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Bull. Chem. Soc. Jpn., **72**, 2103—2109 (1999)

9,10-Dicyanoanthracene-Photosensitized Elimination of 1,1′-Binaphthalene from Dinaphthyl Methylphosphonate and Trinaphthyl Phosphate through the Termolecular Interaction in the Singlet Excited State

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(Received April 9, 1999)

9,10-Dicyanoanthracene (DCA) photosensitized intramolecular binaphthalene elimination of dinaphthyl methylphosphonates and trinaphthyl phosphates occurred through an exciplex between the singlet excited DCA (^{1}DCA*) and the naphthyl group of dinaphthyl methylphosphonate and trinaphthyl phosphates in tetrahydrofuran, 1,4-dioxane, and benzene. Diethyl mononaphthyl phosphates also formed an exciplex with ^{1}DCA*, while no reaction occurred. The binaphthalene elimination occurs via the intramolecular quenching of the exciplex by the second naphthyl group of dinaphthyl methylphosphonate or trinaphthyl phosphate, while the exciplex between the mononaphthyl phosphate and ^{1}DCA* decays to give the starting compounds. The lifetime of the exciplex between the trinaphthyl phosphate and ^{1}DCA* was shorter than that between the mononaphthyl phosphate and ^{1}DCA*. The termolecular interaction (teraromatic groups interaction) of ^{1}DCA* and two naphthyl groups of dinaphthyl methylphosphonate or trinaphthyl phosphate is suggested by the emission observation of the termolecular interaction of two naphthyl groups of trinaphthyl phosphate and 1,4-dicyanobenzene in the singlet excited state during the quenching of the intramolecular excimer of dinaphthyl methylphosphonate or trinaphthyl phosphates by 1,4-dicyanobenzene. The O–P(O)–O spacer is necessary for the binaphthalene elimination during the termolecular interaction in the singlet excited state.

Photochemical reactions of organophosphorus compounds have been widely investigated by many groups.[1—4] Especially, the photochemical reactions of aryl phosphates as caged compounds have been studied with respect to biochemical processes.[2] In contrast with the direct photoirradiation, little is known for the photosensitized reactions of the organophosphorus compounds except for triplet acetone-sensitized photoreactions.[3] We wish to report here an intramolecular binaphthalene elimination from dinaphthyl methylphosphonates and trinaphthyl phosphates through the π-interaction between 9,10-dicyanoanthracene (DCA) and the two naphthyl groups of the methylphosphonates and the phosphates in the singlet excited state. There are many examples of DCA-photosensitized reactions of aromatic compounds such as isomerizations, rearrangements, and cycloreversions involving both intramolecular bond dissociation and formation through the exciplexes in non-polar solvents.[5] On the other hand, few studies have been reported on the DCA photosensitized reactions of organophosphorus compounds. This work is on a DCA-photosensitized reaction involving the intramolecular binaphthalene elimination from the dinaphthyl methylphosphonates and trinaphthyl phosphates. In this paper, the π-interaction of two naphthyl groups and a singlet excited sensitizer is called the termolecular interaction, although the teraromatic groups interaction or intratermolecular interaction may be correct.

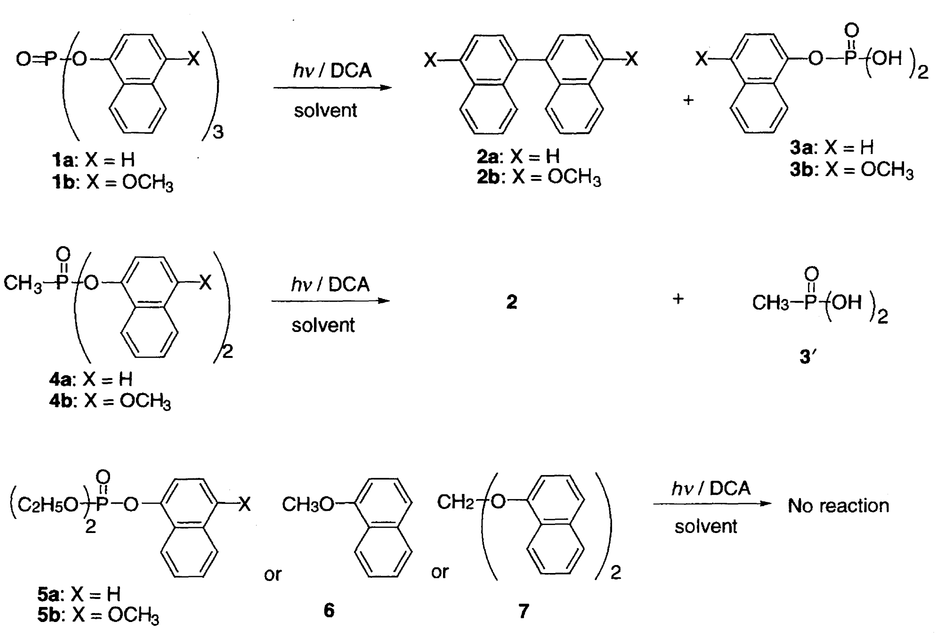
Results

Photoreaction. An argon-saturated tetrahydrofuran containing 5.0×10^{-5} mol dm^{-3} of DCA and 1.0×10^{-2} mol dm^{-3} of tri-1-naphthyl phosphate **1a** was irradiated with a high-pressure Hg lamp (cutoff <355 nm) for 1 h to give 1,1′-binaphthalene **2a** and 1-naphthyl dihydrogenphosphate **3a** in a 1 : 1 ratio with a quantum yield of 2.2×10^{-3} (Table 1).[6] On the photoirradiation of the mixture of DCA and tris(4-methoxy-1-naphthyl) phosphate **1b** in the same manner described above, 4,4′-dimethoxy-1,1′-binaphthalene **2b** and (4-methoxy-1-naphthyl) dihydrogenphosphate **3b** were produced in a 1 : 1 ratio with a quantum yield of 4.0×10^{-3} (Scheme 1). The photoirradiation of the mixture of di-1-naphthyl methylphosphonate **4** and DCA in tetrahydrofuran gave **2** and

Table 1. Quantum Yield of **2** on the DCA-Photosensitized Reaction of **1** and **4**[a)]

Solvent	$\Phi_2/10^{-3}$			
	1a	**1b**	**4a**	**4b**
Tetrahydrofuran	2.2	4.0	1.2	1.4
1,4-Dioxane	0.8	3.8	0.8	0.8
Benzene	0.6	2.0	0.5	0.6

a) Photoirradiation was carried out on a mixture of 5.0×10^{-5} mol dm^{-3} of DCA and 1.0×10^{-2} mol dm^{-3} of **1** or **4**.



solvent: THF, 1,4-dioxane, and benzene

Scheme 1.

methylphosphonic acid **3′**. The formation of **2** also occurred on the photoirradiation of a mixture of DCA and **1** or **4** in 1,4-dioxane and benzene, which are less polar solvents than tetrahydrofuran. On the other hand, no reaction occurred with the photoirradiation of a mixture of DCA and diethyl 1-naphthyl phosphate **5** ((1.0—3.0)$\times 10^{-2}$ mol dm^{-3}) in tetrahydrofuran, 1,4-dioxane, and benzene. It is clearly shown that the binaphthalene elimination occurs intramolecularly in **1** and **4**. No product was obtained during the photoirradiation of mixtures of DCA-1-methoxynaphthalene **6** and DCA-bis(1-naphthloxy)methane **7**.

Products **2** and **3** were also obtained in the photolysis of the mixture of **1** and 1,4-dicyanonaphthalene (DCN) in tetrahydrofuran where DCN was used in the place of DCA.

Fluorescence Quenching. Fluorescence quenching of DCA or DCN by **M** (= **1**, **4**, and **5**) was done. The DCA fluorescence was quenched by **M** in tetrahydrofuran, 1,4-dioxane, and benzene to form a new emission band around 500—650 nm with an isoemissive point. The emission maxima (λ_{max}) of the new emission band shifted to a longer wavelength with an increase of the solvent polarity (Table 2). The similar emission shift was observed in the DCA fluorescence quenching by naphthalene. Therefore, the new emission band is reasonably assigned to an exciplex, ^{1}(**M**/DCA)*, between the naphthyl group of **M** and ^{1}DCA*. Figure 1 shows spectral changes of the DCA fluorescence upon the addition of **1a** and **5a** in benzene. Fluorescence lifetimes of ^{1}(**1a**/DCA)* and ^{1}(**5a**/DCA)* were measured by the single photon counting technique (Table 2). The lifetime of ^{1}(**1a**/DCA)* was about 30% shorter than that of ^{1}(**5a**/DCA)*. It is indicated that ^{1}(**1a**/DCA)* is intramolecularly quenched by the second naphthyl group of **1a** to give **2a**. The DCA fluorescence was also quenched by **6** and **7** to form an exciplex ^{1}(**6**/DCA)* and ^{1}(**7**/DCA)* with an emission peak at λ_{max} = 550 and 570 nm, and lifetime of 52 and 33 ns, respectively (Table 2). The DCN fluorescence was also quenched by **1a** and **5a** with formation of an exciplex emission band around 450—600 nm in tetrahydrofuran, 1,4-dioxane, and benzene with an isoemissive point (Fig. 2).

5a is a mononaphthyl ester while **1a** is trinaphthyl ester. The fluorescence spectrum of **5a** was observed at λ_{max} = 338 nm, while the fluorescence due to the intramolecular excimer emission of **1a** was observed at λ_{max} = 410 nm (Fig. 3). The fluorescence quenchings of **5a** and **1a** were done by 1,4-dicyanobenzene (DCB). The **5a** fluorescence was quenched by DCB to form an exciplex emission band at 430 nm with an

Table 2. Emission Maxima (λ_{max}) and Lifetimes ($\tau_{DCA \cdot M}$) of Exciplexes of ^{1}DCA* and **M** (**M** = **1a**,**5a**,**6** and **7**)[a)]

Solvent	**1a**		**5a**		**6**		**7**	
	λ_{max}/ nm	τ/ ns	λ_{max}/ nm	τ/ ns	λ_{max}/ nm	τ/ ns	λ_{max}/ nm	τ/ ns
Tetrahydrofuran	560	40	567	49	b)	b)	b)	b)
1,4-Dioxane	544	44	558	54	b)	b)	b)	b)
Benzene	540	27	555	36	588	52	586	33

a) The fluorescence lifetimes were measured in argon-saturated solutions, [DCA] = 5.0×10^{-5} mol dm^{-3}, [**M**] = 1.0×10^{2} mol dm^{-3}. b) Not determined.

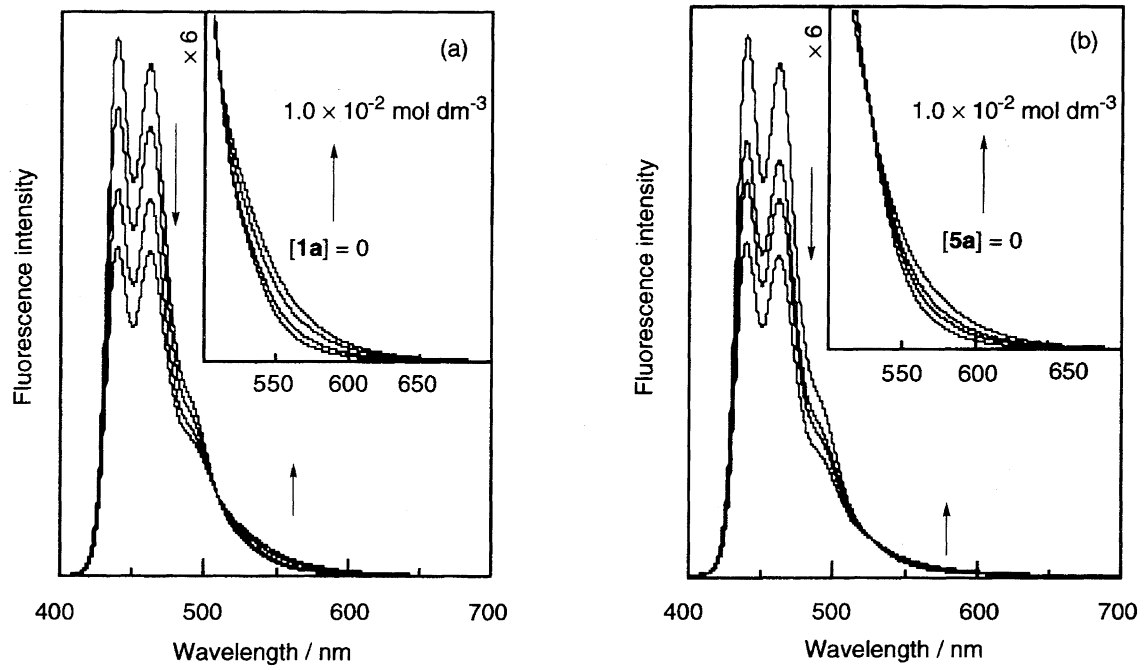


Fig. 1. Spectral changes of the DCA (5.0×10^{-5} mol dm^{-3}) fluorescence at 375-nm excitation with additions of (a) (0—1.0) $\times 10^{-2}$ mol dm^{-3} of **1a** and (b) (0—1.0) $\times 10^{-2}$ mol dm^{-3} of **5a** in benzene. Insets, fluorescence intensities were extended by six times in the region of 500—700 nm.

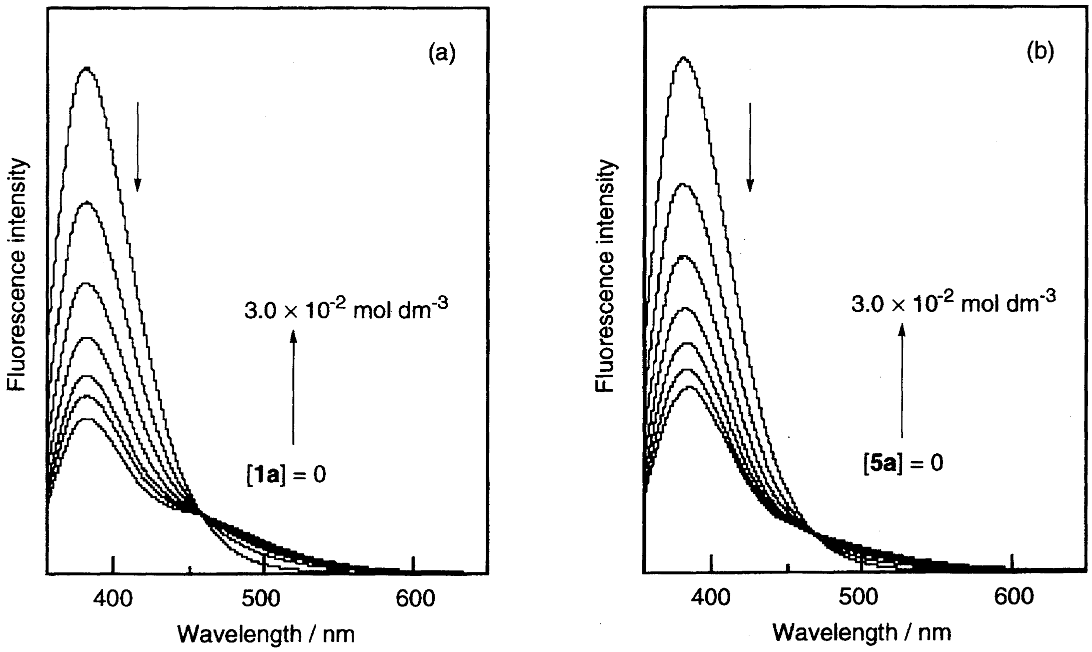


Fig. 2. Spectral changes of DCN (1.0×10^{-4} mol dm^{-3}) fluorescence 355-nm excitation with additions of (a) (0—3.0) $\times 10^{-2}$ mol dm^{-3} of **1a** and (b) (0—3.0) $\times 10^{-2}$ mol dm^{-3} of **5a** in tetrahydrofuran.

isoemissive point at 385 nm (Fig. 4a). The intramolecular excimer emission of **1a** was also quenched by addition of DCB with formation of a new emission band around 475—600 nm with a peak at 510 nm and an isoemissive point at 475 nm (Fig. 4b). This new emission band is obviously assigned to the termolecular complex, ^{1}[Np/Np/DCB]*, of the two naphthyl groups (Np) of **1a** and DCB in the singlet excited state.

The quantum yield of **2a** formation decreased with an increase of the DCB concentration on the photoirradiation of the mixture of **1a** and DCB in tetrahydrofuran, 1,4-dioxane, or benzene. The **2a** fluorescence was quenched in 45% yield, and the quantum yield of **2a** was decreased to 50% in the presence of 2.0×10^{-2} mol dm^{-3} of DCB in tetrahydrofuran. Therefore, ^{1}[Np/Np/DCB]* decays completely to **1a** and DCB.

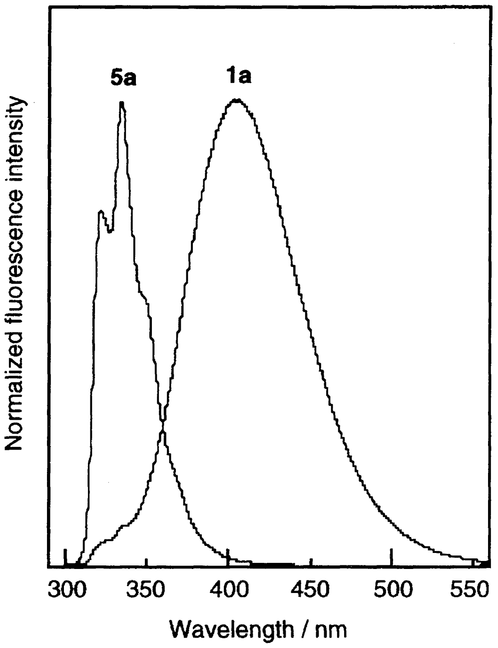


Fig. 3. Fluorescence spectra of **1a** (1.0×10^{-4} mol dm^{-3}) and **5a** (1.0×10^{-4} mol dm^{-3}) at 280-nm excitation in tetrahydrofuran. Fluorescence intensities were normalized at the peaks.

Discussion

The fluorescence of DCA was quenched by **1**, **4**, and **5**—**7**. The $\lambda_{\max}$ values of exciplex emission bands are summarized in Table 2. It is notable that $\lambda_{\max}$ of the exiplex emission band of ^{1}(**1a**/Sens)* (Sens = DCA or DCN) shifted to a shorter wavelength than that of ^{1}(**5a**/Sens)*, while $\lambda_{\max}$ of the exiplex emission band of ^{1}(**7**/DCA)* slightly shifted to a longer wavelength than that of ^{1}(**6**/DCA)*. Since the shorter wavelength of the exciplex emission band corresponds to the higher energy of exciplexes with less charge separation character, the reverse shift of the exciplex emission band between **1a** and **5a** and between **7** and **6** is a response to the character of the O–P(O)–O and O–CH$_2$–O spacers between the two naphthyl groups. It is suggested that the exciplex formation between the naphthyl group and ^{1}Sens* is sterically hindered in ^{1}(**1a**/Sens)* compared with that in ^{1}(**5a**/Sens)* because of the second naphthyl group linked by the O–P(O)–O spacer. The formation of **2** occurred in the DCA-photosensitized reaction of **1** and **4**, but not in **5**. Therefore, it is seen that the formation of **2** proceeds through the termolecular interaction of the two naphthyl groups of **1** and **4** and ^{1}DCA*. Scheme 2 shows a plausible mechanism for the formation of **2a** involving the π-interaction between ^{1}DCA* and two naphthyl groups of **1a**, which causes the elimination of **2a** together with the formation of the corresponding residue **3** in the intramolecular quenching of ^{1}(**1a**/DCA)* by the second naphthyl group of **1a**.

We have previously reported that the *ipso*-coupling produces **2** through the intramolecular excimers of **1** and **4**.[4b] Such π-interactions of the two naphthyl groups in the singlet excited state are found to be necessary for the elimination of **2**. Therefore, the termolecular interaction of the two naphthyl groups and ^{1}DCA* and the *ipso*-coupling between the two naphthyl groups are assumed for the elimination of **2** in the DCA-photosensitized reaction of **1** and **4**.[7]

The termolecular interaction is suggested by the shorter lifetime of ^{1}(**7**/DCA)* than that of ^{1}(**6**/DCA)*. Therefore, ^{1}(**7**/DCA)* must be quenched by the second naphthyl group of **7** via the termolecular interaction. It is well known that a spacer having three atoms is the most effective in the in-

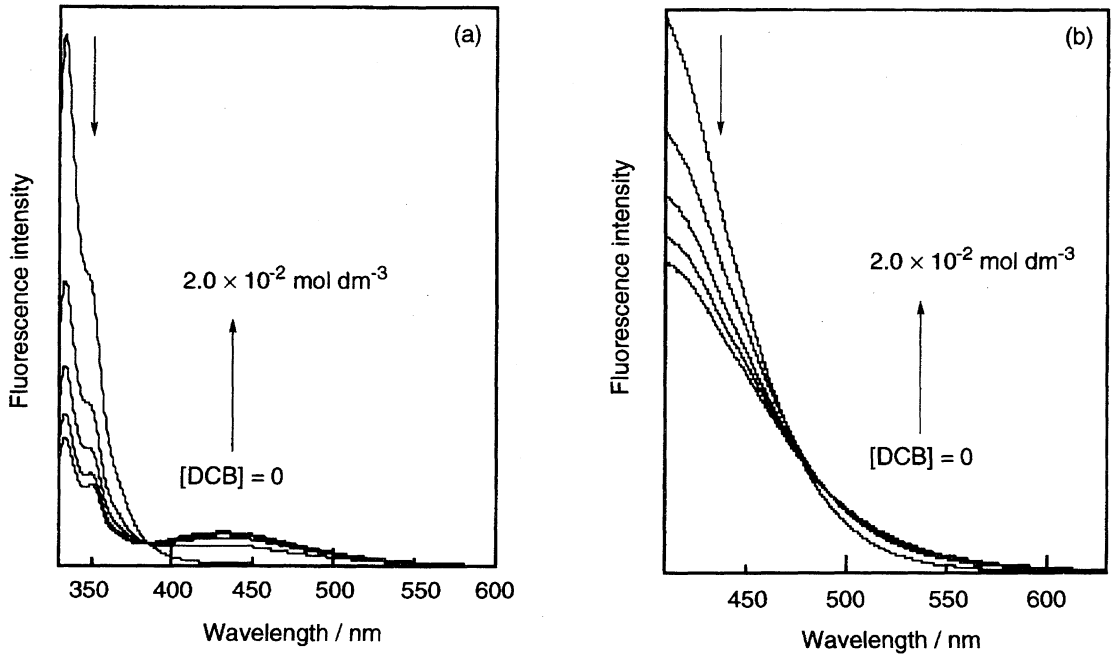
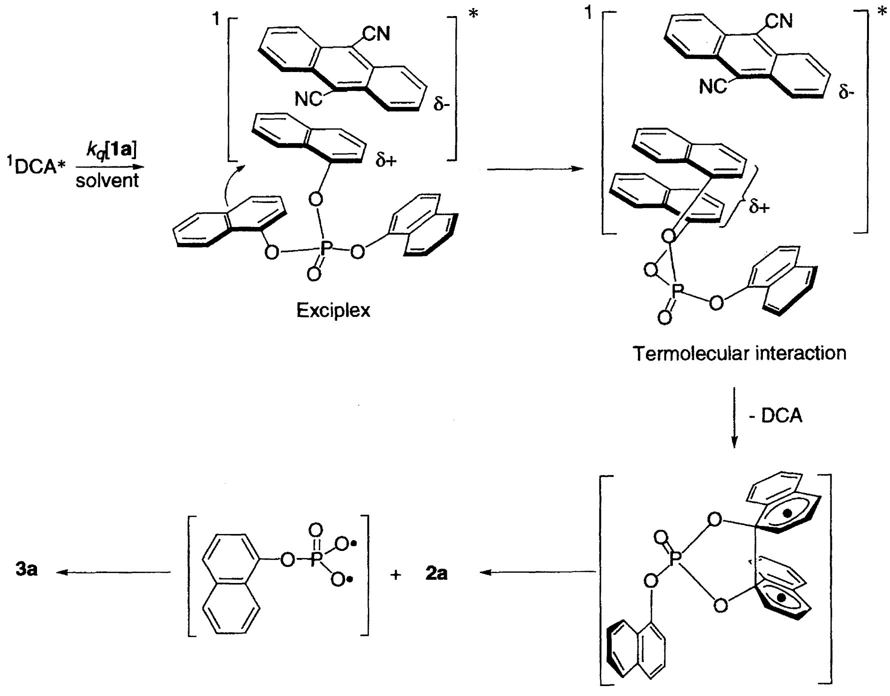


Fig. 4. Fluorescence-spectral changes (a) 1.0×10^{-4} mol dm^{-3} of **5a** and (b) 1.0×10^{-4} mol dm^{-3} of **1a** at 320-nm excitation with additions of (0—2.0) $\times10^{-2}$ mol dm^{-3} DCB in tetrahydrofuran.



Scheme 2.

tramolecular π-interaction between two aryl groups linked with the spacer in the excited state.[8]

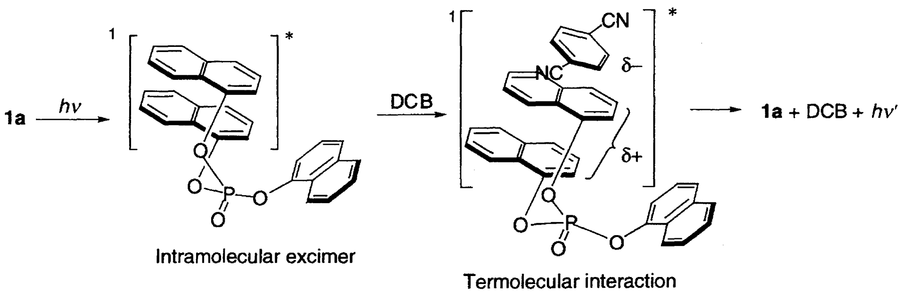
The termolecular interaction of a sensitizer and two naphthyl groups of **1** and **4** was examined by the quenching of excimer emission of **1** and **4** by DCB. It has been established that a termolecular complex (triplex) emission of ^{1}[naphthalene/naphthalene/DCB]* is observed at 450—590 nm with a peak at 480 nm and an isoemissive point at 450 nm with excitation at 313 nm tuned to the naphthalene absorption in nonpolar solvent.[9] The similar emission of ^{1}[Np/Np/DCB]* was observed at 475—600 nm with a peak at 510 nm in the fluorescence measurement of **1a**-DCB mixture. Therefore, the termolecular interaction ^{1}[Np/Np/DCA]* is also suggested to be involved in the quenching of ^{1}[**1a**/DCA]* by the second naphthyl group of **1a**.

Table 1 shows the quantum yields of **2** in the DCA-photosensitized reactions of **1** and **4** in tetrahydrofuran, 1,4-dioxane, and benzene. The larger quantum yield of **2** from **1** than that from **4** can be explained by the number of naphthyl groups of **1** and **4**. It is indicated that the intramolecular quenching of ^{1}(**1**/DCA)* by the second naphthyl group of **1** occurs more easily than that of ^{1}(**4**/DCA)*, because **1** and **4** have three and two naphthyl groups, respectively.

Since the polarization of the exciplex increases with an increase of the solvent polarity, the formation of **2** occurs more efficiently through ^{1}(**M**/DCA)* with a more charge-separated character, $^1(\mathbf{M}^{\delta+}/\mathrm{DCA}^{\delta-})^*$. We have recently reported that the photoirradiation of the mixture of DCA and **1a** or **4a** in argon-saturated acetonitrile, in which an electron transfer occurs to generate the radical anion of DCA (DCA$^{\bullet -}$) and a radical cation of **1a** or **4a** ($\mathbf{1a}^{\bullet +}$ or $\mathbf{4a}^{\bullet +}$), and that the radical cation of **2a** ($\mathbf{2a}^{\bullet +}$) is eliminated to give **2a** in the quantum yield of 1.3×10^{-3} from **1a** and in 1.0×10^{-3} from **4a**, respectively, via an electron transfer from DCA$^{\bullet -}$ to $\mathbf{2a}^{\bullet +}$.[4c,4d] These values are smaller than 1—4×10^{-3} in the DCA-photosensitized intramolecular binaphthalene elimination in tetrahydrofuran, but slightly larger than 0.5—0.8×10^{-3} in 1,4-dioxane and benzene (Table 1). This fact indicates that the elimination of **2a** occurs more efficiently in ^{1}(**1a**/DCA)* or ^{1}(**4a**/DCA)* than in $\mathbf{1a}^{\bullet +}$ or $\mathbf{4a}^{\bullet +}$. The appropriate charge separation in $^1(\mathbf{M}^{\delta+}/\mathrm{DCA}^{\delta-})^*$ may be important in the efficient elimination of **2a**.

The elimination of **2a** occurs via the termolecular interaction in the quenching of ^{1}(**1a**/DCA)* by the second naphthyl group and the *ipso*-coupling between the two naphthyl groups, while ^{1}(**7**/DCA)* decays to give the starting compounds via the termolecular interaction. Therefore, the elimination of **2a** is caused by the character of the O–P(O)–O spacer between the two naphthyl groups during the termolecular interaction. The occurrence of the binaphthalene elimination from ^{1}(**1**/DCA)* and ^{1}(**4**/DCA)* is probably attributed to the electron withdrawing character of the P=O group of the O–P(O)–O spacer of **1** and **4** which may be further increased in the excited state.

We can discuss the structures of the excimers, exciplexes, and termolecular interactions involving **M**, $^1\mathbf{M}^*$, and ^{1}Sens* on the basis of the reactivity of the elimination of **2** by the *ipso*-coupling. Since the intramolecular excimer of $^1\mathbf{M}^*$ (^{1}[Np/Np]*, **M** = **1** and **4**) underwent the elimination of **2**, we have proposed two types of structures with full- and partial overlaps of two naphthyl groups in which two *ipso*-positions are closer to each other for the elimination of **2**.[4b,10] On the other hand, the elimination of **2** did not occur in the intermolecular excimer of $^1\mathbf{M}^*$ and **M** ($^1\mathbf{M}_2^*$, **M** = **5**). It is suggested that $^1\mathbf{M}_2^*$ has a structure with the overlap of two naphthyl groups of $^1\mathbf{M}^*$ and **M** of which two *ipso*-positions are placed in the reverse. Approaching of the two *ipso*-positions of **M** must occur in the π-interaction of



Scheme 3.

three chromophores in ^{1}[Np/Np/Sens]* for the elimination of **2a** during the intramolecular quenching of the exciplex ^{1}(**M**/Sens)* by the second naphthyl group of **M** (**M** = **1** and **4**) as shown in Scheme 2. On the other hand, the elimination of **2** did not occur in ^{1}[Np/Np/DCB]* during the quenching of the intramolecular excimer of ^{1}**M*** (^{1}[Np/Np]*) by DCB. Lengthening of the two *ipso*-positions of **M** is assumed to occur in the π-interaction of ^{1}[Np/Np/DCB]* as shown in Scheme 3. Such structural difference may influence the non-emissive and emissive character of ^{1}[Np/Np/Sens]* and ^{1}[Np/Np/DCB]*, respectively.

In conclusion, the DCA-photosensitized reactions of **1** and **4** in tetrahydrofuran, 1,4-dioxane, and benzene gave **2** through the termolecular π-interaction of two naphthyl groups and DCA in the singlet excited state and then through the *ipso*-coupling during the quenching of the exciplex between ^{1}DCA* and a naphthyl group of **1** and **4** by the second naphthyl group. Although the similar exciplex between ^{1}DCA* and a naphthyl group of **5**, **6**, and **7** formed, but no reaction occurred. These facts indicate that the elimination of **2** is characteristic for **1** and **4** which have two naphthyl groups linked with the O–P(O)–O spacer. The termolecular interaction of ^{1}DCA* and the two naphhthyl groups of **1** and **4** are found to be necessary for the occurrence of the elimination of **2**, and suggested by the observation of the emission of ^{1}[Np/Np/DCB]*.

Experimental

Materials. The substrates **1**, **4**, and **5** were prepared according to the methods described in the literature.[11] The reaction products were isolated by silica gel column chromatography and analyzed by GC-MS, and ^{1}H and ^{13}C NMR spectroscopies. Authentic samples were synthesized to identify the reaction products by GC and HPLC. The analytical data on these materials are shown in our previous paper.[4b] 1-Methoxynaphthalene **6** purchased from Tokyo Chemical Industry Co. was used after distillation. Bis(1-naphthoxy)methane **7** was also prepared by the method described in the literature.[12]

General. The photoirradiation was done using a 300 W high pressure Hg lamp EHBW-300 with a 10 mm thick $BiCl_3$/HCl filter (cutoff < 355 nm) at ambient temperature. The mixture solution was put in a Pyrex tube with bubbling argon to remove dissolved air. The reaction products were identified by GLC and HPLC on a Shimadzu GC-12A instrument using a 2% Silicon OV-17 column and a Shimadzu LC-10AS instrument with a Shiseido Capcell Pak C18 AG 120 column, respectively. The steady-state fluorescence spectra were recorded on a Hitachi 850 type fluorescence spectrometer. The fluorescence spectra of DCA and DCN were measured at a concentration of 5.0×10^{-5} mol dm^{-3} and 1.0×10^{-4} mol dm^{-3}, respectively. The spectral changes of **1a** by addition of DCB were observed at a concentration of 1.0×10^{-4} mol dm^{-3}. The fluorescence lifetime was measured by the single photon counting method using a Horiba Nase-500 instrument. The quantum yields of **2** were measured using benzophenone and benzhydrol system as an actimometer.[13]

We thank Professor Setsuo Takamuku, Professor Yoshiki Okamoto, and the late Professor Reizo Dohno for their helpful discussion, Dr. Akito Ishida, Mrs. Sachiko Tojo, and Miss Tomoko Hashikawa for their help in the experiments. This work was partly supported by a Grant-in-Aid Nos. 09226223, 10132237, 09450319, and 09875209 from the Ministry of Education, Science, Sports and Culture for T. M.

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6 The quantum yields of **2** were determined from the amount of **2** formation and absorbed photons. The conversions of **1** and **4** and chemical yields of **2** were not determined because of the low

values, less than a few %. Since **2** was the main product of the photoreactions, the stoichiometry of the consumption of **1** and **4** and yield of **2** seems to be sufficiently high as a photoreaction.

7 No transient absorption of radical ions such as **1** radical cation and DCA radical anion was observed during 355-nm laser flash photolysis of **1** and DCA in benzene. Therefore, the electron transfer between $^1\mathrm{DCA}^*$ and **1** is not involved in the binaphthalene elimination in benzene.

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