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Spectroscopic studies on molecular interactions of photogenerated tricyanovinylstyrylpyridinyl radicals in acetonitrile at room temperature

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Abstract

Photoinduced electron transfer reactions of newly synthesized 1-hexadecyl-4-(4-tricyanovinylstyryl)pyridinium tetraphenylborate salts in acetonitrile were studied at room temperature by steady-state photolysis and ESR spectroscopy. Upon irradiation, stable tricyanovinylstyrylpyridinyl radicals were formed by interionic electron transfer accompanied with color change from pale yellow to blue. The photogenerated radicals showed a strong tendency to interact with each other or tricyanovinylstyrylpyridinium cations. A fairly strong charge resonance (CR) band was observed in the near infrared region with a peak at 924 nm, which was attributed to the electronic interaction between a tricyanovinylstyrylpyridinyl radical and a tricyanovinylstyrylpyridinium cation. Observed and simulated ESR spectra clearly confirmed the formation of both radical dimers and dimer radical cations in solution. An additional electron-withdrawing cyano group in tricyanovinylstyrylpyridinium tetraphenylborate salts greatly stabilized its dimer radical cation compared with that of dicyanovinylstyrylpyridinium tetraphenylborate salts. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Tricyanovinylstyrylpyridinium tetraphenylborate; Ion pair charge transfer complex; Photoinduced electron transfer; Charge resonance band; Dimer radical cations

1. Introduction

We have been studying photochemical and photophysical properties of ion-pair charge transfer (IPCT) complexes in various microenvironments to control both their linear and nonlinear optical properties photochemically, as potential candidates for future molecular photonics devices [1]. By the use of tetrakis[3,5-bis(trifluoromethyl)phenyl]borate as the counter anions of 4,4′-bipyridinium ions, we have achieved novel photochromism due to photoinduced electron transfer and thermal reverse reactions in microcrystals [2], organic solutions [3], Langmuir–Blodgett films [4] and polymers [5]. Ultrafast color changes, within less than 1 ps upon excitation of IPCT bands, were observed by femtosecond laser photolysis [6,7]. The lifetime of photogenerated blue 4,4′-bipyridinium radicals was controlled by selecting the counter anions, the microenvironment, and the temperature, over a very wide range from 1.2 ps to almost infinity [7]. Novel second harmonic generation from IPCT complexes was also achieved using substituted styrylpyridinium salts [8].

Several years ago, we reported for the first time, the existence of the charge resonance (CR) band as a broad absorption with a peak at 950 nm upon steady photoexcitation of 1-alkyl-4-(4-nitrostyryl)pyridinium tetraphenylborate (NS^+TPB^-) in 1,2-dimethoxyethane (DME) solution [9]. In more polar acetonitrile (ACN), the CR band of NS^+-TPB^- was very weak and showed a peak at 1166 nm [10]. The tendency for self-association of nitrostyrylpyridinium radicals was also indicated from the observed complex absorption changes following prolonged irradiation and/or storage in the dark after irradiation [11]. We also found that the CR band is strongly affected by the structure of a substituent at the 4-position of the styryl group. 1-Alkyl-4-(4-dicyanovinylstyryl)pyridinium tetraphenylborate (DCS^+-TPB^-) showed the CR band at 1050 nm together with a radical dimer CT band at 1120 nm upon irradiation in DME [12]. Very recently we have reported very stable two charge resonance bands with peaks at 948 and 1742 nm for 1,3-bis[4-(4-nitrostyryl)pyridinium)propane tetraphenylborate in acetonitrile at room temperature, which were attributed to sandwich-type and partially overlapped configurations of

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intramolecular dimer radical cations, respectively [13]. For these three cases, dimer radical cations with a characteristic CR band survived for up to 1 h in DME at room temperature. We have also reported ultrafast dynamics of photogenerated radicals and dimer radical cations studied by femtosecond laser photolysis [14]. Both radicals and dimer radical cations were formed within less than 1 ps. The rate of back electron transfer reactions to oxidized donors was faster from dimer radical cations than that from monomer radicals [14].

It is well known that an aromatic radical cation formed by chemical oxidation [15,16], γ-radiolysis [17,18], pulse radiolysis [19–21], or flash photolysis [22–30] makes an electronic interaction with a neutral chromophore to form a dimer radical cation. However, all dimer radical cations except ours are stable only at very low temperature [15–21] or exist as short-lived intermediates at room temperature [22–30]. The interaction between two chromophores in dimer radical cations results in the splitting of both HOMO and LUMO energy into two sub-levels as schematically shown in Fig. 1. The transition between the sub-levels gives a specific absorption in the near infrared (NIR) region, the CR band. The stabilization energy (ΔH) of the dimer radical cation is approximately equal to half the energy of the CR band [31,32]. The interaction is affected by many factors such as attractive charge transfer, repulsive electronic and steric forces. The stronger the interaction, the shorter shifts the wavelength of the CR band in the NIR region.

In order to use photogenerated radicals as materials showing absorption and refractive index changes in the NIR region, we aimed to stabilize them by using a newly synthesized compound with three strong electron-withdrawing cyano groups, 1-alkyl-4-(4-tricyanovinyl-styryl)pyridinium tetraphenylborate ($TCS^{+}C_{16}TPB^{-}$). Upon irradiation, extremely stable radicals and dimer radical cations with specific optical properties were observed even in polar ACN solutions at room temperature.

2. Experimental

2.1. Materials

1-Alkyl-4-(4-tricyanovinylstyryl)pyridinium tetraphenylborate was synthesized by the scheme shown in Fig. 2. The synthesis of intermediate compounds **C**–**E** was similar to that reported by Katz et al [33]. We have considerably improved the yields of compound **C** by modifying the reaction conditions and compound **D** by using visible light irradiation, respectively.

2.1.1. Compound *B*

Terephthaldehyde monodiethylacetal **A** 26 g (125 mmol) and malononitrile 14 g (250 mmol) were dissolved in 120 ml of ethanol with a small amount of piperidine. After refluxing for 30 min, the solvent was removed under reduced pressure to get a red product. Compound **B** [12] was then purified by silica column chromatography (eluent: benzene) as a pale yellow oil (70%). NMR ($CDCl_3$, δ ppm): 1.3 (6H, t, CH_3), 3.6 (4H, q, CH_2), 5.6 (1H, s, CH), 7.8 (1H, s, –CH=), 7.6–8.0 (4H, q, Ph-H).

2.1.2. Compound *C*

25 ml of an aqueous solution of KCN 4.5 g (69 mmol) was added to a solution of **B** 14 g (54 mmol) in 25 ml of ethanol. After heating on a steam bath for 2 min, 40 ml of an aqueous solution containing 12 ml of acetic acid was poured into the solution under stirring. The mixture was partitioned between 150 ml of $CHCl_3$ and 100 ml of water, and the organic layer

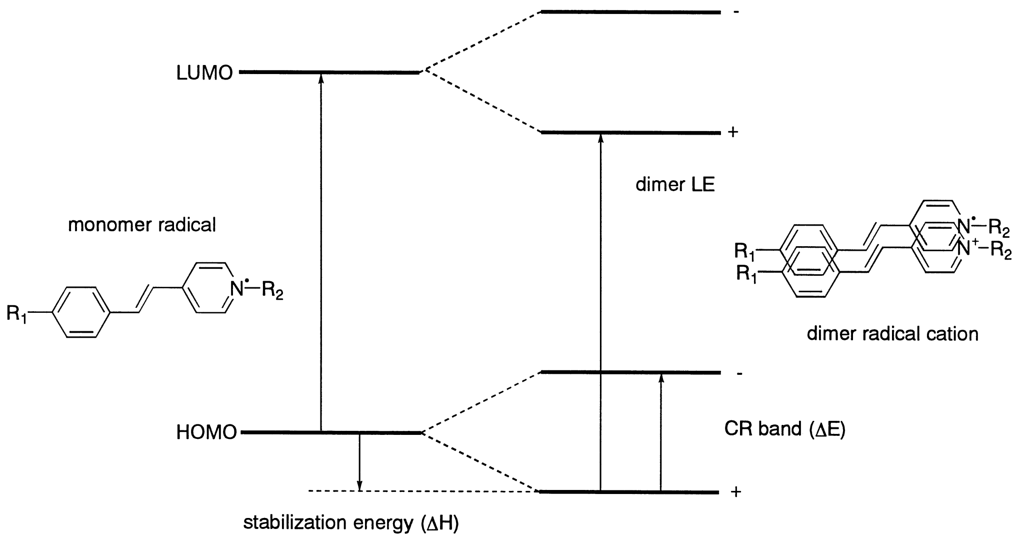

Fig. 1. Schematic representation of the energy levels in monomer radical and dimer radical cations. ΔH: the stabilization energy, ΔE: the charge resonance band.

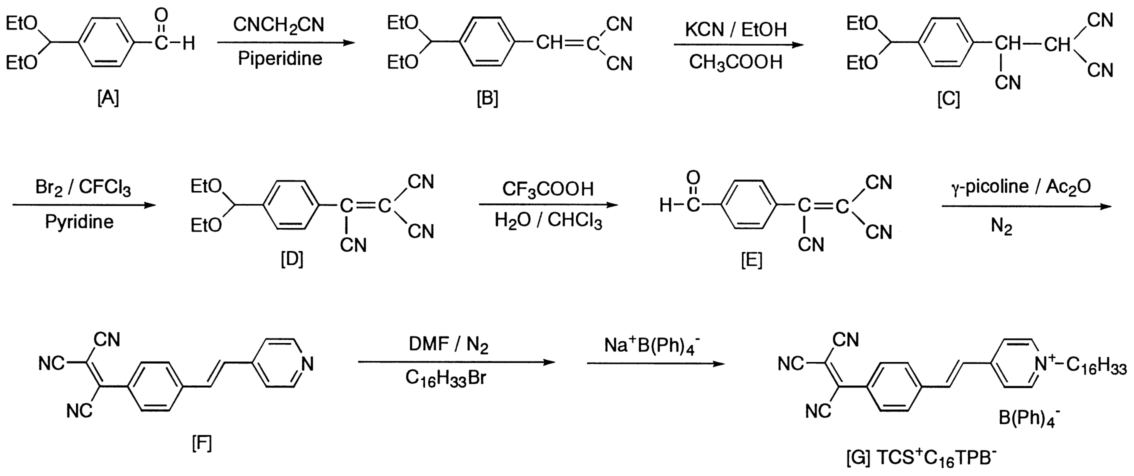

Fig. 2. Synthetic route for 1-hexadecyl-4-(4-tricyanovinylstyryl)pyridinium tetraphenylborate, $TCS^+C_{16}TPB^-$.

was washed twice with water. Purification by silica column chromatography with chloroform yielded a pale green oil (58%). NMR ($CDCl_3$, δ ppm): 1.2 (6H, t, CH_3), 3.6 (4H, q, CH_2), 4.4 (1H, d, CH(CN)), 4.5 (1H, d, $CH(CN)_2$), 5.5 (1H, s, CH), 7.3–7.7 (4H, AB, Ph-H).

2.1.3. Compound **D**

Compound **C** 7 g (23 mmol) was dissolved in 25 ml of pyridine in an ice bath. A cooled solution of bromine 4 g in 10 ml of $CFCl_3$ was added dropwise under visible light irradiation using a 150 W Xe-Hg lamp and an L-39 filter (λ>365 nm). This light was kept on till the end of the reaction. The mixture was kept stirring for 1 h to reach room temperature. It was then poured into water to precipitate a yellow solid. Further purification by column chromatography with benzene containing 1% methanol yielded a pale yellow solid (41%). NMR ($CDCl_3$,δ ppm): 1.2 (6H, t, CH_3), 3.6 (4H, q, CH_2), 5.5 (1H, s, CH), 7.6–8.0 (4H, q, Ph-H).

2.1.4. Compound **E**

A mixture of 12 ml of water and 12 ml of CF_3COOH was added dropwise to a 12 ml of chloroform solution of compound **D** 2.9 g (10 mmol) in an ice bath. After stirring for 3 h during which time it was allowed to reach room temperature, the mixture was partitioned between 80 ml of chloroform and 100 ml of water twice. The organic layer was dried and the solvent was removed under reduced pressure. Yellow crystals were obtained (95%). NMR ($CDCl_3$, δ ppm): 8.19 (4H, s, Ph-H), 10.2 (1H, s, CHO).

2.1.5. Compound **F**

Compound **E** 0.6 g (6 mmol) and an equimolar amount of 4-picoline were heated in an equimolar amount of acetic anhydride at 110°C under a nitrogen atmosphere for 0.5 h. Purification by silica column chromatography (eluent: $CHCl_3$) yielded compound **F** as a yellowish solid (10%). NMR ($CDCl_3$, δ ppm): 7.6–8.0 (8H, m, Ph-H, –CH=CH–, Py-H). 8.7 (2H, d, Py-H).

2.1.6. Compound **G**

97 mg Compound **F** was dissolved in 20 ml of DMF and heated with 125 mg of hexadecylbromide at 110°C for 3 h. The mixture was poured into ether to yield brown precipitate. The product was dissolved in methanol and a solution of sodium tetraphenylborate 200 mg in 5 ml of methanol was added to give a brown solid of compound **G** (60%). NMR for compound **G** (CD_3COCD_3, δ ppm): 0.87 (3H, t, $-CH_3$), 1.3 (26H, m, $-(CH_2)_{13}-$), 4.8 (3H, t, N–CH_2), 6.8–7.3 (12H, m, BPh_4-H), 7.3–7.4 (8H, m, BPh_4-H), 7.4–8.5 (8H, m, Ph-H, –CH=CH–, Py-H), 9.1 (2H, d, Py-H).

2.2. Measurements

Absorption spectra were recorded on a Hitachi U-3500 UV–Vis-NIR spectrophotometer at room temperature. Sample solutions for both absorption and ESR measurements were degassed by repeated freeze-pump-thaw cycles. A 150 W Hg-Xe lamp through an L-39 cut off filter (λ_{ex}>365 nm) and an IR cut-off filter was used as an irradiation source. Sample solutions were stirred with a magnetic stirrer during irradiation unless otherwise indicated. The ESR spectra during and after irradiation without stirring were measured at room temperature with a Bruker ESP300E spectrometer (100 kHz field modulation).

3. Results and discussion

$TCS^+C_{16}TPB^-$ showed dramatic color changes (pale yellow→blue→violet) in acetonitrile (ACN) upon steady irradiation. The difference absorption spectra of TCS^+C_{16}-TPB^- in ACN solutions (0.05 mM) using an unirradiated sample as a reference are shown in Fig. 3 for three different irradiation times. A new absorption in the visible region with a peak at 618 nm and a broad absorption in the NIR with a peak at 924 nm were observed for shorter irradiation times. The newly observed peaks disappeared when air was

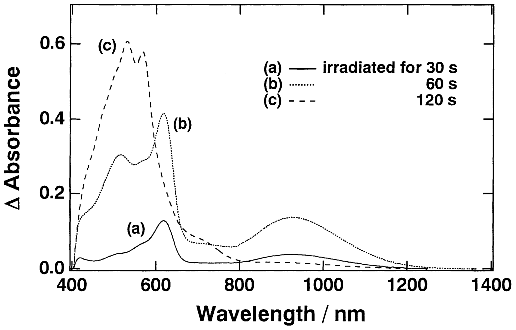

Fig. 3. Difference absorption spectra of $TCS^+C_{16}TPB^-$ (0.05 mM) in ACN solutions upon irradiation. Absorption spectrum before irradiation was used as a reference.

introduced to the irradiated solutions. These results indicate that the new peaks are due to photogenerated radicals which can only exist under oxygen-free conditions. Sharp peaks similar to that at 618 nm in the visible region were observed at 570, 564 and 642 nm for NS^+TPB^- in DME [9,11] and ACN solutions [10], and DCS^+TPB^- in DME solution [12], respectively. They were assigned to the absorption band of the corresponding styrylpyridinyl radicals [9,11,12]. From these results, the sharp peak at 618 nm was attributed to tricyanovinylstyrylpyridinyl radicals formed by photoinduced electron transfer from TPB^- anion to TCS^+C_{16} cation. TPB^- anion is well known to be decomposed after one-electron oxidation [34], which prevents the back electron transfer reaction. The broad NIR absorption bands were observed for irradiated NS^+TPB^- at 950 nm and DCS^+TPB^- at 1050 nm in DME solutions, which have been assigned to the CR band [9,12]. From comparison with these facts and the results mentioned below, the broad NIR absorption at 924 nm for $TCS^+C_{16}TPB^-$ in ACN solutions was assigned to the CR band due to the formation of dimer radical cations. The local excitation (LE) band of dimer radical cations as schematically shown in Fig. 1, thus, also contributed to the absorption with a peak at 618 nm.

After long irradiation, the color of the solutions turned to violet, which corresponded to the appearance of a new absorption with peaks at 530, 567 nm and a concomitant decrease of the peak at 618 nm and the NIR band, as shown in Fig. 3(c). In higher concentrations (0.1–0.5 mM), only the band with the peaks at 530, 567 nm were observed upon irradiation without the peak at 618 nm and the NIR band. This strongly suggests further reactions from photogenerated radicals and dimer radical cations. From the ESR spectra mentioned below, the band with peaks at 530 and 567 nm is most probably attributed to tricyanovinylstyrylpyridinyl radical dimers. The photogenerated tricyanovinylstyrylpyridinyl radicals have thus stronger tendency to interact with each other.

ESR spectrum of $TCS^+C_{16}TPB^-$ after irradiation for 2 min in ACN solution (0.1 mM) is shown in Fig. 4(a). This ESR spectrum observed during irradiation was a very broad singlet. After keeping it in the dark for 30 min, a new ESR spectrum with more than seven sharp peaks appeared, but the broad singlet still remained as shown in Fig. 4(b). After 5 h in the dark, the broad singlet almost disappeared, while the sharp spectrum remained almost unchanged. These results indicate that the broad ESR signal comes from the exchange broadening in radical dimers because of a high local concentration of radicals photogenerated in an ESR tube with an inner diameter of 1 mm. The local high concentration may be kept for a long time, since the diffusion is slow in the ESR tube. After storing in the dark for a long time, the radical dimers will be diluted by the unirradiated TCS^+C_{16} cations to show hyperfine structure, as shown in Fig. 4(b).

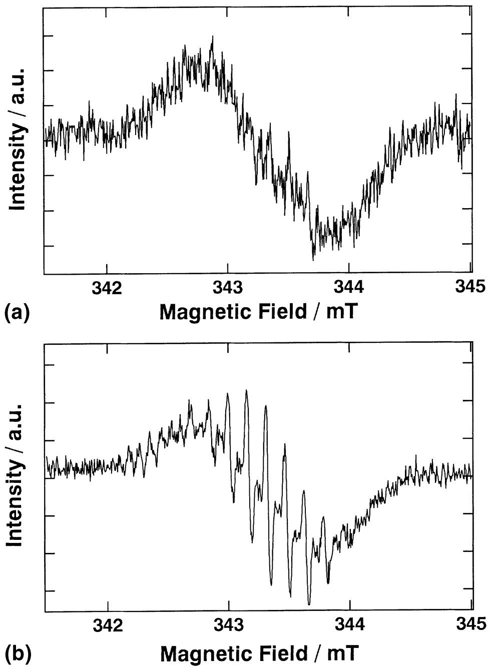

Fig. 4. ESR spectra of $TCS^+C_{16}TPB^-$ (0.1 mM) in ACN solution; during irradiation for 2 min and (b) 30 min in the dark after irradiation.

In order to compare with the ESR results, irradiation was also made in a 1 mm cell without stirring. The difference absorption spectrum after irradiation for 60 s without stirring shows peaks at 535, 570 nm and a small broad absorption in the NIR region, as shown in Fig. 5(a). The difference absorption spectrum after irradiation for 60 s is similar to that of 120 s irradiation in the 1 cm cell under stirring shown in Fig. 3(c). These results are due to slower diffusion in the 1 mm cell which results in higher local concentration of photogenerated radicals at shorter irradiation times. From these results and the ESR results shown in Fig. 4, the peaks at 530–535 and 567–570 nm were attributed to radical dimers. During storage in the dark at room temperature up to 30 min, two peaks at 530 and 567 nm decreased. During storage in the dark at room temperature up to 30 min, new peaks appeared at 614 and 924 nm in concomitant slow decrease of two peaks at 530 and 567 nm showing isosbestic points at 600, 650 and 800 nm as shown in Fig. 5(a). Absorbance

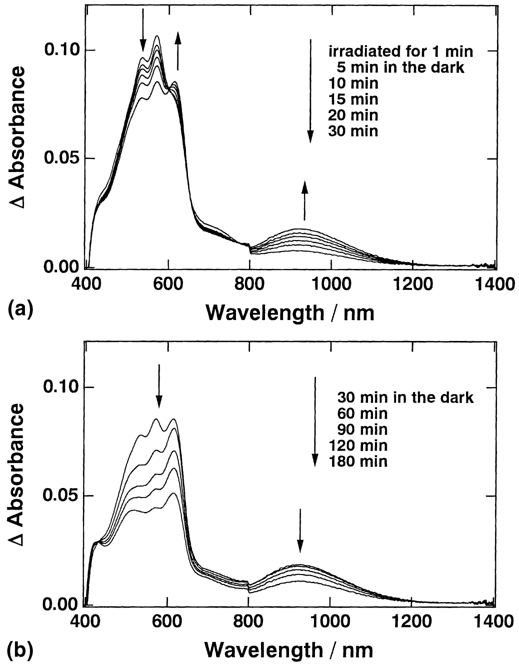

Fig. 5. Difference absorption spectra of $TCS^{+}C_{16}TPB^{-}$ (0.1 mM) in ACN solutions in 1 mm cell in the dark after irradiation for 60 s without stirring. Absorption spectrum before irradiation was used as a reference.

at 924 nm increased more than twice during such a process. The new absorption peaks were attributed to the dimer LE band and the CR band of dimer radical cations, respectively as mentioned above. The dimer LE band observed at 618 nm in Fig. 3 was apparently blue-shifted by the overlap of a strong absorption band of radical dimers in Fig. 5(a). It is thus concluded that radical dimers were converted to dimer radical cations by slow dilution with unirradiated $TCS^{+}C_{16}$. After 30 min standing in the dark, the whole spectrum began to decrease monotonically due to the decay of radicals, as shown in Fig. 5(b).

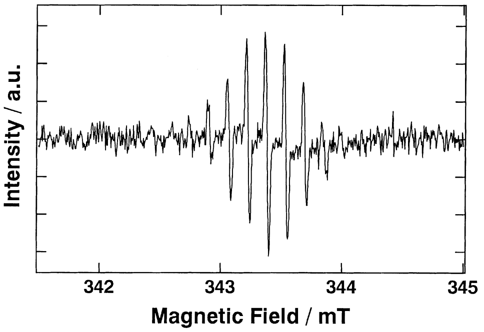

Fig. 6. ESR spectrum of $TCS^{+}C_{16}TPB^{-}$ (0.1 mM) in ACN solution irradiated for 10 min and after mixing with an unirradiated sample.

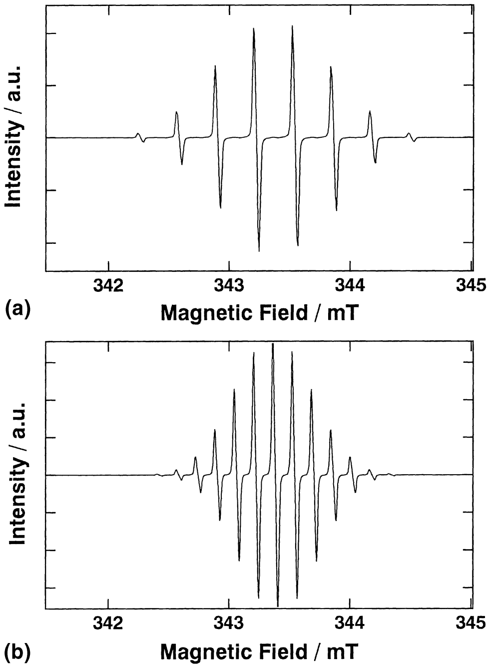

Fig. 7. Simulated ESR spectra of monomer (a) and dimer radical cation (b) using a Lorentzian profile with a component line width of 0.005 mT.

After mixing $TCS^{+}C_{16}TPB^{-}$ irradiated for 10 min in ACN solutions with an unirradiated one in a branch of the ESR tube, the ESR spectrum dramatically changed from a very broad singlet as shown in Fig. 4(a) to well resolved hyperfine structures as shown in Fig. 6. This signal was very stable. In order to assign the observed spectrum, we simulated the ESR spectra of the styrylpyridinyl radical (a) and its dimer radical cation (b) using a Lorentzian shape with a component line width of 0.005 mT as shown in Fig. 7. The observed spectrum showed a maximum intensity at the center, which corresponded to dimer radical cations shown in Fig. 7(b). The following hyperfine coupling (hfc) constants and their half values were used for the radical monomer and dimer radical cation, respectively, $a(\mathrm{H})_{2,6} = a(\mathrm{H})_{\mathrm{NCH_2}} = a(\mathrm{H})_{\mathrm{vinyl}} = a(\mathrm{N}) = 0.32\,\mathrm{mT}$, $a(\mathrm{H})_{3,5}=0.01\,\mathrm{mT}$. These values were obtained from the best fit with the observed spectrum shown in Fig. 6. They are similar to those reported for dicyanovinylstyrylpyridinyl radicals [12].

When the solution irradiated in a 1 mm cell without stirring was mixed with an unirradiated sample, two peaks at 530 and 567 nm in Fig. 8(a) disappeared. At the same time,

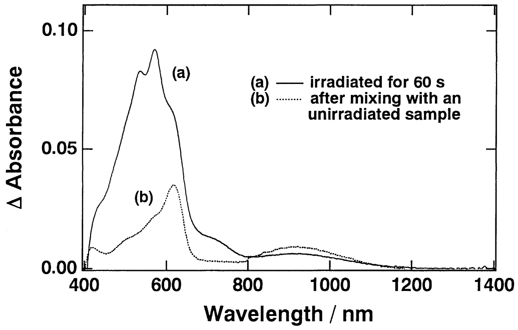

Fig. 8. Difference absorption spectra of $TCS^{+}C_{16}TPB^{-}$ (0.1 mM) in ACN solutions in 1 mm cell (a) after irradiation for 60 s without stirring and (b) after mixing with an unirradiated sample. Absorption spectrum before irradiation was used as a reference.

the sharp peak at 618 nm appeared and the broad peak at 924 nm increased as shown in Fig. 8(b). This result clearly indicated that the radical dimers were diluted with unirradiated TCS^+C_{16} cations to form dimer radical cations. The visible and NIR absorption spectral changes observed after mixing perfectly corresponded with the ESR spectral changes mentioned above.

TCS^+C_{16} cations have only one more electron withdrawing cyano group as compared with DCS^+C_{16} cations, but they showed considerable differences in terms of their photoreactivity. A fairly strong CR band was observed for $TCS^+C_{16}TPB^-$ irradiated in ACN solutions and the dimer radical cations survived for about 2 h, whereas neither visible nor NIR absorptions were observed for $DCS^+C_{16}TPB^-$ in ACN solutions [12]. In less polar 1,2-dimethoxyethane, both $TCS^+C_{16}TPB^-$ and $DCS^+C_{16}TPB^-$ gave radicals and dimer radical cations upon irradiation. But the lifetime and the stabilization energy (ΔH) estimated from the CR band were very different. They were more than 100 h and 65.3 kJ/mol for $TCS^+C_{16}TPB^-$, while less than 1 h and 56.9 kJ/mol for $DCS^+C_{16}TPB^-$. Because of the extra cyano group present in TCS^+C_{16} cations, stronger delocalization of the unpaired electron is expected, which most probably contributed to the increased stability of $TCS^\bullet C_{16}$ radicals and their dimer radical cations.

4. Conclusion

A new tricyanovinylstyrylpyridinium tetraphenylborate salt was synthesized. An additional electron-withdrawing cyano group in tricyanovinylstyrylpyridinium tetraphenylborate salts greatly stabilized its dimer radical cation compared with that of dicyanovinylstyrylpyridinium tetraphenylborate salts. These results will contribute a great deal to the stabilization of photogenerated radicals and also to the molecular control of their photophysical properties in the NIR region. We recently achieved very fast all-optical light modulation and switching in the visible region by photoinduced complex refractive index change in a guided wave mode geometry [35–38]. It is also necessary to have materials showing large absorption or refractive index changes in the NIR region in order to use them as future ultrafast molecular photonics devices compatible with the present light communication system.

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