

Photoinduced electron transfer of the triplet states of C₆₀ and C₇₀ from oxotitanium(IV) tetra-*t*-butyl-phthalocyanine as an electron-donor in polar solvent

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Abstract

Excited triplet state properties of oxotitanium(IV) tetra-*t*-butyl-phthalocyanine (OTiPc) were investigated by the laser flash photolysis observing the transient absorption bands in the near-IR region. Photoinduced energy and electron transfer processes of the excited triplet states of C₆₀ and C₇₀ in the presence of OTiPc take place depending on the solvent polarity. In PhCN, slower formations of the ion radicals after the rapid decays of the triplet excited states of C₆₀ and C₇₀ were observed, suggesting the presence of the intermediates (i.e. triplet exciplex) for electron transfer. In longer time-scale, freely solvated radical ions show second-order back electron transfer.

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1. Introduction

Phthalocyanines (PCs) are also known as promising compounds for photoelectric devices [1]. Especially, oxotitanium(IV) phthalocyanine has been widely applied to various devices [2]. Some of these systems have been applied to the photoelectric conversion systems [3,4]. Although the solid state and film state of oxotitanium(IV) phthalocyanine have been studied extensively [1–4], only a few studies were reported for the properties in solutions because of the low solubility of oxotitanium(IV) phthalocyanine in ordinal organic solvents [5]. To study the properties in solutions, oxotitanium(IV) phthalocyanine was substituted with alkyl groups [6]. In the present study, we employed oxotitanium(IV) tetra-*t*-butyl-phthalocyanine (abbreviated as OTiPc in the present study; [Scheme 1](#)) which is quite soluble in ordinal solvents.

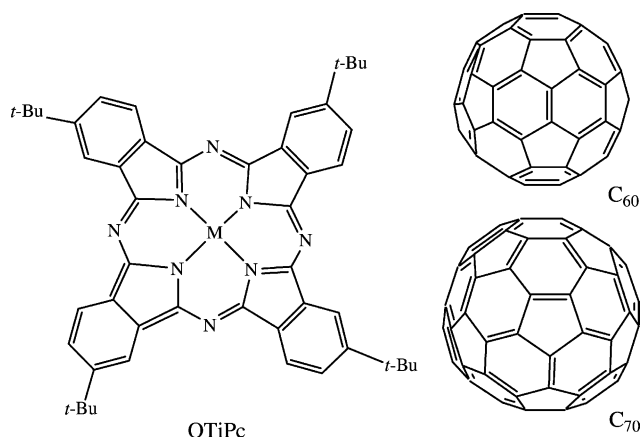
On the other hand, fullerenes such as C₆₀ and C₇₀ are great partners for light-induced electron transfer processes, holding strong promise in the fabrication of photoelectric devices [7]. In the composite system of OTiPc and fullerene, the photoelectron generation has been confirmed [8,9]. In the mixture systems of porphyrins (free and zinc)

or phthalocyanines (free and zinc) with fullerenes (C₆₀ and C₇₀) in polar solvents, photoinduced electron transfer takes place via the triplet states of these chromophores [10,11]. In the non-polar solvents, the energy transfer would be anticipated, but it was quite rare to observe the energy transfer, but collisional quenching or exciplex formation has been presumed [10,11]. In the connected systems of phthalocyanines with C₆₀ with covalent bond [12,13], the charge-separation usually takes place via the singlet excited state similar to the connected systems of porphyrins with C₆₀ [14,15].

Such efficient photo-conversion is a consequence of the remarkable acceptor properties of C₆₀ and C₇₀ [7]. On the other hand, ZnPc and H₂Pc are good electron-donors toward the triplet states of C₆₀ and C₇₀ [10,11]. In the present study, we investigated that when the central metal was placed with Ti=O, which seems to be having electron-withdrawing ability, the electron transfer takes place smoothly from OTiPc to the triplet states of C₆₀ and C₇₀ ([Scheme 1](#)).

In the present study, we have observed the transient absorption spectra of C₆₀ and C₇₀ by nanosecond laser flash photolysis in the visible and near-infrared regions, from which efficient photoinduced electron transfer of C₆₀ and C₇₀ with OTiPc was confirmed. The electron transfer rate constant was compared with H₂Pc and ZnPc as references.

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Scheme 1. Molecular structures of OTiPc, C₆₀ and C₇₀.

2. Experimental

2.1. Materials

C₆₀ (>99.9%) and C₇₀ (>99%) were purchased from Texas Fullerene Corp. Oxotitanium(IV) tetra-*t*-butyl-phthalocyanine (OTiPc) were prepared by the method described in the literature [16]. Toluene and benzonitrile (PhCN) used as solvents were of spectroscopic grade and HPLC grade, respectively.

2.2. Apparatus

Oxidation potential (E_{ox}) of OTiPc was measured by a voltammetric analyzer (BAS CV-50W) in a conventional three electrode-cell equipped with Pt-working and counter electrodes with an Ag/Ag⁺ reference electrode at a scan rate of 100 mV s⁻¹. In each case, solution contained (1.0–5.0) × 10⁻³ mol dm⁻³ of a sample with 0.1 mol dm⁻³ of tetrabutylammonium perchlorate (Nakalai Tesque); the solution was deaerated with Ar-bubbling before measurements.

Transient absorption spectra and time profiles were measured using a laser photolysis apparatus with a SHG of Nd:YAG laser (532 nm; 6 ns fwhm) as an exciting source. For the short time-scale transient absorption in the near-IR region, a Ge-APD module (Hamamatsu, C5334) was used to monitor the light from a pulsed Xe-lamp [17]. For long time-scale measurements, an InGaAs-PIN detector was used for monitoring the light from a continuous Xe-lamp. The details of the experimental set-up have been described elsewhere [18]. All the solutions were deaerated by Ar-bubbling before measurements, to eliminate the influence of oxygen. All measurements were carried out at 23 °C.

2.3. Molecular orbital calculations

Structure optimization and molecular orbital calculations of OTiPc were performed by HF/3-21G level using Gaussian-98 pack.

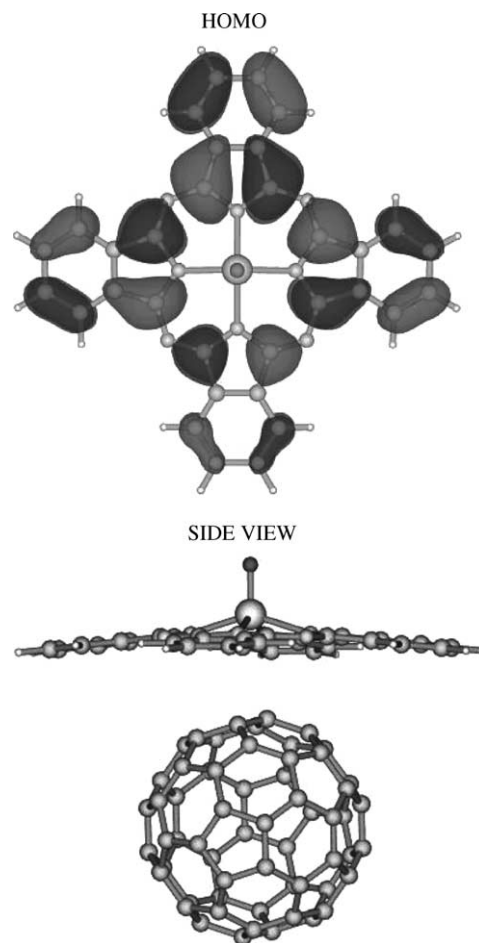


Fig. 1. Optimized structure and relative size with C₆₀ (lower panel) and electron distribution of HOMO of OTiPc (upper panel).

3. Results and discussion

3.1. Molecular orbital calculations

The π -electron of HOMO distributes in the whole molecule except Ti=O (upper panel in Fig. 1), suggesting that the electron-donor ability of the Pc-moiety is not decreased by the electron-withdrawing Ti=O. The optimized structure of OTiPc obtained by the MO calculation is shown in lower panel in Fig. 1, in which the phthalocyanine plane slightly bends with Ti=O at the top position. This curvature seems to be quite suitable to make encounter complexes (i.e. exciplexes) with C₆₀ and C₇₀. Although *t*-butyl groups were omitted in the MO calculations, these groups seem not to disturb the approach of C₆₀, as shown in lower panel of Fig. 1. For C₇₀, similar consideration is possible, even in the every directional approach of oval sphere of C₇₀.

3.2. Steady-state absorption spectra

The steady-state absorption spectra are shown in Fig. 2. The absorption peaks of OTiPc at 350 and 620–705 nm are

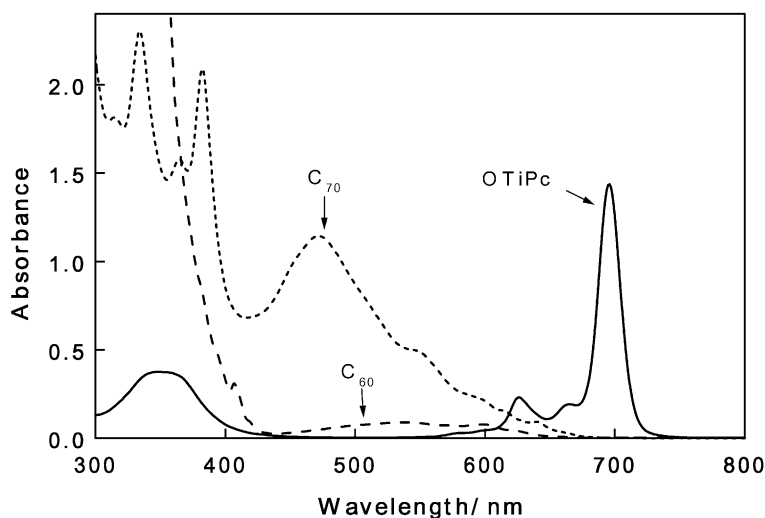


Fig. 2. Steady-state absorption spectra of OTiPc (0.005 mM), C₆₀ and C₇₀ (0.05 mM) in toluene.

characteristic of the Soret and Q-band, respectively. In the 450–550 nm region, there is no absorption under the ordinal concentration. On the other hand, C₆₀ and C₇₀ have appreciable absorption in this region; thus, the selective excitation of C₆₀ and C₇₀ was possible with the SHG laser light (532 nm) from YAG laser. In the longer wavelength region than 740 nm, there is a window to observe the transient absorptions of C₆₀ and C₇₀ and OTiPc.

The absorption intensities of the mixture of C₆₀ (or C₇₀) with OTiPc are almost the same with the superposition of both constituents in the whole visible region, indicating no considerable charge-transfer interaction between C₆₀ (or C₇₀) and OTiPc in the ground state. On further addition of C₆₀ and C₇₀ to OTiPc, the peak at 700 nm does not shift appreciably, which also indicates no interaction between C₆₀ or C₇₀ and OTiPc in the ground state.

3.3. Fluorescence spectra and lifetime

Steady-state fluorescence spectrum is shown in Fig. 3, in which the time profile is shown. The peak at 720 nm is a mir-

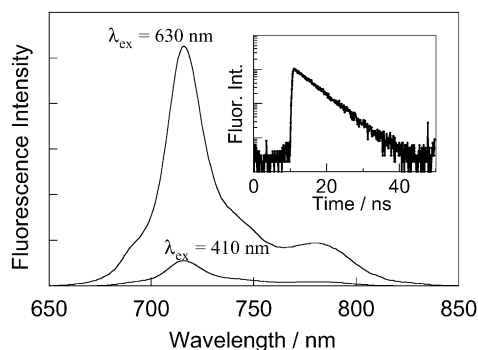


Fig. 3. Steady-state fluorescence spectra of OTiPc (0.02 mM) in toluene. Inset: Logarithm of fluorescence intensity at 730 nm vs. time.

ror image of the absorption band at 700 nm. The time profile shows a single exponential decay with the fluorescence lifetime of 5.1 ns. On addition of C₆₀ and C₇₀ to OTiPc, the fluorescence peak at 720 nm does not shift appreciably, which indicates no interaction between C₆₀ or C₇₀ and OTiPc even in the excited singlet state. The fluorescence intensity at 720 nm also did not decrease on addition of C₆₀ and C₇₀, which also indicates no dynamic quenching of the excited singlet state of OTiPc with C₆₀ or C₇₀ in the investigated concentration range (<10 mM).

3.4. Nanosecond transient absorption spectra

With the laser-light excitation of OTiPc in deaerated toluene, transient absorption spectra were observed as shown in Fig. 4. Immediately after the laser pulse, the transient absorption band appears at 1400 nm, which shows rapid decay as shown in time profile in Fig. 2. The rapid decay rate was evaluated to be larger than $1 \times 10^8 \text{ s}^{-1}$; since both the laser pulse duration (ca. 6 ns) and detector time constant (ca. 10 ns) of the nanosecond laser photolysis system employed in the present study permit to observe the photophysical and photochemical events longer than about 10 ns, this rapid decay may correspond to the fluorescence lifetime of 5.1 ns. Thus, the 1400 nm band with rapid decay is described to the S₁–S_n transition of OTiPc.

After the decay of 1400 nm band, a band appears at 1300 nm with slow decay. In the longer time-scale measurement, the slow decay rate was evaluated to be $1.5 \times 10^4 \text{ s}^{-1}$, which corresponds to the lifetime of 67 μs. On the addition of O₂, the decay rate of the 1300 nm band was increased; thus, the 1300 nm band was attributed to the triplet state of OTiPc (³OTiPc*). This lifetime of ³OTiPc* is longer than that of ZnPc (45 μs) [11].

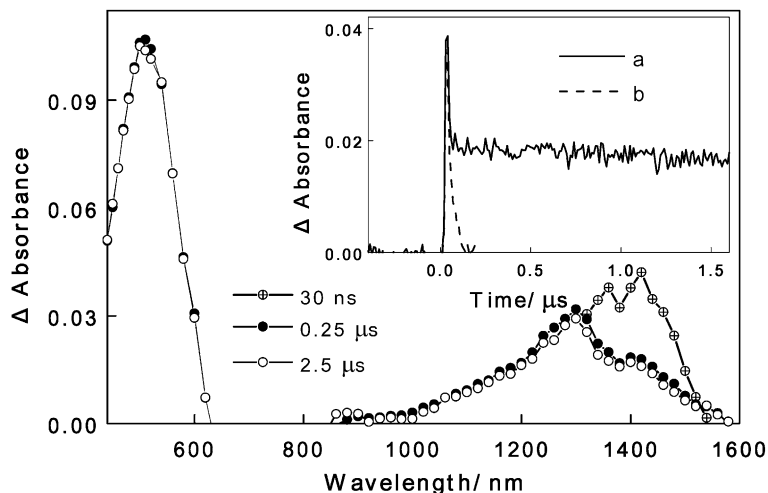
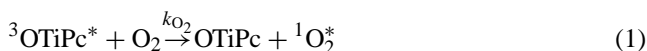


Fig. 4. Nanosecond transient absorption spectra of OTiPc (0.2 mM) in deaerated toluene observed by laser excitation of 532 nm light. Inset: Decay time profiles at 1400 nm in (a) Ar-saturated and (b) O₂-saturated toluene.

3.5. Energy transfer

With an increase in the concentration of O₂, the decay rates of ³OTiPc* increase giving the second-order rate constant for the energy transfer from ³OTiPc* to O₂ as shown in Eq. (1). On employing the O₂ concentration in O₂-saturated toluene solution as $1.1 \times 10^{-2} \text{ M}$ [19], the k_{O_2} value was evaluated to be $1.5 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$, which is slightly larger than that of ZnPc ($6.1 \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$) [10]:



In the mixture of OTiPc with C₇₀ in toluene, the 532 nm laser light excites both OTiPc and C₇₀ as shown in Fig. 5, where the triplet state of C₇₀ was observed at 980 nm in the spectrum at 100 ns. The time profile at 980 nm shows the quick decay in the presence of OTiPc and the absorp-

tion intensity at 1300 nm shows the rise concomitant decay of the 980 nm band. This indicates that the energy transfer takes place from ³C₇₀* to OTiPc in toluene. The rate constant for this energy transfer (k_{ent}) was evaluated to be $3.3 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$ for ³C₇₀* and $2.0 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$ for ³C₆₀*, which are slightly smaller than the diffusion controlled limit (k_{diff}) in toluene ($1.2 \times 10^{10} \text{ M}^{-1} \text{ s}^{-1}$) [19]:



In the case of ZnPc, although the increases of the decay rates of ³C₆₀* and ³C₇₀* were observed in benzene, the rise of the ³ZnPc* was not observed [10,11], suggesting that energy transfer does not take place; probably collision of ³C₆₀* or ³C₇₀* with ZnPc quenches the triplet excited states of C₆₀ or C₇₀. On the other hand, OTiPc clearly shows energy transfer, probably because suitable relative conformation for energy

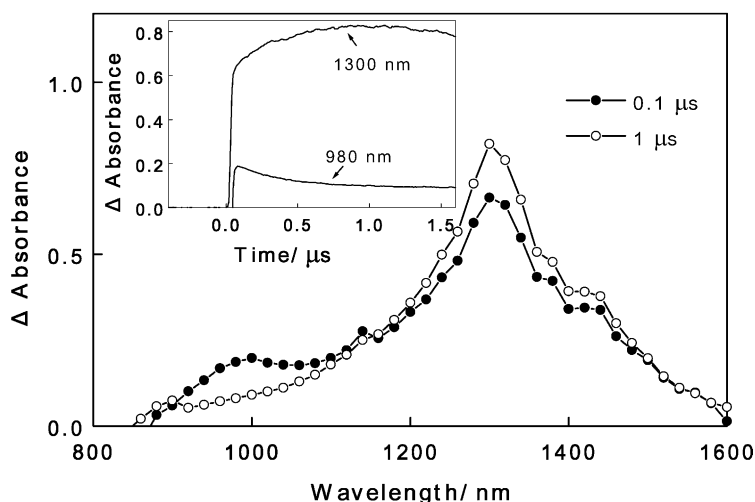


Fig. 5. Nanosecond transient absorption spectra observed by the laser excitation of C₇₀ (0.1 mM) in the presence of OTiPc (1.0 mM) in deaerated toluene. Inset: Time profiles at 980 and 1300 nm.

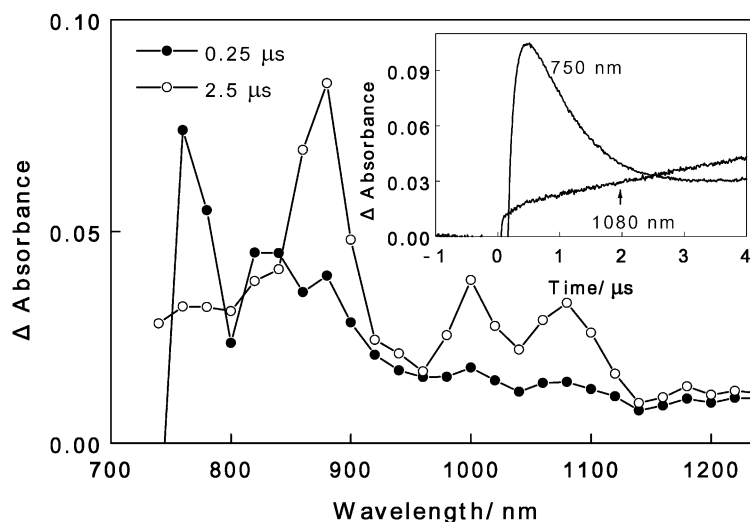
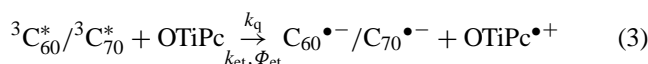


Fig. 6. Nanosecond transient absorption spectra observed by the laser excitation of C_{60} (0.1 mM) in the presence of OTiPc (0.5 mM) in deaerated PhCN. Inset: Time profiles at 750 and 1080 nm.

transfer may be possible between C_{60} or C_{70} and OTiPc in toluene by some electronic and steric reasons.

3.6. Electron transfer

In PhCN, the quite different transient absorption spectra from those in toluene were observed by the photoexcitation of C_{60} in the presence of OTiPc as shown in Fig. 6. The 750 nm band can be attributed to ${}^3C_{60}^*$. With concomitant decay of the 750 nm band, the rises of the absorption bands were observed at 880, 1000, and 1080 nm, in which the 1080 nm band can be attributed to the radical anion of C_{60} ($C_{60}^{\bullet-}$) [20], and the 880 nm band was ascribed to the radical cation of OTiPc ($OTiPc^{\bullet+}$), since the 880 nm band was reported for $OTiPc^{\bullet+}$ [4]. The 1000 nm band may also be ascribed to the overlap of the second absorption peak of $C_{60}^{\bullet-}$ with the absorption of $OTiPc^{\bullet+}$, although such a long wavelength band has not been reported in the literature [5]. These observations indicate that electron transfer takes place from OTiPc to ${}^3C_{60}^*$ (Eq. (3)). In this case, the absorption band at 1300 nm of ${}^3OTiPc^*$ was not observed, excluding the possibility of energy transfer from ${}^3C_{60}^*$ in PhCN. Direct excitation of OTiPc producing ${}^3OTiPc^*$ was also excluded in the presence of C_{60} . For C_{70} , similar transient absorption spectra showing the quick decay of ${}^3C_{70}^*$ at 980 nm and sequential rise of $C_{70}^{\bullet-}$ at 1380 nm with the absorption of $OTiPc^{\bullet+}$ at 880 and 1000 nm, indicating that electron transfer takes place from OTiPc to ${}^3C_{70}^*$ (Eq. (3)):



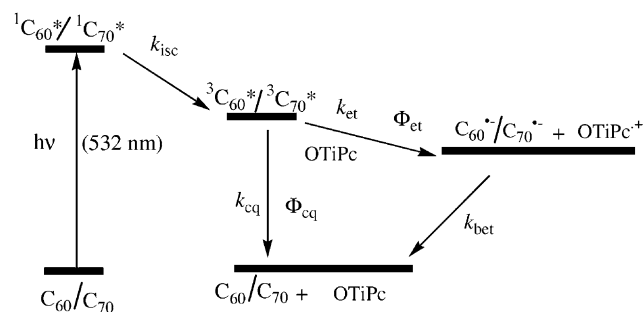
Oxidation potential of OTiPc ($E_{ox}(OTiPc)$) was evaluated to be 0.44 V versus ferrocene/ferrocenium in PhCN; thus, the free-energy change for electron transfer from OTiPc to ${}^3C_{60}^*$

was evaluated to be 0.64 eV by the Rehm–Weller equation (Eq. (4)) [21]:

$$\Delta G_{et}^{\circ} = E_{ox}(OTiPc) - E_{red}(C_{60}/C_{70}) - E_T(C_{60}/C_{70}) - E_c \quad (4)$$

Where $E_{red}(C_{60})$ is reduction potential ($=-0.51$ V versus SCE) [22], $E_T(C_{60})$ is the T_1 -energy level ($=1.53$ eV) [23], and E_c is the Coulomb energy (0.06 eV in PhCN) [24]. Thus, the negative ΔG_{et}° supports that electron transfer takes place as a whole. For C_{70} , ΔG_{et}° was evaluated to be -0.69 eV on employing $E_{red}(C_{70}) = -0.50$ V versus SCE [25] and $E_T(C_{70}) = 1.50$ eV [26]. Thus, the energy diagram is shown in Scheme 2.

The decay of ${}^3C_{60}^*$ at 750 nm (inset of Fig. 6) obeys first-order kinetics, which refers to $k_{1st-order}$, because OTiPc concentration is excess of ${}^3C_{60}^*$. From the dependence of $k_{1st-order}$ on the OTiPc concentration, the second-order quenching rate constant (k_q) of ${}^3C_{60}^*$ in the presence of OTiPc was evaluated to be $1.9 \times 10^9 M^{-1} s^{-1}$. However, the rise of $C_{60}^{\bullet-}$ at 1080 nm, which also obeys first-order kinetics ($k_{1st-order}$), seems to be slower than the $k_{1st-order}$ value of the decay of ${}^3C_{60}^*$ at the same OTiPc concentration



Scheme 2. The energy diagram.

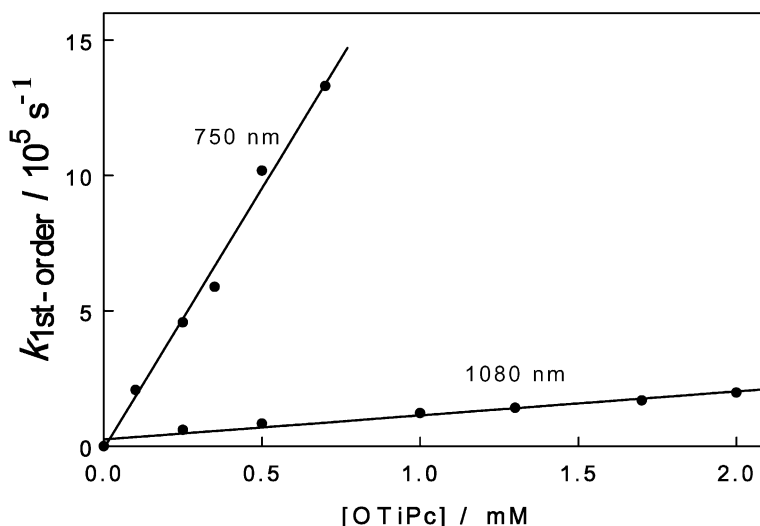
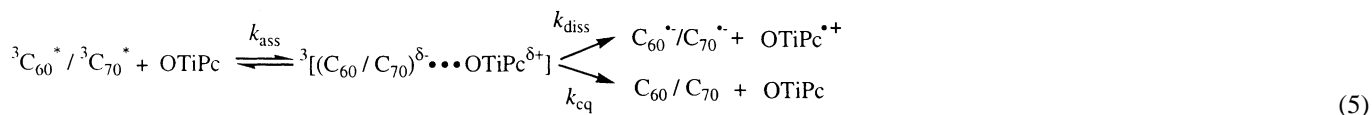


Fig. 7. Pseudo-first-order plots of C₆₀-OTiPc system in deaerated PhCN for the decay of ³C₆₀* at 750 nm and rise of C₆₀*⁻ at 1080 nm.

(inset of Fig. 6), which suggests some intermediate process between the decay of ³C₆₀* and rise of C₆₀*⁻ such as stable triplet exciplexes. As shown in Fig. 7, the difference between the decay of ³C₆₀* and rise of C₆₀*⁻ was quite large. When exciplex was assumed, the rise of C₆₀*⁻ at 1080 nm is attributed to the first-order rate constant for the dissociation of the triplet exciplex ($k_{1st-order} = k_{diss}$); however, the observed first-order rise-rate constants ($k_{1st-order}$) slightly depend on the OTiPc concentration, probably because reversible processes are included in the triplet exciplex formation. The k_{diss} value was evaluated to be ca. $2 \times 10^5 \text{ s}^{-1}$ from the observed k_{rise} value at high concentration of OTiPc:



The efficiency of electron transfer via ³C₆₀* can be evaluated by the ratio $[\text{C}_{60}^{\bullet-}]/[{}^3\text{C}_{60}^*]$. The initial absorbance of ³C₆₀* at 750 nm produced by one laser shot was decreased with the OTiPc concentration, because of emission of OTiPc. Under the low concentration region of OTiPc, the quantum yield (Φ_{et}) of electron transfer via ³C₆₀* was evaluated to be ca. 0.2. Such a low Φ_{et} value even in polar PhCN suggests the presence of deactivation process of ³C₆₀* without electron transfer, i.e. collisional quenching [27,28].

For C₇₀-OTiPc in PhCN, the transient absorption spectra showed that electron transfer takes place over all, since the decay of ³C₇₀* at 980 nm and rises of C₇₀*⁻ at 1380 nm [28] and of OTiPc*⁺ at 880 and 1000 nm were observed. However, the first-order decay rate ($k_{1st-order}$) of ³C₇₀* at 980 nm was far faster than the first-order rise rate ($k_{1st-order}$) of C₇₀*⁻ at 1380 nm (OTiPc*⁺ at 880 and 1000 nm) as shown in Fig. 8. It is notable that the time-scale of lower panel for the rises of OTiPc*⁺ at 880 nm is longer than that of upper panel for the decays of ³C₇₀* at 980 nm in Fig. 8.

In Fig. 9, the time profile at 980 nm is shown under one OTiPc concentration, where decay is attributed to ³C₇₀* and rise to OTiPc*⁺. Thus, decay of ³C₇₀* and rise of OTiPc*⁺ are shown in one panel at the same time-scale, which confirms the big time lag between the decay of ³C₇₀* between the rise of OTiPc*⁺. The second-order quenching rate constant of ³C₇₀* ($k_q = 4.1 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$) was evaluated, which is similar value to that of ³C₆₀*. The observed $k_{1st-order}$ values depend on the OTiPc concentration, probably because reversible processes were included in the triplet exciplex formation. The k_{diss} value for C₇₀-OTiPc was evaluated to

be $(3-4) \times 10^5 \text{ s}^{-1}$ at high OTiPc concentration; the k_{diss} value for C₇₀-OTiPc is quite similar to C₆₀-OTiPc, suggesting similar stability of the triplet exciplexes.

3.7. Back electron transfer

In the long time-scale measurements, the absorption intensities of the radical ions begin decaying after reaching maximal intensities. Such long time decay of the radical ions obeys second-order kinetics as shown in inset of Fig. 10 in PhCN, suggesting that back electron transfer takes place after the radical ions were solvated by PhCN. From the slope of the linear line, the ratio of k_{belt} to the molar extinction coefficient of the radical ions (ϵ_{ri}) was obtained. On assuming the reported ϵ_{ri} values for C₆₀*⁻ ($12000 \text{ M}^{-1} \text{ cm}^{-1}$ [26,29]) and for C₇₀*⁻ ($4000 \text{ M}^{-1} \text{ cm}^{-1}$ [27]), the k_{belt} values were evaluated to be 1.2×10^{10} and $7.2 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$, respectively. The k_{belt} value for C₇₀*⁻ is close to the diffusion

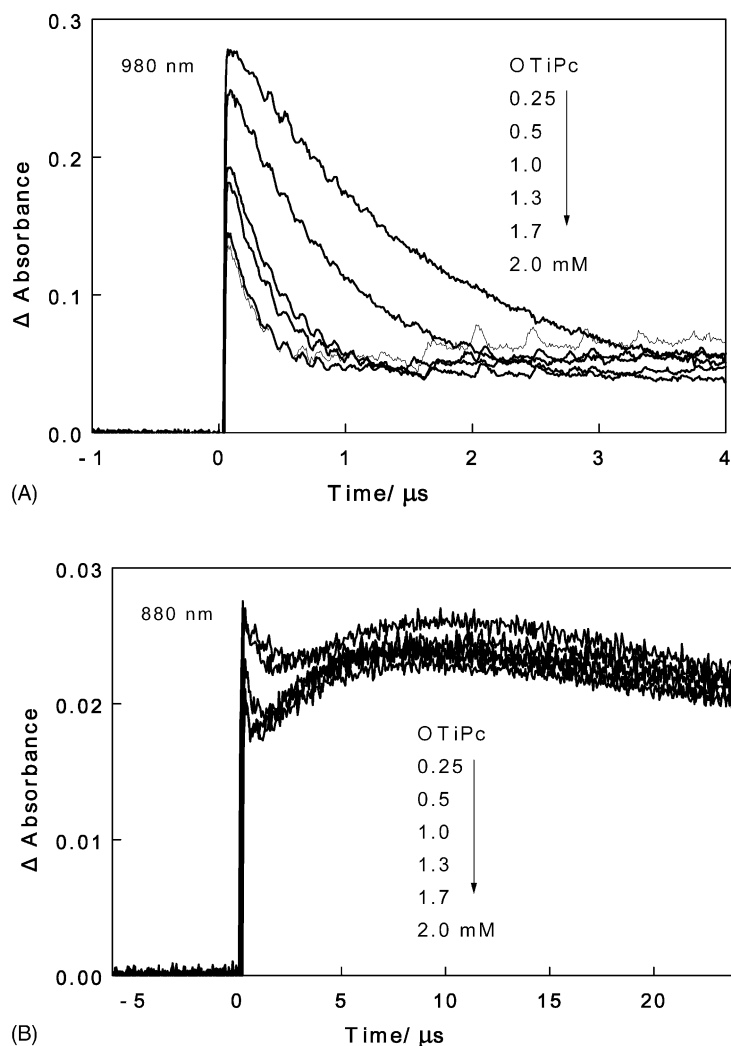


Fig. 8. Time profiles observed after laser excitation of C_{70} (0.1 mM) in the presence of OTiPc (0.25–2.0 mM) in deaerated PhCN: (upper panel) the decays of ${}^3\text{C}_{70}^*$ at 980 nm in short time-scale, and (lower panel) the rises of $\text{OTiPc}^{\bullet+}$ at 880 nm in long time-scale.

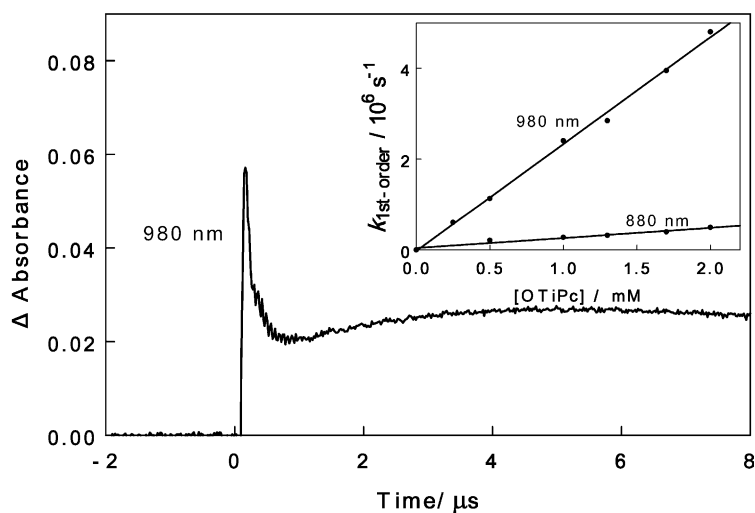


Fig. 9. Time profiles observed after nanosecond laser excitation of C_{70} (0.1 mM) in the presence of OTiPc (2 mM) in deaerated PhCN at 980 nm. Inset: Pseudo-first-order plots.

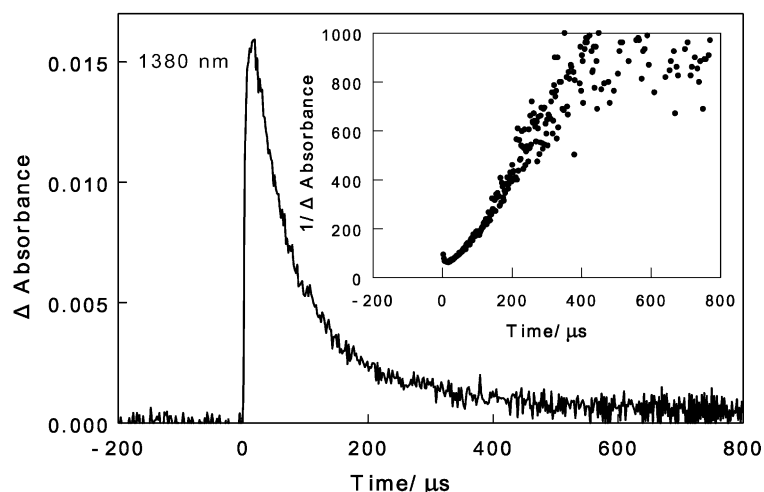
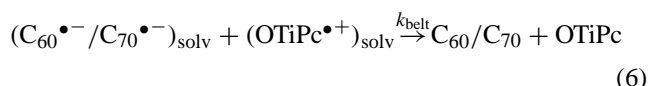


Fig. 10. Decay of $C_{70}\bullet^-$ at 1380 nm in long time-scale in the presence of equimolar of $OTiPc\bullet^+$ in PhCN.

controlled limit ($k_{diff} = 5.3 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$), but the k_{belt} value for $C_{60}\bullet^-$ is slightly higher than k_{diff} :



4. Conclusion

The photoexcited states of TiOPc were examined. In the presence of C_{60} and C_{70} , energy and electron transfer processes from the excited triplet states of C_{60} and C_{70} to TiOPc were confirmed the laser flash photolysis observing the transient absorption bands in the near-IR region in toluene and in PhCN, respectively. For C_{70} , slower formation of the ion radicals after the rapid decay of the triplet excited states of C_{70} was observed, suggesting the presence of the intermediates (i.e. triplet exciplex) for electron transfer. Second-order back electron transfer of free radical ions in PhCN was also confirmed.

Acknowledgements

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