged continuous irradiation similarly to the earlier studies [20–22]. After attainment of the photostationary state of the system its coloration was gradually faded and the absorbance at the long wavelength band belonging to the merocyanine isomers decreased. This spectral behavior is due to irreversible processes of the photodestruction of photochromic system. The photodestruction of the thienopyrroline spiropyrans occurs faster than for their indoline analogues. This trend is illustrated by kinetics of the photoinduced degradation of thienopyrroline, **3b**, and indoline, **4b**, SPPs (Fig. 6).

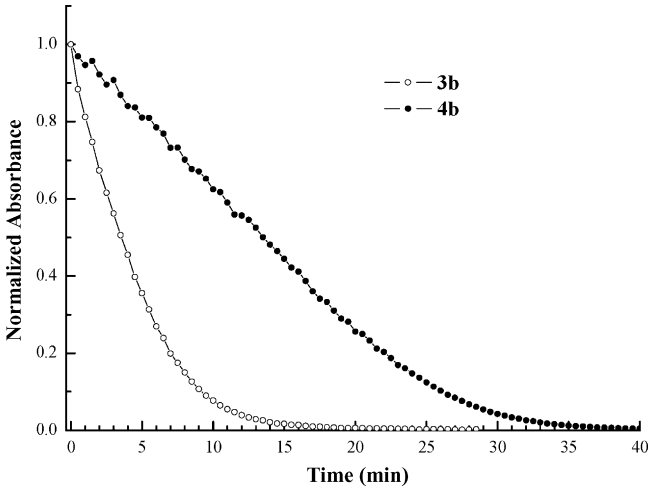


Fig. 6. Comparative kinetics at the respective absorption band maxima of the merocyanines **3b**(**B**) and **4b**(**B**) in toluene under continuous irradiation 365 nm.

According to the earlier data [1,23] photodestruction of spiropyrans is related mainly to the degradation of the merocyanine isomers. Since thienyl group may enhance an intersystem crossing more pronounced photodestruction of the thienopyrroline spiropyrans is likely to result from more effective population of the merocyanine's triplet state which is responsible for photodestruction [24–26].

Table 4
Rate constants of the dark reactions of SPPs in toluene and ethanol solutions at 293 K

Compounds	**3a**	**4a**	**3b**	**4b**	**3c**	**4c**
$k{\cdot}10^{-2}$ s^{-1} (toluene)	–	114	3.0	4.1	71	80.0
$k{\cdot}10^{-2}$ s^{-1} (ethanol)	5.4	15.8	0.005	1.7 0.02	19.2	>500

4. Conclusions

Photochromic spiropyrans of a novel thienopyrroline series were synthesized. As compared with the indoline analogues the thienopyrroline spiropyrans display: (i) blue shifted absorption bands of the cyclic isomers, (ii) red shifted absorption and fluorescence bands of the merocyanine isomers, (iii) lower values

of rate constants for the back thermal processes and (iv) poorer fatigue resistance properties.

Acknowledgments

This work was financially supported by the Russian Foundation for Basic Research (Projects No. 06-03-32988, No. 05-03-08087 and No. 05–03–33191) and by the President of Russian Federation (Project No. NSh-4849.2006.3).

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