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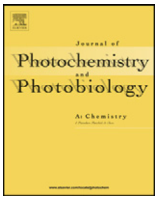

Charge transfer fluorescence of *trans*-stryrylpyridinium iodides

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ABSTRACT

The photoprocesses of *trans*-1-methyl-4-[4-R-styryl]pyridinium iodide (R = H, P1) and derivatives with a cyano, a nitro and a methoxy group at the phenyl moiety, P2–P4, respectively, were studied in solution. In solvents of relatively low polarity, e.g. tetrahydrofuran, where contact ion pairs are present, the fluorescence spectrum of the styrylpyridinium is significantly red-shifted and the quantum yield is strongly enhanced. These findings are due to photoinduced electron transfer from I^- to the excited singlet state of the cation. The features of complementary *trans*-styrylquinolinium iodides are in good accordance.

1. Introduction

The photophysical and photochemical properties of quaternary azastilbenes (also denoted as stilbazolium salts) in solution are the subject of various studies [1–9]. Several iodides of the structural type *trans*-1-methyl-4-[4-R-styryl]pyridinium (^+A_t-R) with R: H, cyano, nitro, methoxy (P1, P2, P3, P4, respectively) have been investigated [4–7]. Quaternary dialkylaminoazastilbenes, such as *trans*-1-methyl-4-[4-dimethylaminostyryl]pyridinium iodide (P-NMe_2), have frequently been applied as fluorescence sensors [10–18]. UV irradiation of the *trans*-isomer of parent P1 yields the *cis* isomer, but this does not take place for P-NMe_2 [9]. The quantum yield (Φ_c) of *trans* → *cis* isomerization of the iodides P1–P4 as well as the perchlorates (P′1–P′4) in a polar solvent, such as acetonitrile or water, is $\Phi_c = 0.4–0.5$ [4–7]. A similar mechanism has been reported for styrylquinolinium salts [4–9].

Other photochemical studies deal with related betaines, e.g. *trans*-1-methyl-4-[4-R-styryl]pyridinium, R = OH, for which, in aqueous solution, an equilibrium between the hydroxide and the deprotonated forms with $pK_a = 8.5$ has to be considered [19–21]. Characteristic spectroscopic properties of styrylaromatic salts have been reviewed [1,2]. Photodimerization is another photoprocess of quaternary azastilbenes [22]. The non-linear optical properties of styrylpyridinium ions are the subject of various studies [23,24]. The properties of photochromic molecules and materials and the ion-pair charge transfer (CT) complexes have been summarized [24]. Photoinduced electron transfer can play a decisive role when the anion is iodide, in contrast to perchlorates or other innocent anions [2,5]. The spectroscopy of specific pyridinium iodides is of special interest, because these salts exhibit an intermolecular CT absorption band [25–30]. The CT interaction is also the subject of various studies concerning salts with aromatic cations [31–33]. The intermolecular CT fluorescence is a rare phenomenon in the literature [34].

Here, the photophysical CT properties of iodides of *trans*-1-methyl-4-[4-R-styryl]pyridinium with R: H, CN, NO_2 and OMe, P1–P4, respectively, and *trans*-1-methyl-4-[4-R-styryl] quinolinium iodides with R: H, NO_2 and OMe (Q1, Q3, Q4) were studied. The fluorescence maxima of most iodides in solvents of low polarity are strongly red-shifted, especially for P3 and Q3. The corresponding perchlorates (P′1–P′4) and (Q′1, Q′3, Q′4) show no CT effects and were used for the purpose of comparison, Chart 1.

2. Experimental

The salts were the same as used previously [3–9]. The molar absorption coefficients in methanol solution are $\varepsilon_{348} = 2.5 \times 10^4$ and $\varepsilon_{370} = 2.4 \times 10^4\ M^{-1}\ cm^{-1}$ for P1 and P4, respectively [7]. The solvents (Aldrich, Merck) were of the purest spectroscopic quality, methyltetrahydrofuran (MTHF); dioxane, tetrahydrofuran (THF), dichloromethane, dimethyl sulfoxide (DMSO) and acetonitrile were Uvasol quality. The absorption spectra were monitored on a diode array spectrophotometer (HP, 8453). The iodides were dissolved in THF either by using a DMSO stock solution (<1%) or with ultrasound. In the former case, the absorption spectra change

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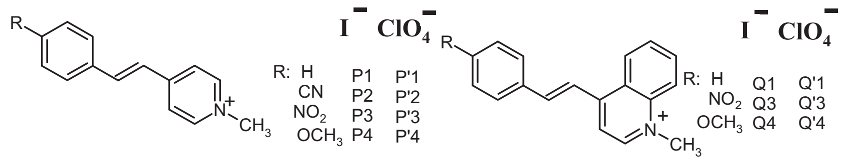


Chart 1.

Table 1

Maximum of *trans* isomer, isosbestic point, relative CT absorption and quantum yield of *trans* → *cis* isomerization.[a]

Comp.	Solvent	λ_t (nm)	λ_i (nm)	A_{450}/A_{max}	Φ_c[b]
P′1	THF	344	305	<0.001	0.5
P1	THF	344	310	0.10	0.03
	Dichloromethane	357	302	0.002	0.012
	Acetonitrile	343	290	<0.001	0.5
P′2	THF	344	310	<0.001	0.5
P2	THF	344	310	0.09	0.03
P3	THF	343	303	0.10	0.02
	Dichloromethane	345	310	0.002	0.07
	Acetonitrile	338	300	<0.001	0.4
P4	THF	360		0.09	0.4
Q1	THF	375		0.10	0.4
Q3	THF	374	313	0.09	0.005
	Acetonitrile	372	314	<0.001	0.4

[a] In air-saturated solution.
[b] Under argon using λ_{irr} = 366 nm.

with time (in the 1–200 min. range) in the manner that the CT absorption bands (A_{CT}) at 280–300 and ca. 450 nm became maximum. On the other hand, a too long storage (>1 d) was found to decrease both A_{CT} and the fluorescence intensity markedly. A spectrofluorimeter (Varian Cary, eclipse) was employed to measure the fluorescence spectra. The quantum yield of fluorescence was obtained using 9,10-diphenylanthracene in air-saturated cyclohexane ($\Phi_f = 0.6$) and rhodamine 101 in ethanol ($\Phi_f = 0.9$) as references. The quantum yield Φ_c of *trans* → *cis* isomerization was obtained using P1 in argon-saturated acetonitrile as reference, $\Phi_c = 0.5$ [7]. The ratio of absorption coefficients $\varepsilon_t/\varepsilon_c$ and the quantum yields of *cis* → *trans* and *trans* → *cis* photoisomerization are:

$$\left(\frac{cis}{trans}\right)_0 = \frac{\varepsilon_t \times \Phi_c}{\varepsilon_c \times \Phi_t} \tag{I}$$

Electrochemical measurements were performed in an air-tight cell under an argon atmosphere in anhydrous acetonitrile with Bu_4NPF_6 as the supporting electrolyte, using a potentiostat (EG&G, 273A) with a 0.01 N $AgNO_3/Ag$ in acetonitrile as reference electrode. Glassy carbon disk electrodes served as working and counter electrodes. The potentials are given as peak potentials of square wave voltammograms vs. ferrocene/ferrocenium values: $-E^{\circ\prime}$ = 1.34, 1.19, 1.14 and 1.40 V for P1–P4, respectively. The measurements refer to 24 °C.

3. Results

3.1. Absorption

The absorption spectra of *trans*-styrylpyridinium salts in polar solvents, e.g. acetonitrile or ethanol, have maxima (λ_t) at 340–380 nm. The peak at λ_t = 344 nm is the same for either P′1 or P1, but the iodide absorbs weekly at 450 nm, in contrast to the perchlorate. An example of the thermal absorption changes in THF is shown in the inset of Fig. 1. The absorption at 280 and 450 nm increases with time and at λ_t decreases. These changes become minor after a few h. As a measure of this CT absorption, the relative contribution A_{450}/A_{max} is given in Table 1; this ratio is 0.002–0.1 in THF and much smaller in dichloromethane. An example of the concentration effects is shown in the inset of Fig. 2, indicating the absence of aggregation.

Photolysis of a *trans* isomer leads to the *cis* isomer and eventually a photostationary state, the latter of which results from a further decay pathway, i.e. of the singlet-excited *cis* isomer leading to the *trans* and *cis* isomers. For a given *trans*-styrylpyridinium iodide Φ_c is substantial in acetonitrile but smaller in dichloromethane and very small in THF. The time dependence of A_{350} indicates the efficient *trans* → *cis* photoisomerization of the perchlorate in contrast to the iodide (Fig. 3, inset). Examples of the absorption changes vs. time of irradiation at 366 nm are shown in Fig. 3. The absorption spectra of P′1 in THF upon irradiation at 366 nm show an isosbestic point at λ_i = 300 nm.

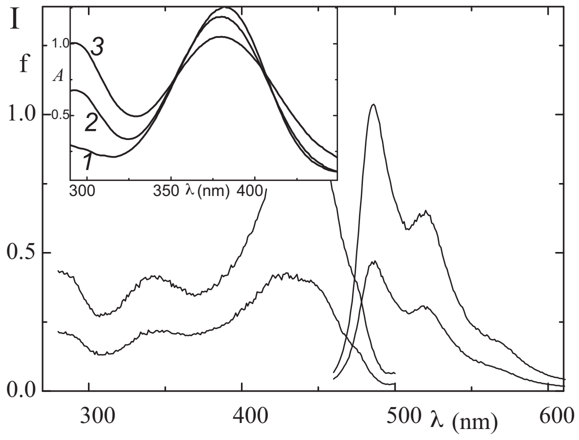


Fig. 1. Fluorescence emission (λ_{ex} = 450 nm) and excitation (λ_f = 530 nm) spectra in THF at 10 and 100 min after mixing of 0.5% P4 in DMSO; inset: absorption spectra at 1, 10 and 100 min (1–3), respectively.

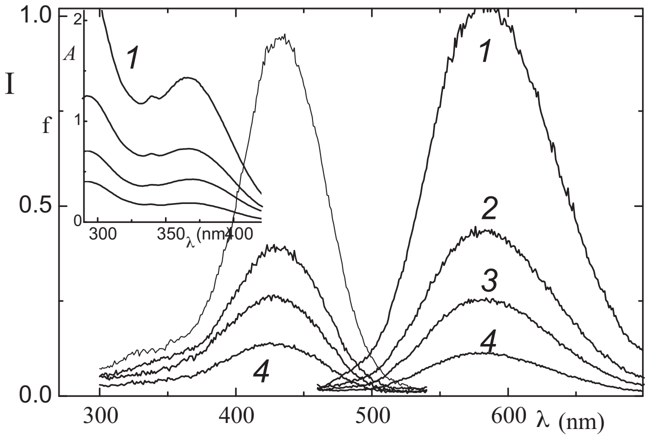


Fig. 2. Fluorescence emission (λ_{ex} = 450 nm) and excitation (λ_f = 600 nm) spectra of Q3 in THF-DMSO (100:1) upon each 50% dilution by THF (1–4, respectively); inset: corresponding absorption spectra.

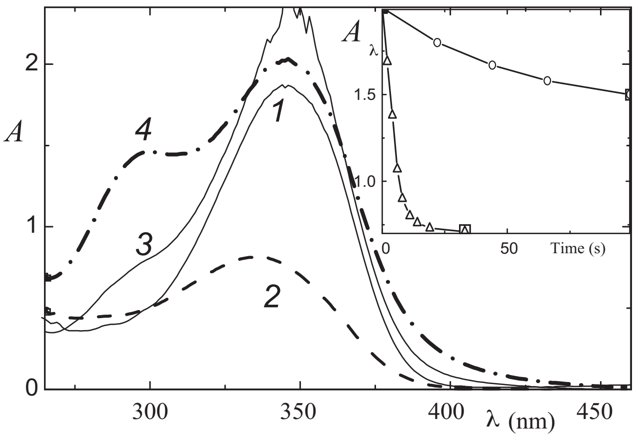


Fig. 3. Absorption spectra of P′1 in argon-saturated THF at 0 (1) and 20 s irradiation at 366 nm (2) and P1 in THF at 0 and 200 s (3 and 4); inset: time dependences at 350 nm of P′1 (△) and P1 (o).

3.2. Fluorescence

The fluorescence of *trans*-styrylpyridinium iodides P1–P3 in acetonitrile has a maximum (λ_f) at 420–440 nm (Table 2). The fluorescence excitation spectrum with peaks at λ_{ex} = 340–360 nm is similar to the absorption spectrum. The quantum yield Φ_f of any styrylpyridinium iodide is very low in THF, dichloromethane or acetonitrile, if excited at λ_t = 340–360 nm. However, upon excitation of P1–P4 in THF at 450 nm, a new fluorescence emission spectrum appears which has a red-shifted maximum (λ_f^{CT}). This peak is 490 nm for P1/Q1 and 590 nm for P3/Q3. The fluorescence excitation spectrum has likewise a red-shifted maximum (λ_{ex}^{CT}) with respect to λ_t. The λ_f^{CT} and λ_{ex}^{CT} values are compiled in Tables 3 and 4 together with Φ_f^{CT} which in THF is 0.08–0.1. Examples of the fluorescence emission and excitation spectra are shown in Figs. 1 and 4. For Q3 in THF it can be seen that λ_{ex}^{CT} is independent of the dye concentration using a ca. 10-fold variation up to 50 μM (Fig. 2). The styrylpyridinium iodides exhibit a one-electron reduction at potentials influenced by the substituent. Fig. 5 shows a plot of $1/\lambda_f^{CT}$ of P1–P4 in THF, corresponding to $\lambda_f^{CT} = 485$–590 nm, as a function of $-E^{\circ\prime}$, indicating a linear relationship of the excited singlet state CT level on the anionic peak potential.

Table 2
Fluorescence maximum and quantum yield.[a]

Comp.	Solvent	λ_{ex} (nm)	λ_f (nm)	Φ_f[b]
P′1	THF	345	424	0.0006
	Dichloromethane	358	425	0.0006
	Acetonitrile	345	428	0.001
P′3	THF	345	440	0.005
	Acetonitrile	340	440	0.005
P′4	THF	364	503	0.004

[a] In air-saturated solution.
[b] λ_{exc} = 350 nm.

Table 3
CT fluorescence emission and excitation maximum and quantum yield.[a]

Solvent	E_T^N	P1: λ_f^{CT} (nm)	Φ_f^{CT}	P3: λ_f^{CT} (nm)	Φ_f^{CT}
Dioxane	0.16			590	
Trichloroethylene	0.16			590	
MTHF	0.18			590	0.08
THF	0.21	485	0.08	590	0.1
Chloroform	0.26	485	0.003	590	<0.01
Dichloromethane	0.31	485	<0.001	590	<0.001

[a] In air-saturated solution using λ_{exc} = 450 nm, λ_f = 530 nm, 600 nm for P3/Q3.

Table 4
CT fluorescence emission and excitation maxima and quantum yield in THF.[a]

Comp.	λ_{ex}^{CT} (nm)	λ_f^{CT} (nm)	Φ_f^{CT}
P1	440	485, 520sh	0.10
P2	440	510, 540sh	0.09
P3	430	590	0.1
P4	450	490, 525sh	0.08
Q1	450	490, 520sh	0.1
Q3	440	590	0.08

[a] In air-saturated solution using λ_{exc} = 450 nm, λ_f = 530 nm, 600 nm for P3/Q3.

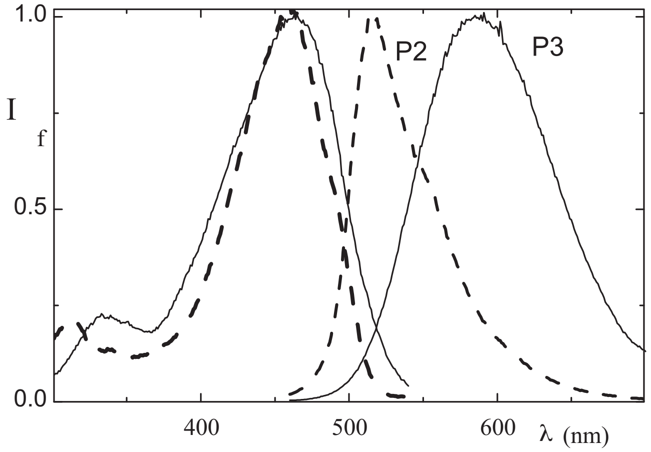


Fig. 4. Fluorescence emission (λ_{ex} = 450 nm) and excitation (λ_f = 530 nm) spectra of P2 and P3 in THF.

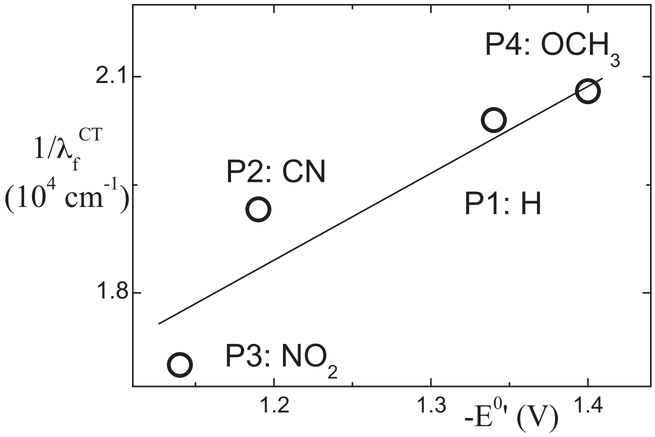


Fig. 5. Plot of the reciprocal fluorescence maximum of the styrylpyridinium iodides in THF (λ_{ex} = 450 nm) as a function of the anionic peak potential.

4. Discussion

In polar solvents $^{+}A_t$ and X^- are free ions [3]. In solvents of low polarity the anion is in close vicinity of the cation and via solvent separated ion pairs ($^{+}A_t/I^-$) contact ion pairs ($^{+}A_t\,X^-$) are formed, see Scheme 1. To characterize the solvent polarity, the emperical parameter E_T^N, based on a pyridinium *N*-phenolate betaine, has frequently been used [35]. The ground state properties of stilbazolium salts are determined by the solvent polarity in the manner that the ions are free for $E_T^N > 0.4$ and present as contact pairs for $0.15 < E_T^N < 0.3$ [3].

4.1. Photoprocesses of ions

The absorption spectra of various *trans*-stilbazolium salts in polar solvents have maxima at λ_t = 340–375 nm which are indepentent of the type of anion [2,3]. The ground state of a cation is characterized by an activation barrier between the *trans* and *cis* geometries. The thermal *cis* → *trans* back-reaction is accessible at room temperature and sensitive to structure and solvent polarity [3]. The quantum yields Φ_t and Φ_c of isomerization in acetonitrile are substantial, based on values for P1–P4. The triplet pathway for *trans* → *cis* photoisomerization, due to a high quantum yield of intersystem crossing (Φ_{isc}), is a unique feature of arylethylenes which are substituted by a nitro group [2,4–6]. Excitation of P3 in a polar solvent, e.g. ethanol or acetonitrile, leads from the excited singlet ($^{1*+}\mathrm{A_t}$) via the triplet state ($^{3*+}\mathrm{A_t}$) after further intersystem crossing into both isomers [4–7]. In contrast to P3, excitation of other cations not containing a nitro group leads from $^{1*+}\mathrm{A_t}$ into the *cis*–*trans* mixture via singlet states [7]. A conceivable mechanism is constituted by reactions (1′)–(6′).

$$^{+}\mathrm{A_t} \quad h\nu \rightarrow {}^{1*+}\mathrm{A_t} \tag{1′}$$

$$^{1*+}\mathrm{A_t} \quad h\nu_f \rightarrow {}^{+}\mathrm{A_t} \tag{2′}$$

$$^{1*+}\mathrm{A_t} \quad \rightarrow {}^{3*+}\mathrm{A_t} \tag{3′}$$

$$^{1*+}\mathrm{A_t}(\text{or } {}^{3*+}\mathrm{A_t}) \rightarrow {}^{+}\mathrm{A_p} \tag{4′5′}$$

$$^{+}\mathrm{A_p} \rightarrow \alpha^{+}\mathrm{A_c} + (1-\alpha)^{+}\mathrm{A_t} \tag{6′}$$

$$^{1*+}\mathrm{A_t}\,(\text{or } {}^{3*+}\mathrm{A_t}) + \mathrm{D} \rightarrow \mathrm{A}^{\bullet} + \mathrm{D}^{\bullet+} \tag{7′}$$

If the donor is iodide, a radical could be intermediate to the reduced photoproduct. The electron transfer reaction (7′) from iodide or amines to the excited singlet and triplet states of *trans*-4-R-styrylpyridinium and corrresponding styrylquinolinium salts in polar solvents have been presented earlier [5–8]. The triplet state can be quenched by oxygen and/or is the observed key intermediate for electron transfer from an appropriate donor, when oxygen is removed. To account for the latter, reaction (7′) is proposed. Generally, the radical pathway competes with *trans* → *cis* photoisomerization. The potential energy surface of any styrylpyridinium cation should have a minimum at the perpendicular geometry of the excited singlet state, from where the radiationless transition to the perpendicular configuration of the ground state ($^{+}\mathrm{A_p}$) takes place. Linear correlations have been reported between solvent polarity and adiabatic and diabatic transition energies for the thermal *cis* → *trans* isomerization of solvatochromic hydroxy-styrylpyridinium type merocyanines [19–21]. A relationship between the rate of thermal *cis* → *trans* isomerization and the solvent polarity is also known for related systems [3].

The major deactivation of the *trans* isomer of the singlet-excited cation $^{1*+}\mathrm{A_t}$ is *trans* → *cis* photoisomerization and fluorescence plays a minor role. At low temperatures, however, the contribution of the two pathways interchange to low Φ_c and large Φ_f [4–8]. This is a consequence of inhibited twisting into the perpendicular excited singlet state in rigid media. The deactivation pathway of $^{1*+}\mathrm{A_t}$ at room temperature, besides fluorescence to the *trans* isomer, is internal conversion to the $^{+}\mathrm{A_p}$ transition state, either directly (P1, P2, P4) or after intersystem crossing (P3). Here, α is the probability to react into the *cis*-isomer $^{+}\mathrm{A_c}$. The dimethylamino derivative P-NMe$_2$ does not follow this pattern since the excited singlet state is too low in energy to overcome the barrier in the excited singlet state, required for a path to the *cis*-isomer. Nevertheless, the major deactivation is an activated internal conversion step at the *trans* geometry due to intramolecular CT [9].

4.2. CT fluorescence

Excitation of the contact ion pair ($^{+}\mathrm{A_t}\ \mathrm{I^-}$) leads to the excited singlet state with CT character, step (1), and to the emission of light, step (2).

$$(^{+}\mathrm{A_t}\mathrm{I^-})\, h\nu^{CT} \rightarrow 1*(^{+}\mathrm{A_t}\mathrm{I^-}) \tag{1}$$

$$(^{1*}\mathrm{A_t}\mathrm{I^-}) \rightarrow h\nu_f^{CT} \rightarrow (^{+}\mathrm{A_t}\mathrm{I^-}) \tag{2}$$

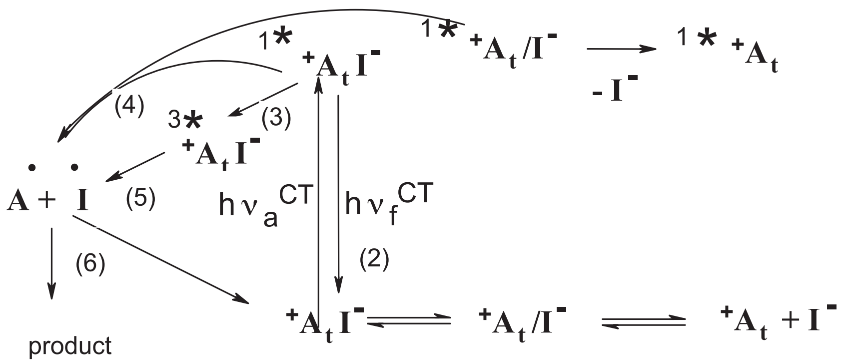
The fluorescence emission and excitation CT spectra of the styrylpyridinium iodides in THF show a mirror image relationship, see Figs. 1, 2 and 4. The blue (P4), green (P2) or yellow (P3/Q3) emission is efficient, see the substantial Φ_f^{CT} values in Tables 3 and 4. Concerning these effects there are only a few cases to compare in the literature [34] and no Φ_f^{CT} values as yet. A related effect dealing with emission and excitation CT spectra is phosphorescence of pyridinium iodides at low temperatures [26].

The plot of $1/\lambda_f^{CT}$ of the four *trans*-styrylpyridinium iodides vs. $-E^{\circ\prime}$ (Fig. 5) reveals a linear dependence of the excited singlet state level on the potential. Note that the fluorescence bands of P1, P2 and P4 have a red-shifted shoulder and therefore the $1/\lambda_f^{CT}$ midpoint would be somewhat lower. No differences in λ_t, λ_f or Φ_f were found for the methoxystyrylquinolinium iodide Q4 or perchlorate Q′4 in THF (420, 560 nm or 0.01, respectively) in contrast to the *trans*-methoxystyrylpyridinium salts P′4 and P4, indicating the absence of CT interaction in the quinolinium case. The fluorescence of styrylpyridinium iodides frequently deals with free cations of P-NMe$_2$ type [9–18], but for these molecules no intermolecular CT fluorescence is known as yet. The presence of such a dialkylamino group may cause a fast deactivation via internal conversion at the *trans* geometry, thus competing with emission.

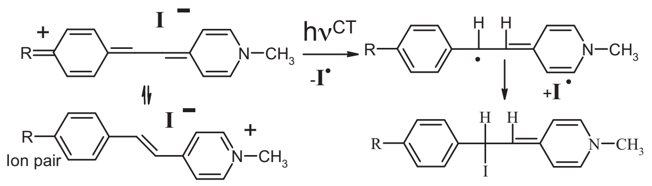
4.3. Deactivation pathways for ion pairs

In competition to fluorescence, intersystem crossing (3) occurs and formation of a radial pair (4/5), which may react by back electron transfer or leads to products (6).

$$^{1*}(^{+}\mathrm{A_t}\mathrm{I^-}) \rightarrow {}^{3*}(^{+}\mathrm{A_t}\mathrm{I^-}) \tag{3}$$



Scheme 1.



Scheme 2.

$$^{1*}({}^{+}A_tI^{-}) \rightarrow (A^{\bullet} + I^{\bullet}) \qquad (4)$$

$$^{3*}({}^{+}A_tI^{-}) \rightarrow (A^{\bullet}+I^{\bullet}) \qquad (5)$$

$$A^{\bullet}+I^{\bullet} \rightarrow ({}^{+}A_tI^{-}) + \text{products} \qquad (6)$$

Electron transfer steps (4) or (5) take place for iodides due to the low ionization potential, but not where perchlorates are present. For a possible reduction product, see Schemes 1 and 2. The major reaction is electron back transfer to the undamaged ion pair. The transient absorption spectra of the *trans*-nitrostyryl salts reveal a triplet state after the 308 or 353 nm pulse for iodides (P3, Q3) in polar solvents and for the perchlorates (P′3, Q′3) in any medium examined. The longer lived transient of P3 or Q3 in THF is ascribed to the nitrostyrylaromatic radical, i.e. electron transfer occurs also from the triplet state [6].

Fluorescence quenching of aromatic compounds in acetonitrile by iodide has been investigated [32]. For benzoquinolizinium iodides a significant solvatochromism has been studied [28]. For a bispyridinoethylene (BPE^{2+}) in acetonitrile, electron transfer with a quantum yield of 0.1, producing $I_2^{\bullet-}$ and the corresponding BPE$^{\bullet+}$ radical, has been reported [33]. It should be recalled that pyridinium iodides are model compounds to reveal the degree of CT character in a given solvent [25–30]. Interestingly, the CT absorption of a pyrylium iodide has been introduced by Kosower as an emperical solvent polarity parameter: *Z*-value [29]. This *Z*-value is in good agreement proportional to the E_T^N-value of numerous solvents [35,36].

5. Conclusions

The effects of solvents on the ground and excited singlet states of *trans*-4-R-styrylpyridinium and complementary styrylquinolinium iodides with R = OCH$_3$, H, CN and NO$_2$ were examined. The CT absorption and fluorescence features are due to photoinduced electron transfer from iodide to the excited singlet state of the cation. They are most pronounced in THF, where contact ion pairs are present. The excited singlet state level depends on the reduction potential. A major finding is that the quantum yield of CT fluorescence is relatively large: Φ_f strongly enhanced upon excitation of the CT band centered at 440–450 (or 300) nm with respect to that of the hypsochromicly shifted main absorption band.

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