# Spectral kinetic characteristics of the photoisomerization products of naphthylmethylideneiminospironaphthopyran induced by photolysis at different wavelengths

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The spectral kinetic characteristics of intermediates generated by photolysis with light at the wavelengths 337 and 430 or 470 nm of the photobifunctional compound (PBC), 1,3-dihydro-5-(2-hydroxy-1-naphthylmethylideneimino)-1,3,3-trimethylspiro[2*H*-indole-2,3-[3*H*]-naphtho[2,1-*b*]pyran], whose molecule combines the spironaphthopyran and hydroxyazomethine fragments, and also parameters of model compounds, *viz.*, naphthylmethylideneimine and spironaphthopyran, were studied in methanol and toluene. The relative quantum yields of formation of different intermediates of PBC were measured relatively to model compounds, namely, *trans*-keto isomer formed due to *cis—trans*-isomerization and prototropic equiliration in the azomethine fragment, and in the merocyanine form generated by spiro bond opening. It was found that the photolysis of the PBC with light at the wavelengths  $\lambda = 430$  or 470 nm nearly no produces the merocyanine form, whereas the relative yield of the *trans*-keto tautomer is ~0.6. For PBC photolysis at  $\lambda = 337$  nm, the yield of the merocyanine form is ~0.2 and the yield of the *trans*-keto isomer decreases substantially (≤0.2). The solvent nature affects the kinetic behavior of the system. The consistency of the isomerization and proton transfer processes is discussed.

**Key words:** photobifunctional compounds, spiropyran, azomethine, photochromism, proton transfer, *cis—trans*-isomerization, laser photolysis.

Investigations in the field of design and research for the properties of photocontrolled hybrid photochromic systems are intensively developed at present. On the one hand, photochromic probes and labels are of interest that make it possible to monitor the properties of a modified object using light.<sup>1</sup> On the other hand, these compounds provide the basis for the development of materials for molecular electronics, namely, optical switches, logical devices, *etc.*<sup>2,3</sup>

We developed the methods for synthesis of the so-called photobifunctional compounds (PBCs) containing two structural fragments different in photochemical properties.<sup>4,5</sup> Spiro cycle opening with the formation of the relatively long-lived colored merocyanine form absorbing in the long-wavelength region is possible in one of the fragments, whereas in another the intramolecular proton transfer and *cis—trans*-isomerization occur resulting in the colored shorter-lived isomer absorbing in a shorter-wavelength region.<sup>6</sup> This specific feature of PBCs can be fruitful for their possible application as molecular switches, especially in the case of selective photoinitiation of different processes by the variation of the exciting light wavelength and the medium.

In this work the spectral kinetic characteristics of the intermediate products (IPs) formed upon photolysis of the earlier<sup>5</sup> synthesized 1,3-dihydro-5-(2-hydroxy-1-naphthyl-methylideneimino)-1,3,3-trimethylspiro[2*H*-indole-2,3-[3*H*]-naphtho[2,1-*b*]pyran] (1) with light at the wavelengths  $\lambda = 337$  and 430 or 470 nm in methanol and toluene were studied by nanosecond laser photolysis. The model compounds, *viz.*, 1-(phenyliminomethyl)-2-naphthol (2) and 1,3-dihydro-1,3,3-trimethylspiro[2*H*-indole-2,3'-[3*H*]-naphtho[2,1-*b*]pyran] (3), were also studied.

Compound **3** was chosen as a fragment of the PBC, because the initial closed form **A** of compound **3** has an intense absorption band at 337 nm and shows nearly no absorption in the region of 400–500 nm (Fig. 1). Photoexcitation of compound **3** with light at the wavelength  $\lambda = 337$  nm affords the open merocyanine form **B** (Scheme 1) with a rather high quantum yield (~0.2).<sup>7,8</sup>

An important property of model naphthylmethylideneimine 2 is the fact that the compound exists in a MeOH

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Scheme 1



solution predominantly as *cis*-ketone  $\mathbf{K}_{cis}$  (Scheme 2) and in toluene as *cis*-enol form  $\mathbf{E}_{cis}$  (we mean the *cis*-rotamer relatively to the hydroxy group of the naphthyl residue and



**Fig. 1.** Absorption spectra of solutions  $(5 \cdot 10^{-5} \text{ mol } \text{L}^{-1})$  of model compound **2** in methanol (*1*) and toluene (*2*) and compound **3** in toluene (*3*).

the azomethine group in the stable *trans*-configuration) with a noticeable content of  $\mathbf{K}_{cis}$ .<sup>9–11</sup> The  $\mathbf{K}_{cis}$  form predominantly absorbs in the wavelength region  $\geq$ 400 nm, providing the possibility of its selective photoexcitation, for example, with light at the wavelength  $\lambda = 430$  or 470 nm (see Fig. 1).



It is assumed that, as in the well studied salicylideneanilines,  $^{11-16}$  the photoexcitation of compound **2** at both 337 and 430 or 470 nm can induce *cis—trans*-isomerization and proton phototransfer with the formation of the corresponding *trans*-forms.

## Experimental

Compounds 1 (see Ref. 5), 2 (see Ref. 17), and 3 (see Ref. 18) were synthesized by earlier described procedures. Purity of the compounds was monitored by TLC, measuring melting points, NMR spectroscopy, and elemental analysis.

Methanol and toluene (for UV spectroscopy) were used to prepare solutions.

Absorption spectra in the visible and UV regions were recorded on a MultiSpec-1501 spectrophotometer.

Absorption spectra and the kinetics of formation and decay of IPs were recorded in the time interval  $\geq 10$  nm on an nanosecond laser photolysis technique with electronic absorption detection.<sup>19,20</sup> An N<sub>2</sub> laser (PRA LN 1000, pulse duration 1 ns, pulse energy 1 mJ, radiation wavelength 337 nm) working in the frequency mode ( $\leq 10$  Hz) or a dye laser (PRA LN, radiation wavelength 102, 430, or 470 nm, pulse duration 0.5 ns, pulse energy 0.2 mJ) with an N<sub>2</sub> laser pump were used as excitation sources. The kinetic curves were averaged (over 4—128 laser pulses) by a UF.258 high-performance analog-to-digital converter (Sweden) connected with a personal computer based on a Pentium 4 processor. The data presented in the work represent the average values obtained by processing at least ten kinetic curves under the indicated conditions.

The relative yields of IP I were determined by comparison of the absorption intensities of the IPs at the maxima of bands measured at the same absorption of the initial solutions at the excitation wavelength assuming that the molar absorption coefficients of the corresponding IPs are equal.

All measurements were performed at ambient temperature.

#### **Results and Discussion**

**Intermediate products of photolysis of model 3.** Pulse photoexcitation of form **A** of compound **3** in methanol and

toluene solutions with light at the wavelength  $\lambda = 337$  nm results in the formation of a single photoproduct: merocyanine form **B** with the characteristic absorption spectrum with maxima at 560 and 550 nm, respectively (Fig. 2).<sup>7,8</sup> The kinetics of disappearance of form **B** is described by the first-order equation with rate constants of 14 (in MeOH) and 42 s<sup>-1</sup> (in toluene). Form **A** of this compound does not absorb in the region of  $\lambda \ge 380$  nm (see Fig. 1, spectrum 3) and, therefore, no measurements were performed for photoexcitation with long wavelengths of light.

Intermediates of photolysis of model compound 2. Pulse photoexcitation of compound 2 in methanol and toluene solutions with light at the wavelengths  $\lambda = 337$  and 430 or 470 nm, *i.e.*, at the absorption bands of both tautomers  $\mathbf{E}_{cis}$  and  $\mathbf{K}_{cis}$ ,<sup>9–11</sup> generates only one IP with the absorption spectrum (in methanol a maximum in the differential spectrum at ~475 nm and a band at 450 nm; in toluene a maximum in the differential spectrum at ~430 nm and a shoulder at 470 nm (Fig. 3)), which is substantially different from the spectrum of the  $\mathbf{K}_{cis}$  form (two bands at 455 and 435 nm with a close intensity, see Fig. 1, spectra *I* and *2*) by the intensity ratio of two absorption bands.

In toluene at the low initial concentration of the IP, the kinetics of its decay obeys the monoexponential law with the rate constant in dilute solutions ~25 s<sup>-1</sup>. At rather high initial concentrations of the IP, the IP disappears according to a bimolecular law with a high rate constant of ~6  $\cdot$  10<sup>8</sup> L mol<sup>-1</sup> s<sup>-1</sup> (in the calculation the molar absorption coefficient at the maximum of the differential absorption spectrum was accepted to be 1  $\cdot$  10<sup>4</sup> L mol<sup>-1</sup> cm<sup>-1</sup>).<sup>9-11</sup>

The IP in methanol disappears considerably more rapidly, and its decay kinetics obeys the monoexponential law with the rate constant in dilute solutions equal to ~9.9  $\cdot$  10<sup>3</sup> s<sup>-1</sup>. An increase in the concentration of compound **2** accelerates the decay of the IP. The corresponding linear dependence on the concentration of compound **2** gives the value of the rate constant of IP "quenching" by the ground state of **2** equal to ~3  $\cdot$  10<sup>7</sup> L mol<sup>-1</sup> s<sup>-1</sup>.



A

**Fig. 2.** Absorption spectra of the intermediates obtained by laser photolysis of compound **3** in methanol (*1*) and toluene (*2*) with light at the wavelength  $\lambda = 337$  nm (the absorbance of the initial solutions at  $\lambda = 337$  nm is 0.4) immediately after a laser pulse.



Fig. 3. Absorption spectra of the intermediates obtained by laser photolysis of compound 2 in toluene (1) and methanol (2) with light at the wavelength  $\lambda = 337$  nm (the absorbance of the initial solutions at  $\lambda = 337$  nm is 0.4) immediately after a laser pulse.

The obtained kinetic characteristics of the IP and specific features of its absorption spectrum are similar to the known characteristics for the IPs of photochemical reactions of other salicylideneanilines.<sup>6,11–16</sup> These IPs were identified earlier as the *trans*-keto form ( $\mathbf{K}_{trans}$ ) of salicylideneanilines. The solvation of  $\mathbf{K}_{trans}$  in proton-donor solvents and the proton exchange with the initial molecules and with other protons favor the decay of  $\mathbf{K}_{trans}$ .<sup>6,15</sup>

It can be assumed that the IP observed 10 ns after photoexcitation is also the  $\mathbf{K}_{trans}$  form of compound 2. In this case, at  $\lambda \ge 400$  nm, where the absorption of the initial solution is mainly caused by the  $\mathbf{K}_{cis}$  form of 2, no bleaching (absorption decrease) of the solution is observed even upon photolysis with the long-wavelength light. The decay kinetics of the induced absorption is independent of the observation wavelength. It is difficult to substantiate the assumption about the fact that the molar absorption coefficient of the  $K_{trans}$  form of 2 is substantially higher than the analogous characteristic of the  $\mathbf{K}_{cis}$  form of 2 in the whole range of  $\lambda \ge 400$  nm. It is most likely that the initial concentration of the  $\mathbf{K}_{cis}$  form of **2** is appreciably recovered after 10 ns due to prototropic equilibrium, and in toluene (where the equilibrium is shifted to  $\mathbf{E}_{cis}$ ) the concentration is recovered almost completely, whereas in methanol (where the equilibrium is shifted to  $\mathbf{K}_{cis}$ ) the recovery is only partial, which results in a substantial difference between the observed differential spectra of the  $\mathbf{K}_{trans}$  form of 2 in toluene and methanol (see Fig. 3).

The data obtained do not allow one to draw a conclusion about the detailed mechanism of formation and decay of the  $\mathbf{K}_{trans}$  form, in particular, to reveal whether the processes of isomerization and proton transfer are consecutive or occur correlatively through one transition state (see further). However, the result of photolysis independent of the wavelength of the exciting light and of the solvent can indicate that proton transfer and isomerization occur in the same electron-excited state, for example, in the twisted charge-transfer state.<sup>21</sup>

Intermediates of photolysis of the PBCs in MeOH. A comparison of the absorption spectra of compounds 1 and 2 in a methanol solution (Figs 1 and 4) suggests that molecules 1 in a methanol solution exist predominantly in the *cis*-keto form  $A^{K_{cis}}$ . It is expected that the introduction of electron-donor substituents into the phenyl fragment of molecule 2 would shift the prototropic equilibrium to  $K_{cis}$ .<sup>11</sup>

Pulse photoexcitation of  $\mathbf{A}^{\mathbf{K}_{cis}}$  in methanol solutions with light at the wavelength  $\lambda = 430$  or 470 nm results in the reversible bleaching (absorption decrease) at  $\lambda \leq 500$  nm and in a shorter-lived and less intense induced absorption (Figs 5 and 6). The bleaching spectrum (see Fig. 5, spectrum 2) corresponds to the absorption of  $\mathbf{A}^{\mathbf{K}_{cis}}$  (see Fig. 4, spectrum 1), whereas the spectrum of induced absorption (calculated by the subtraction of the spectra observed immediately after a laser pulse and after the disappearance of the induced absorption) contains a maximum at ~490 nm (see Fig. 5, spectrum 3).

By analogy to **2**, it can be assumed that this IP is the *trans*-keto form  $A^{K_{trans}}$  in which no opening of the C–O spiro bond occurred (Scheme 3).



**Fig. 4.** Absorption spectra of solutions  $(5 \cdot 10^{-5} \text{ mol } \text{L}^{-1})$  of compound **1** in methanol (*1*) and toluene (*2*).



**Fig. 5.** Differential absorption spectra of the intermediates obtained by laser photolysis of compound **1** with light at the wavelength  $\lambda = 430$  nm in methanol (0.03 mmol L<sup>-1</sup>) 0.01 µs (*I*) and 1 ms (*2*) after a laser pulse. Spectrum *3* is the difference spectrum resulted from spectra *2* and *3*.



**Fig. 6.** Kinetics of the change in the absorption of the solution at 530 (1), 500 (2), and 450 nm (3) upon laser photolysis of compound **1** in methanol (0.05 mmol L<sup>-1</sup>) with light at the wavelength  $\lambda = 430$  nm.





The decay kinetics of this tautomer obeys the firstorder law with the rate constant  $2.8 \cdot 10^3 \text{ s}^{-1}$  (see Fig. 6), which is somewhat lower than a similar value for  $\mathbf{K}_{trans}$  of model compound **2**. The relative yield of  $\mathbf{A}^{\mathbf{K}_{trans}}$  as compared to  $\mathbf{K}_{trans}$  of compound **2** upon photolysis at  $\lambda = 430$  nm is 0.64.

Unlike the  $\mathbf{K}_{trans}$  form of **2**, the disappearance of  $\mathbf{A}^{\mathbf{K}_{trans}}$  is not accompanied by the recovery of the absorption of the initial compound  $\mathbf{A}^{\mathbf{K}_{cis}}$  at  $\lambda \ge 400$  nm. The recovery of the initial absorption at  $\lambda \ge 400$  nm is rather slow (see Fig. 6). The kinetics of the process also obeys the first-order law with a rate constant of 92 s<sup>-1</sup>. It is difficult to substantiate such a low rate constant for the process of backward proton transfer). It can be assumed that the rate-determining step of recovery of the initial mixture of tautomers **1** is the *trans*—*cis*-isomerization of enol.

Thus, it can be concluded that  $\mathbf{A}^{\mathbf{K}_{trans}}$  is transformed first not into  $\mathbf{A}^{\mathbf{K}_{cis}}$ , but, most likely, into *trans*-enol tautomer  $\mathbf{A}^{\mathbf{E}_{trans}}$  absorbing at  $\lambda \leq 400$  nm (Scheme 4).

#### Scheme 4



**A<sup>E</sup>** trans

Hereinafter the values of k are given in  $s^{-1}$ .

This process is comparatively fast due to proton exchange with molecules of the solvent MeOH.

Then the relatively slow *trans—cis*-isomerization occurs accompanied by the prototropic equilibration (Scheme 5).

Pulse photoexcitation of  $\mathbf{A}^{\mathbf{K}_{cis}}$  in methanol solutions with light at the wavelength  $\lambda = 337$  nm is also accompanied by the formation and decay of a small amount of  $\mathbf{A}^{\mathbf{K}_{trans}}$  (the relative yield is 0.091) and the disappearance of the absorption of the initial mixture of tautomers **1** at



Fig. 7. Differential absorption spectra of the intermediates obtained by laser photolysis of compound 1 with light at the wavelength  $\lambda = 337$  nm in methanol (the absorbance of the initial solutions at  $\lambda = 337$  nm is 0.4) 0.01 µs (1), 1 ms (2), and 20 ms (3) after a laser pulse.

 $\lambda \ge 400$  nm followed by recovery (Fig. 7). The kinetic parameters of these processes coincide with analogous values obtained by photolysis with light at the wavelength  $\lambda = 430$  or 470 nm.

However, a relatively long-lived induced absorption is observed in addition to that of  $A^{K_{trans}}$ . This absorption is characterized by the spectrum (a maximum at 590 nm) similar to the spectrum of form **B** of model compound **3** but shifted to the long-wavelength region (see Fig. 7). The relative yield of form **B** of PBC **1** as compared to the yield of form **B** of compound **3** is 0.17.

The decay kinetics of form **B** of PBC **1** obeys the monoexponential law with a rate constant of 37 s<sup>-1</sup>. This value is noticeably higher than the corresponding value for model compound **3**, which is due to the weak electron-acceptor character of the *cis*-keto form of the salicylideneimine substituent in the indoline fragment.

Thus, it can be assumed that the observed isomer of PBC 1 is the  $\mathbf{B}^{\mathbf{K}_{cis}}$  form (Scheme 6).



Scheme 5



It seem hardly possible that the observed isomer of the open form was form  $\mathbf{B}^{\mathbf{K}_{trans}}$ , since the recovery of the initial absorption of compound 1 at  $\lambda \leq 500$  nm was observed before the disappearance of form **B** of the PBC (see Fig. 7).

Intermediates of photolysis of the PBCs in toluene. An analysis of the state of the model compounds in toluene solutions and a comparison of their absorption spectra with the absorption spectrum of compound 1 in toluene (see Figs 1 and 4) suggest that molecules 1 exist in toluene mainly as form  $A^{E_{trans}}$ , but the fraction of keto form  $A^{K_{cis}}$  remains rather noticeable.

Pulse photoexcitation of compound 1 in toluene with light at the wavelength  $\lambda = 430$  or 470 nm results, as in the case of methanol solutions, in the reversible bleaching of the solution at 400 nm  $\leq \lambda \leq 500$  nm, but no induced absorption is observed immediately after a laser pulse (Figs 8 and 9). The bleaching spectrum (see Fig. 8, spec-



**Fig. 8.** Differential absorption spectra of the intermediates obtained by laser photolysis of compound **1** with light at the wavelength  $\lambda = 430$  nm in toluene (0.05 mmol L<sup>-1</sup>) 0.01 µs (*I*) and 2 ms (*2*) after a laser pulse. Spectrum *3* is the difference spectrum resulted from spectra 2 and *1*.



**Fig. 9.** Kinetics of the change in the absorption of the solution at 470 (*I*) and 450 nm (*2*) upon laser photolysis of compound **1** (0.05 mmol L<sup>-1</sup>) with light at the wavelength  $\lambda = 430$  nm.

trum *I*) correlates with the absorption of compound **1** (see Fig. 4, spectrum 2). However, the recovery kinetics of the initial absorption is described by the biexponential law with rate constants of 970 and 220 s<sup>-1</sup> (see Fig. 9). So, at first (the rate constant is 970 s<sup>-1</sup>) the IP is formed characterized by the absorption spectrum with maxima at ~470 and ≤450 nm (see Fig. 8, spectrum 3). By analogy with the data on photolysis of compound **1** in methanol and photolysis of compound **2** in methanol and toluene, it can be assumed that this IP is the  $A^{K_{trans}}$  form. Its relative yield in toluene as compared to the yield of form  $K_{trans}$  of model compound **2** is 0.57.

The decay kinetics of  $\mathbf{A}^{\mathbf{K}_{trans}}$  in toluene obeys the firstorder law with a rate constant of 220 s<sup>-1</sup> and results in the recovery of the initial absorption (see Fig. 9). Thus, in toluene, unlike the proton solvent methanol, the rate-determining step of the process of returning to the initial state of tautomeric equilibrium is not the *trans*—*cis*-isomerization of enol but the isomerization of  $\mathbf{A}^{\mathbf{K}_{trans}}$  to  $\mathbf{A}^{\mathbf{K}_{cis}}$ , and  $\mathbf{A}^{\mathbf{K}_{trans}}$  is formed, most likely, from  $\mathbf{A}^{\mathbf{E}_{trans}}$ , which is the primary photolysis product (appears less than within 10 ns).

The processes that occur in a toluene solution upon the excitation of compound **1** with light at  $\lambda = 430$  and 470 nm can presumably be presented as Scheme 7.



Pulse photoexcitation of compound **1** in a toluene solution with light at  $\lambda = 337$  nm is also accompanied by the



**Fig. 10.** Differential absorption spectra of the intermediates obtained by laser photolysis of compound **1** with light at the wavelength  $\lambda = 337$  nm in toluene the absorbance of the initial solutions at  $\lambda = 337$  nm is 0.4) 10 ms (1), 2 ms (2), and 0.01 µs (3) after a laser pulse.

formation and decay of a small amount of  $\mathbf{A}^{\mathbf{K}_{trans}}$  (the relative yield of 0.2) followed by the recovery of the absorption of the initial mixture of PBC tautomers at  $\lambda \le 500$  nm (Fig. 10). The kinetic parameters of these processes coincide with analogous values obtained by photolysis with light at  $\lambda = 430$  or 470 nm.

However, a relatively long-lived induced absorption of form **B** with a maximum at 570 nm is observed along with  $A^{K_{trans}}$ . The absorption spectrum of form **B** of compound **1** is similar to that of form **B** of model compound **3** but is slightly shifted to the long-wavelength region (see Fig. 10). The relative yield of form **B** of PBC **1** as compared to form **B** of compound **3** is 0.23. The decay kinetics of form **B** of PBC **1** obeys the monoexponential law with a rate constant of 34 s<sup>-1</sup>. This value, unlike analogous data obtained for experiments in methanol, is even somewhat lower than the corresponding value for compound **3**, which is due, most likely, to the weak electron-donor character of the enol form of the salicylideneimine substituent in the indoline fragment. It can be concluded that the isomer observed is form **B**<sup>E</sup>*cis*.

Thus, three photochromic photoprocesses can occur in parallel in the studied PBC 1: spiro bond opening followed by *cis—trans*-isomerization leading to the formation of the merocyanine form in the spironaphthopyran fragment and the proton transfer and photoisomerization in the azomethine fragment. During these processes an induced absorption is formed in the long-wavelength region, whereas the reversible bleaching of the system occurs in the short-wavelength region. The ratio of efficiencies of different processes depends substantially on the wavelength of the exciting light. The initial and intermediate absorption spectra and the kinetic behavior of the system depend substantially on the solvent nature, which gives additional possibilities of controlling photochromism along with the wavelength of the photolyzing light. This work was financially supported by the Russian Academy of Sciences (Program of the Presidium of the Russian Academy of Sciences No. 7).

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