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# Solvent-dependent intramolecular exciplex formation of *N*-benzyl-*N*-methylamine and *N*,*N*-dibenzylamine

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## Abstract

The fluorescence properties of *N*-benzyl-*N*-methylamine (BMA) and *N*,*N*-dibenzylamine (DBA) have been studied in several solvents of different polarities to examine the nature of the intramolecular interaction between phenyl and amino groups. BMA shows the fluorescence of the phenyl group and DBA shows the fluorescence of the phenyl group and the intramolecular excimer emission in non-polar solvents. New fluorescence bands appeared in the long-wavelength region for BMA and DBA in certain polar solvents. These bands showed red-shifts with increasing solvent polarity. Upon the addition of trichloroacetic acid to the BMA solution in acetonitrile (AN) the intensity of the long-wavelength band decreased and the intensity of the fluorescence of the phenyl group increased. The intensity of the long-wavelength band was low and that of phenyl group fluorescence was high in alcohols compared with those in THF and AN. These findings are caused by the loss of the electron-donation ability of the amino group owing to the protonation and hydrogen-bonding with the alcohol molecule. Similar spectral changes were observed for DBA. These results indicate that the long-wavelength bands can be attributed to the intramolecular exciplexes between phenyl and amino groups. The formation of the complex between the exciplex and a dipolar molecule was observed in THF–AN mixtures.

*Keywords:* Exciplex; Solvent effects; Fluorescence; Protonation; Polar effect

## 1. Introduction

It is well known that the fluorescence of aromatic compounds shows quenching and exciplex formation in the presence of tertiary aliphatic amines in non and slightly polar solvents [1–11]. The intensity and position of the exciplex emission are highly solvent-dependent, and a large red shift and decrease in the intensity were observed with increasing solvent polarity [12–14]. Formation of intramolecular exciplexes between phenyl and amino groups was investigated in several ω-pheny-α-(*N*,*N*-dimethylamino)alkanes ($Ph–(CH_2)_n–N(CH_3)_2$) by Schryver and co-workers [15–18], and intramolecular exciplex emissions were observed for $n = 2–5$ and 11.

We reported previously that the effects of the addition of alcohols on the intra- and intermolecular exciplex systems (4-phenyl-1-*N*,*N*-dimethylaminobutane, toluene-TEA, naphthalene-TEA and styrene-TEA systems) can be separated into solvent polarity and hydrogen-bonding effects [19–23].

Sizuka et al. [24] studied the intramolecular fluorescence quenching of phenylalkylamines ($Ph–(CH_2)_n–NH_2$: $C_1$, $C_2$ and $C_3$ indicating the number of methylene unit) and they found that the quenching of normal fluorescence involving intramolecular electron transfer and subsequent proton transfer from an amino group to an excited phenyl group is significantly dependent on the number of methylene units, solvent polarity and temperature (solvent viscosity). In non-polar and weakly polar solvents static quenching resulting from a sandwich-like or holding structure occurs markedly for $C_3$, but not for $C_1$ and $C_2$. In strongly polar solvents dynamic quenching takes place in the order of $C_1$, $C_3$ and $C_2$, and this is thought to be due to a charge-transfer interaction. Therefore, the charge-transfer interaction between phenyl and amino groups in $C_1$ must be large. The lack of exciplex emission of this compound is due to the fast proton transfer in the excited state. Although much information about the intramolecular interaction of an excited phenyl group with an amino group was obtained by using $C_1$, $C_2$ and $C_3$, these compounds do not give the exciplex emission.

El-Bayoumi co-workers [25–27] reported that intramolecular exciplex fluorescence of 2-naphthylmethylamine has been observed in polar solvents. They also observed a decrease in the

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ratio of intensity of the exciplex emission to that of monomer fluorescence and a red shift of exciplex emission as the solvent polarity is increased. The quenching of exciplex emission was enhanced in polar hydroxyl solvents. This is the first observation of the emission from the intramolecular exciplex for aminomethyl aromatic compounds.

Recently our preliminary experiments indicated that the fluorescence of the phenyl group and the intramolecular excimer emission were observed for *N*,*N*-dibenzylamine (DBA) in cyclohexane, while in polar solvents such as acetonitrile and some alcohols, the third emission band appeared in the longer wavelength region. The shape of the emission spectrum of DBA in AN was independent of the DBA concentration. It is possible that this new band is attributed to an intramolecular exciplex between phenyl and amino groups.

In this study we report the occurrence of exciplex emission of DBA and *N*-benzyl-*N*-methylamine (BMA). The dependence of the position and intensity of exciplex emission on the polarity of solvent is discussed. The effects of alcohols and acids on the emission spectra of DBA and BMA are also discussed. The fluorescence of DBA and BMA is very weak compared with that of toluene and 1,3-diphenylpropane. This shows that the intramolecular quenching of the fluorescence of phenyl group by the amino group occurs effectively in DBA and BMA. However, since the fluorescence of DBA and BMA is weak, the interaction of the excited phenyl group with the amino group has not been studied in detail for these compounds. Examination of the behavior of exciplex emission can give detailed information about the interaction of the excited phenyl group and the amino group in DBA and BMA.

## 2. Experimental

All reagents were obtained from commercial sources. DBA and BMA (extra pure reagent) were purified by trap-to-trap distillation under vacuum. Methanol (MeOH), ethanol (EtOH), butanol (BuOH), cyclohexane (CH), tetrahydrofuran (THF) and acetonitrile (AN) were of spectroscopic grade and were used as supplied. 2,2,2-Trifluoroethanol (TFE) and trifluoroacetic acid (TCAA) (extra pure grade) were used as available.

The steady-state fluorescence spectra were measured using a Simadzu spectrophotometer, model RF-5300PC (band pass, typically 10 nm) at 20 °C. The solutions were degassed by four freeze-pump-thaw cycles immediately prior to measurement for most cases. The effects of TCAA on the emission spectra of BMA and DBA were measured immediately after the preparation of the solutions without degassing, because the spectra were slightly dependent upon time in the presence of TCAA. The emission spectra of DBA and BMA in the presence of oxygen were similar in shape but were weaker in intensity than those in the absence of oxygen. Excitation was effected at 255 nm in each case, a wavelength at which the radiation is nearly exclusively absorbed by a phenyl group of DBA and BMA. The concentration of DBA and BMA was chosen to be $1.44 \times 10^{-3}$ mol dm$^{-3}$ to avoid intermolecular interaction; the shapes and positions of the emission bands did not depend on concentrations of BMA and DBA below $5.0 \times 10^{-3}$ mol dm$^{-3}$. The quantum yields of the fluorescence of DBA and BMA in CH and AN were estimated using the fluorescence of toluene as a standard ($\phi = 0.14$ in CH and 0.10 in AN [24]). The values obtained were $\phi = 0.0027$ (BMA) and 0.0030 (DBA) in CH and $\phi = 0.0014$ (BMA) and 0.0011 (DBA) in AN.

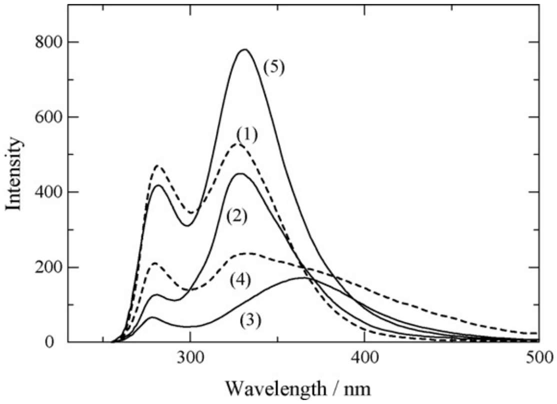

Fig. 1. Emission spectra of DBA in different solvents: [DBA] = 1.44 × $10^{-3}$ mol dm$^{-3}$; (1) CH, (2) THF, (3) AN, (4) EtOH, and (5) TFE.

## 3. Results and discussion

The absorption spectrum of BMA is comparable to that of an equimolar mixture of toluene and methylpropylamine, and that of DBA is consistent with that of a 2:1 mixture of toluene and methylpropylamine. As pointed out by Sizuka et al. [24] for phenylalkylamines, no significant ground state interaction exists between two chromophores in BMA and DBA.

The emission spectrum of DBA in CH was found to consist of two component bands (band A at 279 nm and band B at 320 nm). As previously reported, bands A and B were assigned to the fluorescence of the phenyl group and the fluorescence from the intramolecular excimer between the two phenyl rings [28].

Fig. 1 shows the emission spectra of DBA in several different solvents. In polar solvents the third emission band (band C) was observed in the long-wavelength region. The wavelength of the maximum of band C was sensitive to the polarity of the solvent. Band C showed a red-shift in the order of BuOH, EtOH and MeOH, but this band disappeared in TFE. This band has not been reported yet. The shape of the emission spectrum of DBA

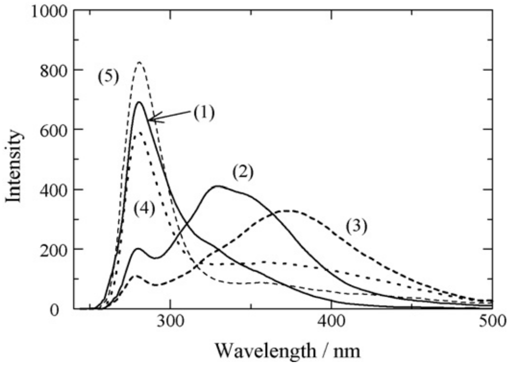

Fig. 2. Emission spectra of BMA in different solvents: [BMA] = 1.44 × $10^{-3}$ mol dm$^{-3}$; (1) CH, (2) THF, (3) AN, (4) BuOH, and (5) MeOH.

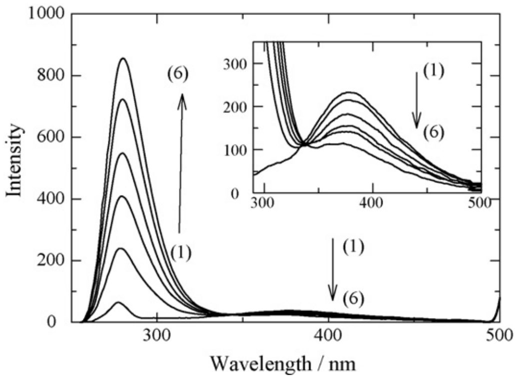

Fig. 3. Emission spectra of BMA in AN-TCAA mixtures: [BMA] = $2.88 \times 10^{-4}$ mol dm$^{-3}$; [TCAA] are: (1) 0 mol dm$^{-3}$, (2) $5.80 \times 10^{-5}$ mol dm$^{-3}$, (3) $1.15 \times 10^{-4}$ mol dm$^{-3}$, (4) $1.73 \times 10^{-4}$ mol dm$^{-3}$, (5) $2.30 \times 10^{-4}$ mol dm$^{-3}$, and (6) $2.88 \times 10^{-4}$ mol dm$^{-3}$. Inset: [BMA] = $1.44 \times 10^{-3}$ mol dm$^{-3}$; [TCAA] are: (1) 0 mol dm$^{-3}$, (2) $2.88 \times 10^{-4}$ mol dm$^{-3}$, (3) $5.76 \times 10^{-4}$ mol dm$^{-3}$, (4) $8.64 \times 10^{-4}$ mol dm$^{-3}$, (5) $1.15 \times 10^{-3}$, and (6) $1.44 \times 10^{-3}$ mol dm$^{-3}$.

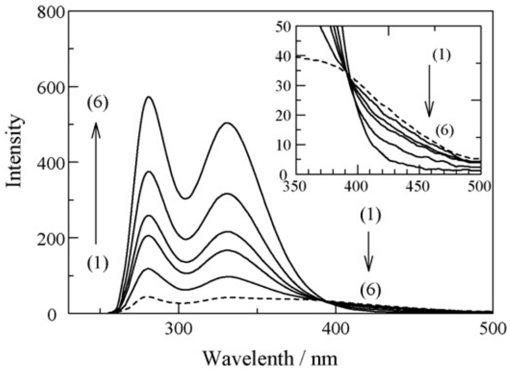

Fig. 4. Emission spectra of DBA in EtOH-TCAA mixtures: [DBA] = $1.44 \times 10^{-4}$ mol dm$^{-3}$; [TCAA] are: (1) 0 mol dm$^{-3}$, (2) $2.56 \times 10^{-4}$ mol dm$^{-3}$, (3) $5.12 \times 10^{-4}$ mol dm$^{-3}$, (4) $7.68 \times 10^{-4}$ mol dm$^{-3}$, (5) $2.30 \times 10^{-4}$ mol dm$^{-3}$ and (6) $1.02 \times 10^{-3}$ and $1.28 \times 10^{-3}$ mol dm$^{-3}$.

in polar solvents was not dependent on the concentrations of DBA below $5.0 \times 10^{-3}$ mol dm$^{-3}$. This indicates that band C is not assigned to an intermolecular excited species.

Fig. 2 shows the emission spectra of BMA in several different solvents. In all solvents the emission spectra consist of two bands (bands A′ and C′). Band C′ appeared as a shoulder on long-wavelength side of band A′ in CH. Judging from the position and shape, band A′ was assigned to the fluorescence of the phenyl group. As shown in Fig. 2, the dependence of band C′ on solvent polarity was very similar to that of band C of DBA. This indicates that these bands are assigned to similar species.

Fig. 3 shows the effect of the addition of TCAA on the emission spectrum of BMA in AN. As shown in Fig. 3, the intensity of band C′ decreased and that of band A′ increased with increasing TCAA concentration. An isoemissive point existed in the spectral change. This indicates that there is just one cause of the spectral change induced by TCAA. The addition of TCAA caused the protonation on the amino group or the hydrogen-bonding interaction between the amino group of BMA and TCAA. Since the amounts of TCAA were very small in this experiment, the polarity of the medium was scarcely affected by the addition of TCAA. Fig. 4 shows the effect of the addition of TCAA on the emission spectrum of DBA in EtOH. As Fig. 4 shows, the intensity of band C decreased and those of bands A and B increased with increasing TCAA concentration. These spectral changes were also explained by the protonation on the amino group or the hydrogen-bonding interaction between the amino group and TCAA.

As mentioned above, the addition of TCAA increased the intensity of normal fluorescence of BMA and those of both normal and excimer fluorescence of DBA. The maximum intensities were obtained in an approximately 2:1 molar mixture of TCAA and BMA (DBA). The intensities of fluorescences of BMA (and DBA) in CH were considerably weaker than those maximum intensities. As mentioned above, the quantum yields of fluorescences of BMA and DBA were much smaller than those of toluene in CH and AN. These findings indicate that the fluorescences of BMA and DBA were largely quenched by the amino group even in CH. Wang and Morawetz examined the fluorescence behavior of DBA adducts with various Lewis acids in ethanol and observed that DBA showed the enhancement of both normal and excimer fluorescence when associated with Lewis acids [28].

Similar effects of TCAA on the emission spectra were observed for 4-phenyl-1-*N*,*N*-dimethylaminobutane (PDAB) [19,20] and toluene- [21], naphthalene-TEA [22] systems in THF. The effects of TCAA observed in these systems were attributed to the hydrogen-bonding between TEA and TCAA (or the protonation of the amine). The protonated (or hydrogen-bonded with TCAA) amino group lost its electron-donating ability. The decrease in the concentration of free amine suppressed the formation of the exciplex and resulted in the increase in the intensity of the fluorescence of aromatic compounds and the decrease in that of the exciplex emission.

El-Bayoumi and co-workers [25,26] observed solvent-induced intramolecular exciplex fluorescence of 2-naphthylmethylamine. This emission band shows a similar red-shift with increasing solvent polarity compared to those of bands C and C′ observed in this study. Therefore, bands C of DBA and band C′ of BMA can be assigned to intramolecular exciplexes between an excited phenyl group and an amino group. As they indicated, since the exciplexes between aromatic compound and amines have large dipole moments, they are stabilized in polar solvents. Lowering the energy of the exciplex would reverse the order of energy level for the excited phenyl group and the exciplex and be favorable for exciplex formation in polar solvents.

Previously we reported that the addition of alcohols increased in the intensity of phenyl group fluorescence and decreased in the intensity of intramolecular exciplex emission for PDAB in THF [19,20]. The addition of alcohols causes hydrogen-bonding with the amino group and a decrease in the electron-donating ability of amino nitrogen. This effect suppresses the formation of the intramolecular exciplex and the quenching of phenyl group fluorescence through the exciplex formation. Similar effects of alcohols on emission spectra were observed in intermolecular exciplex systems (toluene-, naphthalene- and styrene-triethylamine systems [21–23]).

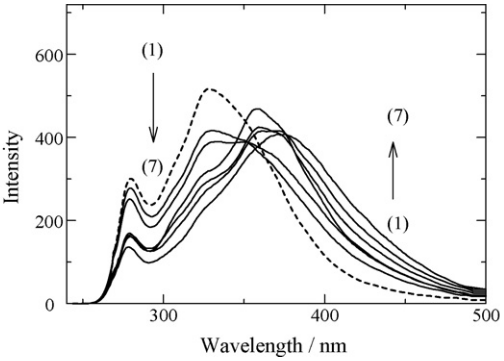

Fig. 5. Emission spectra of BMA in THF–AN mixtures: [BMA] = $1.44 \times 10^{-3}$ mol dm$^{-3}$; vol.% of AN are: (1) 0, (2) 10, (3) 20, (4) 40, (5) 60, (6) 80, and (7) 100.

As shown in Fig. 2, the intensity of band C′ in alcohols was low and that of band A′ is high for BMA compared with the intensities of corresponding bands in THF and AN. Similar results were obtained for DBA. Band C was weak and bands A and B were intense in alcohols compared with those in THF and AN. These findings were consistent with the assignment of bands C and C′ to the intramolecular exciplexes. The emission spectrum of DBA in TFE consists of two bands like those obtained in the presence of TCAA. Since TFE makes strong hydrogen-bonding with a proton accepter, the electron donating ability of the amino group of DBA was decreased by the addition of TFE and the intramolecular quenching of the excited phenyl group by the amino group was largely suppressed. Therefore, the intensities of monomer and excimer emissions become larger in TFE compared with those in THF and AN.

Chandross and Thomas [29] observed a change in the fluorescence spectrum of an intramolecular naphthalene-alkylamine exciplex in benzene upon the addition of AN. They explained this spectral change by the 1:1 complex formation between the exciplex and a small dipolar molecule. They examined the spectral changes using many other dipolar molecules such as dimethylformamide, propionitrile and hexamethylphospharamine and obtained similar results. Amine-hydrocarbon exciplexes generally have a large dipole moment (>10 D) and they can interact with small dipolar species.

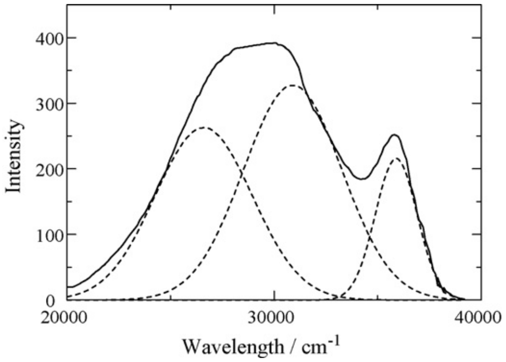

Fig. 6. Decomposition of the emission spectrum obtained for BMA in a THF–AN mixture (vol.% of AN is 20).

Table 1
Relative intensities (and peak wavelengths) of emission bands for BMA observed in THF–AN mixtures

| vol.% of AN | Band A′ | Band C′ | Band D′ |
|---|---|---|---|
| 0 | 0.15 (279 nm) | 0.58 (322 nm) | 0.27 (360 nm) |
| 10 | 0.15 (279 nm) | 0.50 (323 nm) | 0.35 (368 nm) |
| 20 | 0.13 (279 nm) | 0.46 (323 nm) | 0.41 (372 nm) |
| 40 | 0.10 (279 nm) | 0.34 (323 nm) | 0.56 (373 nm) |
| 60 | 0.10 (279 nm) | 0.33 (323 nm) | 0.57 (373 nm) |
| 80 | 0.09 (278 nm) | 0.32 (324 nm) | 0.59 (375 nm) |
| 100 | 0.08 (278 nm) | 0.29 (324 nm) | 0.63 (379 nm) |

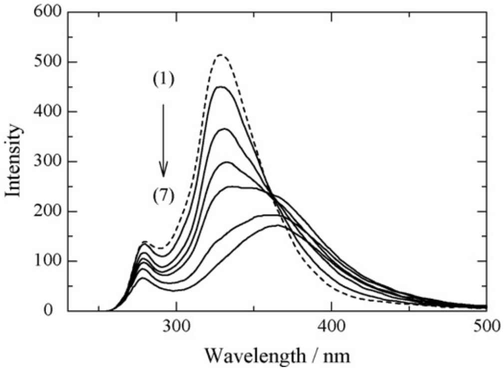

Fig. 7. Emission spectra of DBA in THF–AN mixtures: [BMA] = $1.44 \times 10^{-3}$ mol dm$^{-3}$; vol.% of AN are: (1) 0, (2) 10, (3) 30, (4) 50, (5) 70, (6) 90, and (7) 100.

Fig. 5 shows the emission spectra of BMA in some THF–AN mixtures. As shown in Fig. 5, band C′ had a shoulder at the longer wavelength side in THF. Fig. 6 shows the decomposition of the emission spectrum obtained in a THF–AN mixture (20 vol.% of AN) into three Gaussian curves. The emission spectra of BMA in THF, AN and other THF–AN mixtures could also be decomposed into three component bands. The decomposition of the spectrum of BMA in THF–AN mixtures showed that band C′ (named above) consisted of two component bands, hereafter expressed as band C′ for the short-wavelength band and band D′ for the long-wavelength. The relative intensities and peak wavelengths of these bands are shown in Table 1. Fig. 7 shows the emission spectra of DBA in THF–AN mixtures. Fig. 8 shows the decomposition of the emission spectrum of DBA in a THF–AN

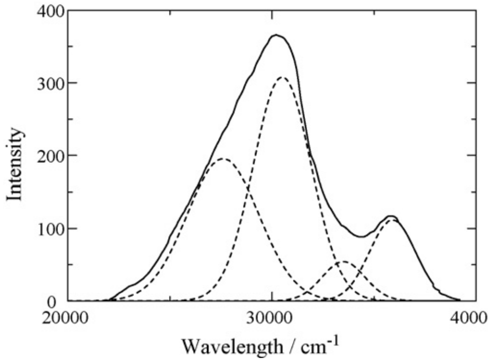

Fig. 8. Decomposition of the emission spectrum obtained for DBA in a THF–AN mixture (vol.% of AN is 30).

Table 2
Relative intensities (and peak wavelengths) of emission bands for DBA observed in THF–AN mixtures

| vol.% of AN | Band A | Band B | Band C | Band D |
|---|---|---|---|---|
| 0 | 0.13 (279 nm) | 0.06 (299 nm) | 0.59 (327 nm) | 0.22 (360 nm) |
| 10 | 0.13 (279 nm) | 0.06 (299 nm) | 0.52 (327 nm) | 0.29 (361 nm) |
| 30 | 0.13 (279 nm) | 0.06 (299 nm) | 0.45 (328 nm) | 0.36 (362 nm) |
| 50 | 0.14 (279 nm) | 0.06 (299 nm) | 0.40 (327 nm) | 0.40 (364 nm) |
| 70 | 0.13 (279 nm) | 0.06 (299 nm) | 0.35 (328 nm) | 0.46 (366 nm) |
| 90 | 0.14 (278 nm) | 0.06 (299 nm) | 0.27 (328 nm) | 0.53 (369 nm) |
| 100 | 0.13 (279 nm) | 0.06 (299 nm) | 0.21 (329 nm) | 0.60 (369 nm) |

mixture (30 vol.% AN) into four Gaussian curves, expressed as bands A–D. The relative intensities and peak wavelengths of these bands are shown in Table 2.

The peak wavelengths of bands A′ and C′ for BMA and bands A–C were almost independent of the amount of AN, but the peaks of bands D′ and D showed red-shifts with increasing AN content. The relative intensities of bands C′ and D′ (or C and D) changed with the amount of AN.

As mentioned above, the decrease in the emission intensity of exciplex and the appearance of a new band upon the addition of AN and other small dipolar species for *N*,*N*-dimethyl-3-(1-naphthyl)propylamine were reported by Chandross and Thomas [29]. They proposed the formation of a stoichiometric complex between the exciplex and a dipolar molecule to explain the spectral change.

To explain the experimental results obtained for BMA in THF–AN mixtures, the following reactions were considered:

$$\text{Ph–A} + h\nu \rightarrow \text{Ph}^*\text{–A}, \quad I_0$$

$$\text{Ph}^*\text{–A} \rightarrow \text{Ph–A} + h\nu_\text{A}, \quad k_0$$

$$\text{Ph}^*\text{–A} \rightarrow (\text{Ex})_\text{free}, \quad k_1$$

$$(\text{Ex})_\text{free} + \text{THF} \rightleftarrows (\text{Ex})_\text{THF}, \quad K_\text{THF}$$

$$(\text{Ex})_\text{free} + \text{AN} \rightleftarrows (\text{Ex})_\text{AN}, \quad K_\text{AN}$$

$$(\text{Ex})_\text{free} \rightarrow \text{Ph–A} + h\nu_{\text{C}'}, \quad k_2$$

$$(\text{Ex})_\text{THF} \rightarrow \text{Ph–A} + \text{THF} + h\nu_{\text{D}'}, \quad k_3$$

$$(\text{Ex})_\text{AN} \rightarrow \text{Ph–A} + \text{AN} + h\nu_{\text{E}'}, \quad k_4$$

where Ph–A denotes BMA in the ground state, $\text{PH}^*$–A the BMA having an excited phenyl chromophore, $(\text{Ex})_\text{free}$ a free exciplex and $(\text{Ex})_\text{THF}$ and $(\text{Ex})_\text{AN}$ are excited complexes formed by the exciplex and THF and AN molecules, respectively. These complexes are stabilized by dipole–dipole interactions, as pointed out by Chandross and Thomas [29]. The emission bands emitted from $(\text{Ex})_\text{free}$, $(\text{Ex})_\text{THF}$ and $(\text{Ex})_\text{AN}$ are expressed by bands C′, D′ and E′. Since bands D′ and E′ are located at very close position and these bands show red-shifts with increasing solvent polarity. These bands could not be separated in practice and they are shown as band D′ together in Table 1 (bands D and E for DBA are shown as band D in Table 2).

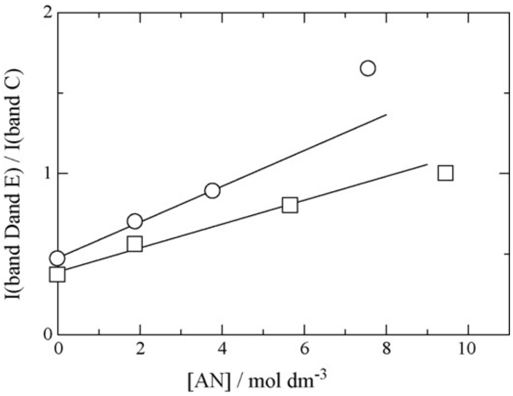

Fig. 9. Plots of the left-hand side of Eq. (1) against [AN] for BMA (○) and DBA (□) in THF–AN mixtures.

The following equation for the ratio of the total intensities of bands D′ and E′ to that of band C′ was derived by a steady-state treatment:

$$\frac{I(\text{bands D}' \text{ and E}')}{I(\text{band C}')} = \frac{1}{k_2}(k_3 K_\text{THF}[\text{THF}] + k_4 K_\text{AN}[\text{AN}]) \tag{1}$$

where $I(\text{band C}')$ is the intensity of the emission from $(\text{Ex})_\text{free}$ and $I(\text{bands D}' \text{ and E}')$ is the total intensity of emissions from $(\text{Ex})_\text{THF}$ and $(\text{Ex})_\text{AN}$. A similar equation was obtained for bands C–E of DBA.

As shown in Fig. 9, linear relationships were observed between the left hand side of Eq. (1) and [AN] in the region where [THF] can be considered to be constant. The red-shift of band D′ (and D) shown in Tables 1 and 2 is caused by changing the component of the complex and by increasing solvent polarity.

## 4. Conclusion

The emission spectrum of BMA consists of three component bands and that of DBA consists of four component bands in polar solvents. For BMA the short-wavelength band (band A′) was attributed to the normal fluorescence and middle- and long-wavelength bands (bands C′ and D′) to the emission bands from the free exciplex and the exciplex complexed with a dipolar molecule. On the other hand, for DBA bands A and B were attributed to the normal and excimer fluorescences, and bands

C and D were attributed to the emission bands from the free exciplex and the exciplex complexed with a dipolar molecule. Bands C and D and bands C′ and D′ behave as a combination band with changes in hydrogen-bonding and protonation abilities of medium. The intensities of bands C and D (C′ and D′) decreased and those of bands A and B (that of band A′) increased upon the addition of TCAA and alcohols.

The intensity changes of bands C and D (C′ and D′) on the addition of AN in THF were explained by the reaction mechanism involving the conversion from the free exciplex to exciplex complexed with a dipolar molecule.

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