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Spectral luminescent properties and dynamics of intramolecular processes in 2,4,5-triarylimidazoles

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Abstract

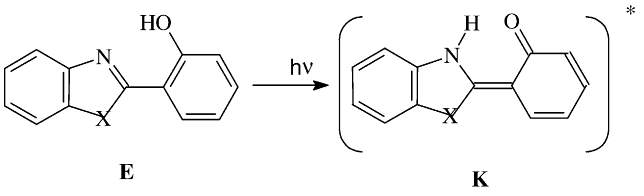
The excited-state intramolecular proton transfer (ESIPT) in some 2,4,5-triarylimidazoles (TAIs) is studied by the methods of steady-state luminescence and femtosecond laser absorption spectroscopy. It is found that ESIPT in 4,5-diphenyl-(2-hydroxyphenyl)imidazoles occurs both in solutions at ambient temperature and in glassy matrices at 77 K. In the case of 4,5-diphenyl-(2-hydroxynaphthyl)imidazole, a rotation of molecular fragments is necessary for ESIPT to occur, which results in the absence of ESIPT at 77 K. The characteristic times of intramolecular processes in the excited state of TAIs are measured by the method of femtosecond absorption spectroscopy. It is shown that the ESIPT time in TAIs depends on the molecular structure—it is extremely short ($\leq$100 fs) in molecules with a planar reaction center and three times as long in TAIs with a non-planar reaction center—being determined by the time of conformational rearrangement.

Keywords: Intramolecular proton transfer; Luminescence; Femtosecond laser spectroscopy; 2,4,5-Triarylimidazoles

1. Introduction

The excited-state intramolecular proton transfer (ESIPT) reactions in organic compounds have been the subject of numerous recent investigations because of the fundamental importance of this process and its potential for practical applications in laser dyes [1–3], photostabilizers [4,5], three-dimensional optical memory devices [6], solar energy concentrators [7], and electroluminescent materials with high photochemical stability [8].

The ESIPT usually involves a very fast transfer of the hydroxyl (or amino) proton to an acceptor such as carbonyl oxygen or a nitrogen atom in an excited state of a molecule, resulting in the formation of the keto-tautomer (**K**) from the initial enole-tautomer (**E**).

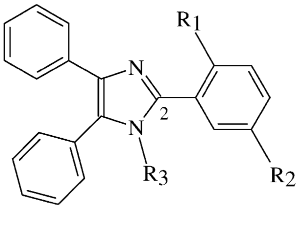


X = S, O, NH

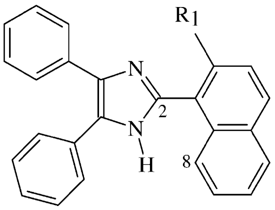
Then, the excited keto-tautomer of the molecule luminesce, undergoing a transition to the ground state, the reverse proton transfer takes place, and the molecule returns to the **E** form.

The ESIPT is usually manifested in steady-state spectra in an significantly large Stokes shift of the fluorescence spectrum relative to the initial absorption spectrum [1–9].

In this paper, we study the ESIPT in some 2,4,5-triarylimidazoles (TAIs) by the methods of steady-state luminescence spectroscopy at different temperatures and femtosecond absorption spectroscopy. The structure of these molecules can be easily changed by introducing various substituents into their peripheral aryl rings, which changes their luminescence significantly [10,11]. The structural formulas of TAIs are:



I, $R_1=R_2=R_3=H$
IIa, $R_1=OH$, $R_2=R_3=H$
IIb, $R_1=OH$, $R_2=Br$, $R_3=H$
IIc, $R_1=OH$, $R_2=H$, $R_3=C_4H_9$



III, $R_1=H$,
IV, $R_1=OH$

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Molecules **I** and **III** without hydroxy substituents, in which ESIPT is absent, are presented for comparison.

2. Experimental details

2.1. Materials

TAIs were prepared and purified as described in [12–14]. Nuclear magnetic resonance and thin-layer chromatography techniques showed no trace of impurities in these samples. Acetonitrile, dioxane, toluene and ethanol were spectral grade and were used as solvents.

2.2. Spectroscopic study

Absorption spectra were measured with a “Specord UV-VIS” spectrophotometer. Luminescence spectra were recorded with a JY-3 spectrofluorimeter. The luminescence spectra were not corrected for the spectral sensitivity of the spectrofluorimeter; however, the spectral sensitivity of an R777-01 photomultiplier used in the spectrofluorimeter was almost constant in the region from 400 to 500 nm.

Femtosecond experiments were performed using a setup assembled in the laboratory of laser spectroscopy at N.N. Semenov Institute of Chemical Physics, RAS. A train of pulses at a wavelength of 616 nm, with a duration of 50 fs, an energy of 300 μJ and a repetition rate of 25 Hz was divided with a beamsplitter into two parts to form a pump pulse and a probe pulse. The second harmonic at a wavelength of 308 nm was used as a pump pulse. A supercontinuum pulse, which was generated by focusing a femtosecond pulse in a quartz cell with distilled water, was used as a probe pulse. The supercontinuum was stable in the region from 380 to 1000 nm, except the 580–630 nm region, where an intense noise was observed due to the supercontinuum properties. For this reason, we ignored this region in the analysis of the spectra. The solutions of TAIs in acetonitrile were studied in a quartz cell of 1 mm thickness. Acetonitrile has no photoinduced absorption upon excitation at 308 nm. The concentration of TAIs was selected so that the absorption of the pump pulse in the cell was about 70–80%. The pump pulse diameter in the focus was 300 μm and that of the probe pulse was 100 μm. A part of the probe pulse was used as a reference beam and also passed through the cell, but in the region not excited by the pump pulse. After passing through the cell, the reference and probe pulses were directed to a prism spectrometer, where their spectra were simultaneously recorded with two CCD cameras. The experimental setup is described in detail in [15].

The supercontinuum pulse is usually strongly chirped and for this reason its different spectral components reach the sample at different instants of time. Therefore, we performed time correction of the recorded spectra. For this purpose, the dependence of the zero delay between pump pulse and the corresponding supercontinuum component on a certain wavelength was obtained from the signal of a reference substance. Pure dioxane was used as a reference substance because it showed photoinduced absorption over the entire supercontinuum. The spectra were corrected as described in [16]. The time resolution was determined from the kinetic curves of the pure dioxane signal. It was 70 ± 5 fs for all the wavelengths in the range under study.

All TAIs exhibited intense fluorescence. A part of this emission entering the spectrometer slit distorted the spectra being registered. But, because excitation region area was 10 times larger than the probe pulse area, we assume that integrated fluorescence of a sample is independent on the delay time. Therefore, we subtracted the fluorescence spectra recorded at negative delay time from all the spectra obtained for other delay times. All the femtosecond experiments were carried out at room temperature.

Semi-empirical quantum–chemical calculations of electron density and molecular geometry optimization were performed using PM3 program (HyperChemTM, Release 5.01 for Windows, Hypercube Inc.).

3. Results and discussion

3.1. Steady-state spectroscopy

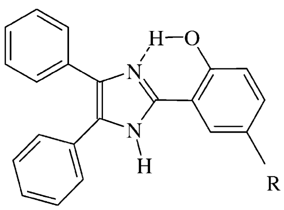
All TAIs studied strongly fluoresce in solutions at room temperature and 77 K. The TAI luminescence excitation spectra in the long-wavelength region in fact coincide with their absorption spectra. This means that fluorescence belongs to the molecules under study. Typical fluorescence spectra are shown in Figs. 1 and 2; fluorescence band

Table 1
Spectral parameters of TAI in different solvents

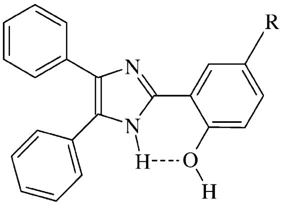
TAI	Parameter	Toluene	Dioxane	Ethanol	
				300 K	77 K
I	λ_{exc} (nm)	318	325	312	325
	λ_{lum} (nm)	382	380	377	373
	ΔE (cm^{-1})	5268	4453	5530	3960
IIa	λ_{exc} (nm)	335	330	327	333
	λ_{lum} (nm)	460	455	438	435
	ΔE (cm^{-1})	8110	8320	7750	7040
IIb	λ_{exc} (nm)	342	342	342	346
	λ_{lum} (nm)	465	460	445	440
	ΔE (cm^{-1})	7735	7670	6770	6175
IIc	λ_{exc} (nm)	320	320	320	–
	λ_{lum} (nm)	470	470	448	–
	ΔE (cm^{-1})	9970	9970	8925	–
III	λ_{exc} (nm)	340	340	330	335
	λ_{lum} (nm)	415	420	425	380
	ΔE (cm^{-1})	5315	5600	6710	3535
IV	λ_{exc} (nm)	365	360	353	350
	λ_{lum} (nm)	460	460	438	378
	ΔE (cm^{-1})	5660	6040	5500	2115

maximum λ_{fl}, fluorescence excitation maximum λ_{exc} and Stokes shifts ΔE are presented in Table 1 for TAI **I**–**IV**.

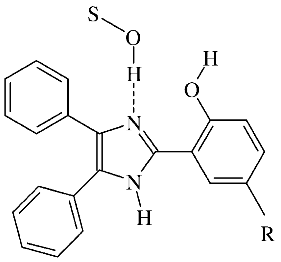
It may be suggested that in aprotic solvents the molecule of hydroxy-substituted TAI exists as two different intramolecular hydrogen-bonded isomers $\mathbf{R}_1$ and $\mathbf{R}_2$. Excitation of the first isomer $\mathbf{R}_1$ should lead to the formation of the keto-isomer **K** due to ESIPT, while excitation of the second isomer $\boldsymbol{R}_2$ must yield the normal emission. But, we found out that the fluorescence spectra of **IIa**–**IIc** in toluene and dioxane contain only an abnormal Stokes-shifted emission band. This means that only the $\mathbf{R}_1$ isomer of these molecules is stable under these conditions.



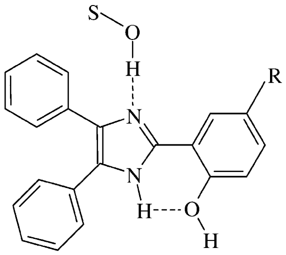
$\mathbf{R}_1$



$\mathbf{R}_2$



$\mathbf{R}_3$



$\mathbf{R}_4$

However, in the case of hydroxyl-containing solvent (EtOH), a short-wavelength emission band appears for **IIa**–**IIc**, which is absent in the fluorescence spectrum of **I** (Fig. 1b). This result corresponds to the data obtained earlier [17,18] for compounds demonstrating ESIPT, and can be explained by the presence of intermolecular hydrogen bonding with solvent molecules leading to the stabilization of solvated isomers $\mathbf{R}_3$ and/or $\mathbf{R}_4$ in which ESIPT is impossible.

The Stokes shift decreases as the temperature is lowered to 77 K, but it remains large enough for **IIa**–**IIc** compared to ΔE for **I** (without the OH group), which suggests that ESIPT occurs at this temperature as well.

The non-planar structures of naphthyl derivatives **III** and **IV** caused by the hindrance between imidazole and naphthyl (in eighth position) hydrogen atoms are significantly different from the planar structures of **I** and **IIa**–**IIc**. The molecular geometry optimization gives the values of 40 and 30° for the dihedral angle between imidazolyl and naphthyl planes for $\mathbf{R}_1$ isomer of **III** and **IV**, respectively. Such a molecular structure influences significantly spectral luminescent properties of molecules **III** and **IV**. As a result, the luminescence spectra of **IV** in toluene and dioxane (Fig. 2a) contain two bands: a short-wavelength band and a band with

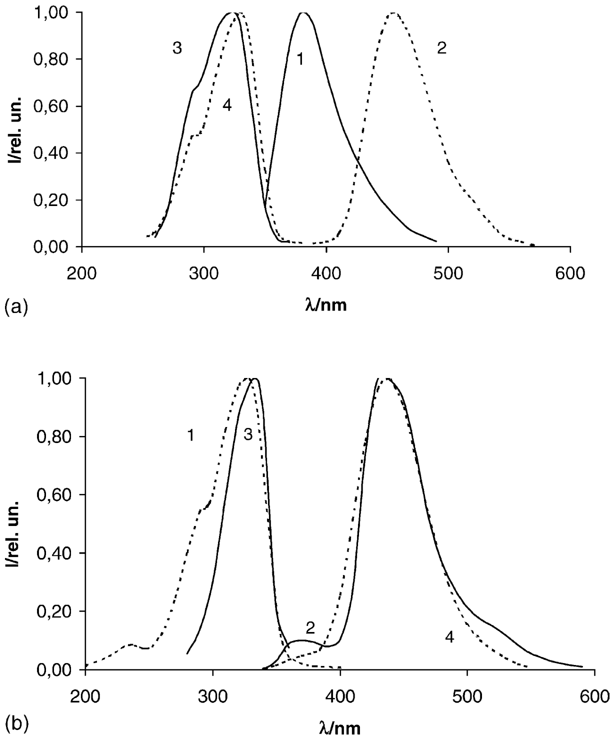


Fig. 1. (a) Fluorescence spectra (1 and 2) and fluorescence excitation spectra (3 and 4) of TAI **I** (1 and 3) and **IIa** (2 and 4) in dioxane at 293 K. (b) Fluorescence spectra (2 and 4) and fluorescence excitation spectra (1 and 3) of TAI **IIa** in EtOH at 293 K (1 and 4) and 77 K (2 and 3).

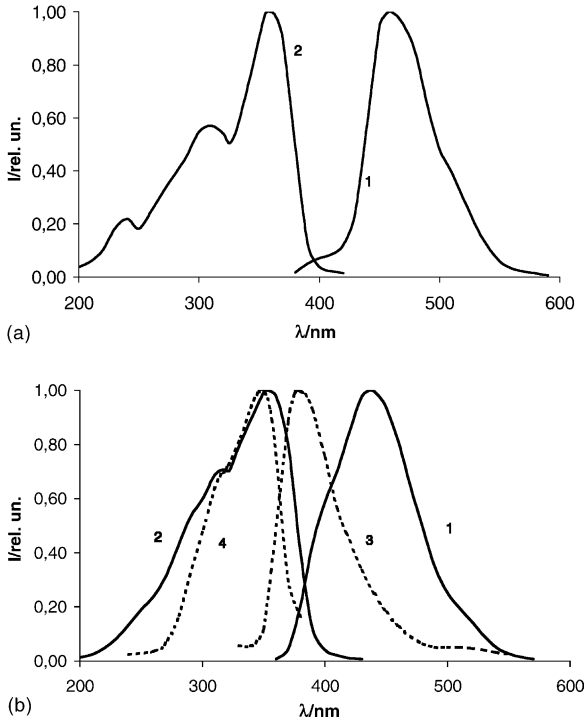


Fig. 2. (a) Fluorescence spectrum (1) and fluorescence excitation spectrum (2) of TAI **IV** in dioxane at 293 K. (b) Fluorescence spectra (1 and 3) and fluorescence excitation spectra (2 and 4) of TAI **IV** in EtOH at 293 K (1 and 2) and 77 K (3 and 4).

a large Stokes shift, which indicates the presence of both $\mathbf{R}_1$ and $\mathbf{R}_2$ isomers in the media without hydroxyl groups. We can assume that the intramolecular hydrogen bond in the $\mathbf{R}_1$ isomer of **IV** is weakened because of non-planar structure leading to the stabilization of the $\mathbf{R}_2$ isomer.

A mixture of two isomers also presents in the solution of TAI **IV** in EtOH at room temperature. But, after cooling this solution to 77 K, the situation changes—the shape of the spectrum and a significantly decreased value of ΔE (2115 cm^{-1}) suggest the presence of only one isomer of **IV** under these conditions—when ESIPT does not occur. We believe that for ESIPT to occur in **IV**, the 2-hydroxynaphthyl fragment of the molecule should turn at some angle to reach the planarity of this fragment with imidazole cycle. Naturally, such a rotation is impossible in glassy matrix at 77 K.

For better understanding the ESIPT mechanism in TAI, we performed quantum–chemical calculations of electron density for the **K** and **E** isomers of the TAI **II** molecule in the

Table 2
Electronic density of atoms N(3) and O for TAI **IIa**

Atom	Form		
	E	**E***	**K**
N(3)	−0.147	−0.172	+0.233
O	−0.264	−0.236	−0.489

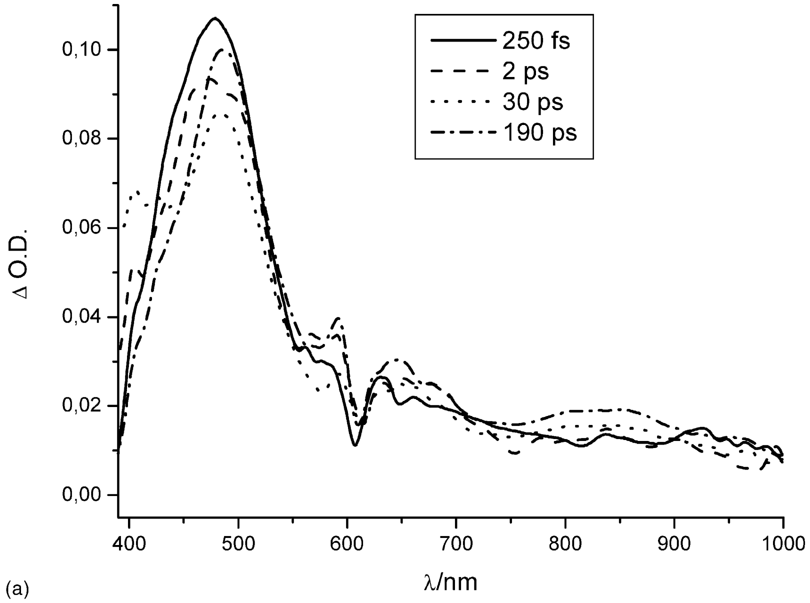


Fig. 3. Dynamics of photoinduced absorption spectra of TAI **I** (a) and **III** (b) in acetonitrile.

ground and the first excited states. The results are presented in Table 2. One can see that excitation of **E** molecule leads to the increase in the electron density at the N(3) atom and its decrease at the O atom resulting in ESIPT and formation of the **K*** isomer in excited state. Then, the **K*** isomer emits luminescence and returns to the ground state **K**, which is characterized by a large positive charge at the N(3) atom and negative charge at the O atom. As a result, a reverse process occurs in the ground state of the molecule, producing an initial molecule in the form **E**.

3.2. Femtosecond spectroscopy

To confirm our proposals discussed in the previous section, we examined the dynamics of photoinduced absorption spectra upon excitation by a femtosecond pulse at 308 nm. First of all, compounds **I** and **III** without ESIPT have been studied. Fig. 3 shows that their spectra change with time similarly. At the first stage, in the period of time up to 250 fs, a wide absorption band with the maxima at 480 nm for **I** and 500 nm for **III** is developed. Then, in the time interval from 250 fs to 1 ps, some spectral broadening is observed and the spectrum remains unchanged up to 600 ps.

According to the steady-state absorption spectra of **I** and **III**, excitation of these molecules by light at 308 nm transfers them into $\mathbf{S}_2$ state or into $\mathbf{S}_1^{\mathrm{v}}$ excited vibration state. In any case, a photoinduced band at 480 nm for **I** (and 520 nm for **III**) should be assigned to the absorption from the first singlet excited electronic state $\mathbf{S}_1$ (the $\mathbf{S}_1 \rightarrow \mathbf{S}_n$ transition) of the initial molecule. Therefore, the characteristic time of the

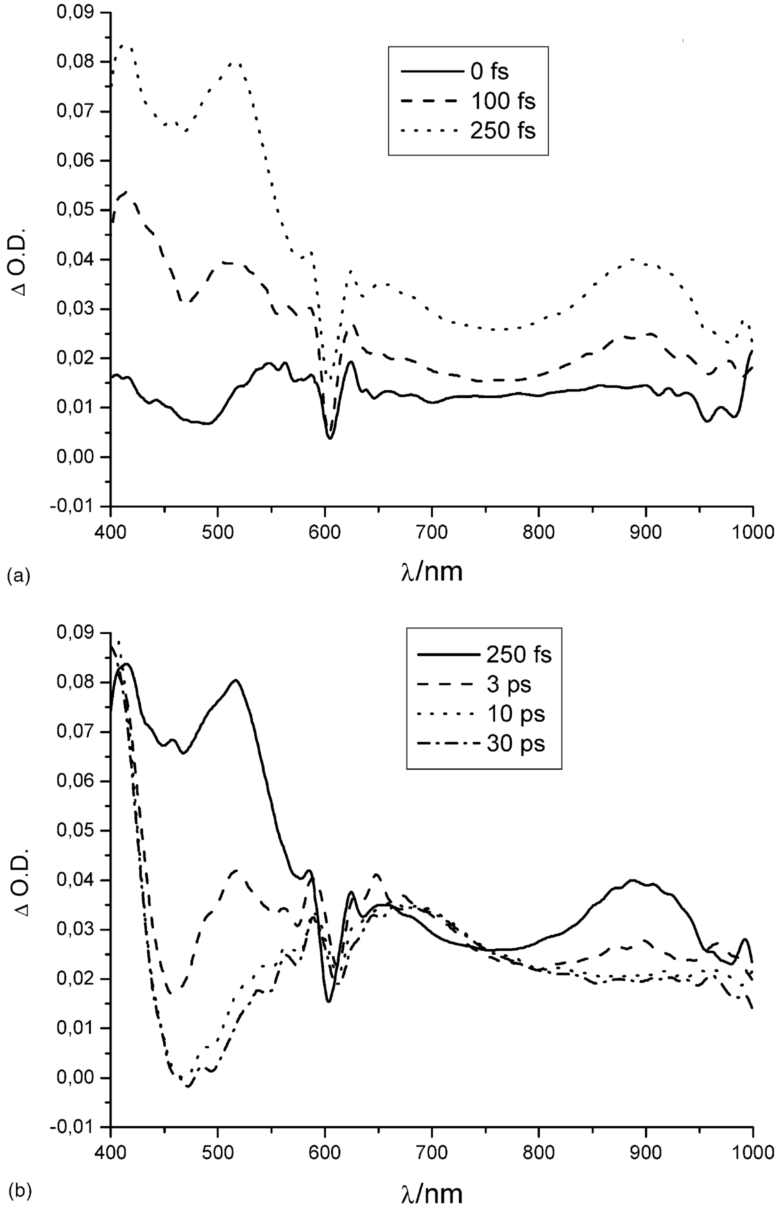


Fig. 4. Dynamics of photoinduced absorption spectra of TAI **IIa** in acetonitrile for the delay times in the intervals: 0–250 fs (a) and 250 fs–30 ps (b).

appearance of this band (100 fs for both the compounds) represents the time of internal conversion and/or vibration relaxation to the $\mathbf{S_1}$ state ($\mathbf{S_2} \to \mathbf{S_1}$ or $\mathbf{S_1^v} \to \mathbf{S_1}$).

The introduction of a hydroxyl group into position 2 of an aryl substituent, qualitatively changes the dynamics of photoinduced absorption spectra due to ESIPT. The character of changes is the same for **IIa**–**IIc** compounds and different for TAI **IV**.

Fig. 4 shows the dynamics of photoinduced absorption spectra for **IIa**. Three absorption bands at 415, 520 and 895 nm increase simultaneously at short times. Then, up to 10 ps, two long-wavelength bands virtually disappear during the characteristic time ∼3.2 ps, whereas the intensity of the short-wavelength band remains unchanged (a small decrease in the optical density is caused by relaxation process). The kinetic curves for **IIa** at these wavelengths are shown in Fig. 6a. Their analysis demonstrates full coincidence of the kinetics at 520 and 850 nm bands, which suggests that these bands belong to one specie. Therefore, one can observe two intermediate products in the photoinduced absorption spectra of **IIa**.

The long-lived (at the femtosecond timescale) species being characterized by the absorption maximum at 415 nm is evidently keto-form $\mathbf{K}^*$ in the first electronic excited $\mathbf{S_1}$ state formed due to ESIPT. We observe this intermediate as a stable one, because it will emit fluorescence only during several nanoseconds, as almost all complex organic molecules do. Therefore, we can evaluate the ESIPT time by the increase in the absorption band at 415 nm. For **IIa**–**IIc**, this time is 100 fs. It should be noted that this value is approximate because it is unknown in which state of $\mathbf{E}^*$ form ($\mathbf{S_2}$(**E**), $\mathbf{S_1^v}$(**S**) or $\mathbf{S_1}$(**E**)) ESIPT occurs. This result is in good agreement with the data in the literature on the ESIPT time for other molecules with a planar reaction center [19–21].

As for the short-lived species with absorption bands at 520 and 895 nm:

(i) it cannot be the $\mathbf{E}^*$ isomer in the $\mathbf{S_1}$(**E**) state because it should undergo the transition into the $\mathbf{S_1}$(**K**) state due to ESIPT for 100 fs;
(ii) it is unlikely a molecule of the $\mathbf{E}^*$ isomer in the excited triplet state $\mathbf{T_1}$(**E**) formed as a result of intersystem crossing because of ultra short time of ESIPT in the $\mathbf{S_1}$(**E**) state;
(iii) it cannot be a molecule of the $\mathbf{K}^*$ isomer in the excited triplet state $\mathbf{T_1}$(**K**) because its formation must be accompanied by a decrease in the intensity of the 415 nm absorption band due to the $\mathbf{S_1}$(**K**) → $\mathbf{T_1}$(**K**) process.

It is reasonable to suppose that it is a molecule of the $\mathbf{E}^*$ isomer in such an excited electronic state, in which ESIPT is in principle impossible. It may be a singlet excited state of the $n\pi^*$ type. The data [22–26] on ESIPT in [2,2′-bipyridile]-3,3′-diole ($Bp(OH)_2$)—a molecule with a reaction center that is similar to that of TAI—support our conclusion. In $Bp(OH)_2$, the $\mathbf{S_2}(n\pi^*)$ level is located closely to the $\mathbf{S_1}(\pi\pi^*)$ level, from which ESIPT occurs. Both these levels are populated in competitive manner during vibration relaxation from highly located levels of the $\mathbf{E}^*$ isomer. In future, the $\mathbf{S_2}(n\pi^*)$ level is non-radiatively deactivated to the $\mathbf{S_0}$ ground state, and the lifetime of the $\mathbf{S_2}(n\pi^*)$ state is several picoseconds.

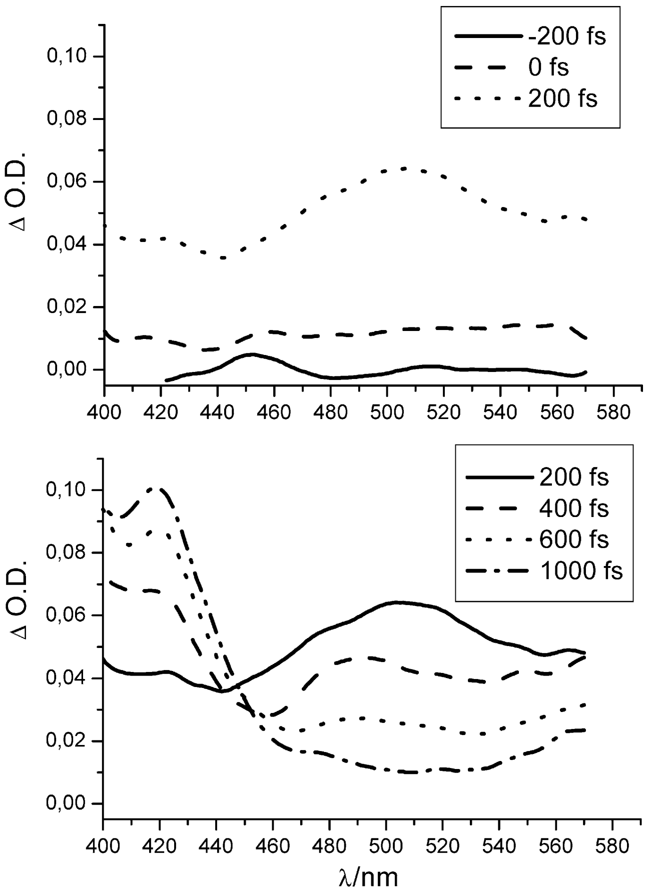
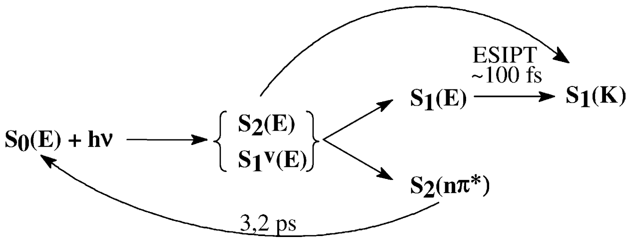


Fig. 5. Dynamics of photoinduced absorption spectra of TAI **IV** in acetonitrile for different delay times.

Therefore, we conclude, that a short-lived species in the molecule **IIa** represents a molecule of $\mathbf{E}^*$ isomer in the $\mathbf{S_2}(n\pi^*)$ state.

Thus, the following scheme of processes taking place after excitation of the **E** isomer of the **IIa** molecule by light at 308 nm can be presented Scheme 1.

The results obtained for **IIb** and **IIc** confirm this scheme. Some differences are only in the position of maxima and lifetimes of the $\mathbf{S_2}(n\pi^*)$ state (see Table 3).



Scheme 1.

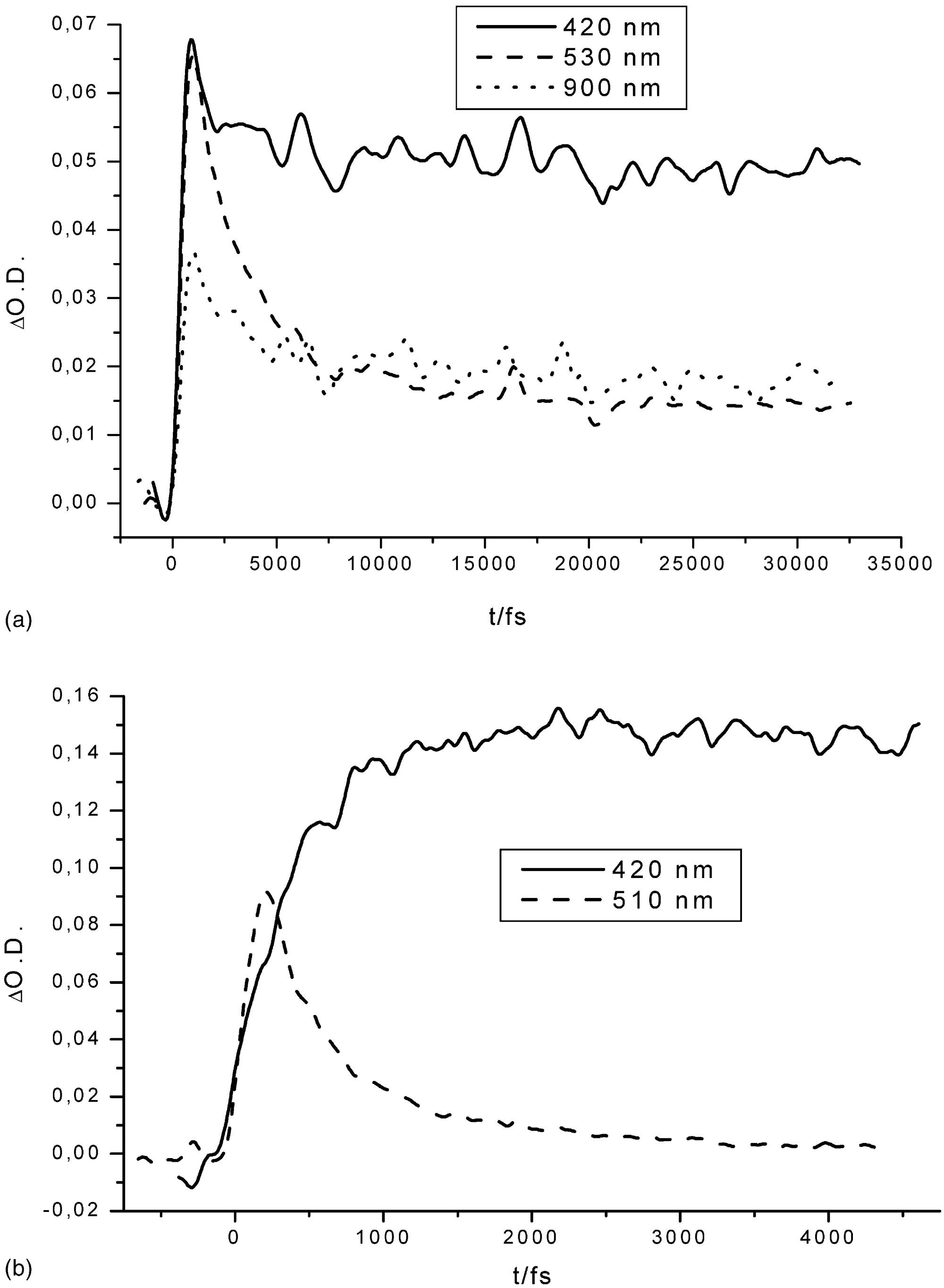


Fig. 6. Dependence of the optical density on time for solutions of TAI **IIa** (a) and **IV** (b) in acetonitrile at different detection wavelengths.

Table 3
Data of femtosecond study of **IIa**–**IIc** in acetonitrile

TAI	Transition $S_2(n\pi^*) \rightarrow S_n(\mathbf{E})$ (nm)	Transition $S_1(\mathbf{K}) \rightarrow S_n(\mathbf{K})$ (nm)	Relaxation time $S_2(n\pi^*) \rightarrow S_0(\mathbf{E})$ (fs)
IIa	520, 895	415	3200
IIb	500, 930	420	1055
IIc	525, 905	∼390	1520

All these conclusions are true for planar OH-containing TAI under study. Dynamics of photoinduced absorption of non-planar **IV** differs significantly from that for **IIa**, as one can see from Fig. 5. First, only one absorption band at 510 nm appears during a time interval of 0–200 fs. Then, this band disappears during 350 fs simultaneously with the appearance of a new absorption band at 420 nm. The latter band remains unchanged up to 600 ps. Fig. 6b shows kinetic curves at 420 and 510 nm. Analogously, a short-wavelength absorption band can be assign to the long-lived fluorescent

$$\mathbf{S_0(E)} + \mathbf{h\nu} \longrightarrow \left\{ \begin{matrix} \mathbf{S_2(E)} \\ \mathbf{S_1{}^{v}(E)} \end{matrix} \right\} \longrightarrow \mathbf{S_1(E)} \xrightarrow{\substack{\text{Rotation} \\ \text{350 fs}}} \mathbf{S_1{}^{r}(E)} \xrightarrow{\substack{\text{ESIPT} \\ \sim\text{100 fs}}} \mathbf{S_1(K)}$$

Scheme 2.

state of keto-isomer $\mathbf{S}_1(\mathbf{K})$. But contrary to the case of **IIa**, here the appearance of the molecule in this state occurs simultaneously with the decrease of the absorption band at 510 nm, which allows us to attribute this band to the molecule in the $\mathbf{S}_1(\mathbf{E})$ state and synchronous changes of the optical density in the absorption bands at 420 and 510 nm to the ESIPT. Consequently, in **IV**, ESIPT occurs 3.5 times slower than in **IIa**, due to the necessity for some rotation of 2-hydroxynaphthyl fragment in the excited state of the $\mathbf{E}^*$ isomer.

Second, Figs. 5 and 6b show that the $\mathbf{S}_1(\mathbf{K})$ state is formed by only one way through the $\mathbf{S}_1(\mathbf{E}) \rightarrow \mathbf{S}_1(\mathbf{K})$ transition. It means that ESIPT does not occur from the $\mathbf{S}_2(\mathbf{E})$ and $\mathbf{S}_1^{\mathrm{v}}(\mathbf{E})$ states in **IV**. The fact is that the lifetime of these states in **IV** is shorter than the time needed for conformational rearrangement of the molecule for ESIPT (∼350 fs).

Third, the $\mathbf{S}_2(n\pi^*)$ state in **IV** is not revealed. In can be explained by the significant changes in the location of the excited electronic levels in **IV** compared to **IIa** because of the annellation of benzene ring.

To sum up, we can represent the Scheme 2 of the process occurring in **IV** upon excitation by light at 308 nm. $\mathbf{S}_1^{\mathrm{r}}(\mathbf{E})$ means the molecule in the $\mathbf{S}_1(\mathbf{E})$ state after a rotation of molecular fragments.

4. Conclusions

We have established that ESIPT and time of this process in hydroxy-substituted TAIs depend on the structure of a molecular reaction center. In TAI molecules having planar reaction center, ESIPT occurs very rapidly both in solutions and glassy matrixes. For ESIPT to occur in molecules with a non-planar reaction center, molecular fragments should rotate, which increases the ESIPT time in solutions and makes ESIPT impossible in a glassy state.

Acknowledgements

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