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# Effects of solvent and cyclodextrin on the photophysical properties of 4-acetylbiphenyl: intramolecular charge transfer associated with hydrogen-bonding effect

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#### Abstract

The photophysical properties of 4-acetylbiphenyl (ACBP) in various solvents and in aqueous  $\alpha$ -cyclodextrin ( $\alpha$ -CD) solution have been investigated using steady-state and time-resolved fluorescence spectroscopy. In non-polar solvents, the fluorescence spectrum with an emission maximum at 310 nm exhibits a vibrational structure with a non-mirror image to the absorption spectrum, reflecting the change in excited-state geometry toward the coplanarity of the biphenyl moiety. As the solvent polarity increases, the fluorescence spectrum denotes the structureless normal emission and an additional large Stokes' shift band around 380 nm. This behavior indicates the formation of an intramolecular charge transfer (ICT) state through relaxation from the normal excited state where the excited-state geometry change is hindered. Especially in water, the ICT emission is further red shifted to 400 nm with the normal emission band at 310 nm, and the relative intensities between 310 and 400 nm emission bands are affected by the excitation wavelength. However, this excitation wavelength dependence is not so large in organic polar solvents and aqueous  $\alpha$ -CD solutions. The 400 nm emission in water exhibits a single exponential decay with a very short decay time (34 ps) while it shows a bi-exponential or triple-exponential decay in organic solvents and aqueous  $\alpha$ -CD solutions. These results suggest that the ICT state in water is stabilized through exciplex formation by the hydrogen-bonding interaction between the acetyl group and water. © 1997 Elsevier Science S.A.

Keywords: 4-Acetylbiphenyl; Intramolecular charge transfer; Cyclodextrin

## 1. Introduction

The twisted biphenyl is known to exhibit a geometry change in the first excited singlet state toward a planar conformation by internal rotation around the central C-C single bond [1–6]. The conformational relaxation is much faster than the fluorescence decay time, and the fluorescence originates from the nearly planar conformation only. Because of this conformational change, the fluorescence spectrum of biphenyl shows a pronounced structure, in contrast to the structureless absorption spectrum [6]. Recent CNDOS calculation and fluorescence studies [7] have shown that the conformational relaxation of the biphenyl moiety in the excited state is not hindered in any solvents, and the ground-state dihedral angle is little affected by the nature of the solvent. However, a nearly perfect hindrance of the conformational relaxation was detected for tetramethyl substituted

biphenyl [7]. This hindrance was suggested to be due to solute–solvent interactions in which the bulky substituents play an important role.

Our previous investigations on the photophysical properties of pre-twisted biphenyl derivatives, 4-biphenylcarboxylic acid (4-BPCA) and 2-biphenyl carboxylic acid (2-BPCA), also showed the solvent dependence of the conformational relaxation [8,9]. For example, in non-polar solvents the pre-twisted biphenyl moiety becomes coplanar on excitation, whereas in polar protic solvents the pre-twisted conformation is preserved and the intramolecular charge transfer (ICT) interaction is manifest. Especially, the pretwisted biphenyl moiety of 2-BPCA in polar protic solvents seemed to be further twisted on excitation because of the enhanced steric hindrance by the ortho carboxyl group through the hydrogen bonding interaction with solvent. Such hydrogen-bonding effects on the excited-state ICT and geometry change have also been demonstrated by the inhibition of the ICT emissions of 4-BPCA and 2-BPCA in aqueous cyclodextrin (CD) solution [10,11]. That is, the excited-

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state ICT interaction of 4-BPCA, a pre-twisted molecule, in aqueous CD solution was shown to be inhibited through the formation of the inclusion complex with  $\alpha$ -CD or  $\beta$ -CD. Especially in  $\alpha$ -CD solution, the 2:1 complex is formed, accommodating the excited-state geometry change of the biphenyl moiety toward coplanarity in competition with the ICT interaction by blocking the interaction of the  $\pi$ -conjugated carboxylic acid group with water. It was also observed that the excited-state ICT process of 2-BPCA is inhibited through the formation of the inclusion complex. The inhibition of ICT was attributed to the reduction of the hydrogen bonding of the carboxylic acid group with water in the CD cavity. However, our understanding of the hydrogen-bonding effect on the excited-state conformational change of biphenyl derivatives and ICT is still far from complete.

In this work, we attempted to study the effects of the solvents and CD on the photophysical properties of 4-acetyl-biphenyl (ACBP) using steady state and time-resolved fluorescence spectroscopies. The excited-state behavior of ACBP in water and aqueous CD solution was observed to be significantly different from that observed in 4-BPCA, an analog of ACBP, being interpreted in terms of the excited-state ICT associated with hydrogen bonding effect.

# 2. Experimental details

ACBP and  $\alpha$ -CD and  $\beta$ -CD were purchased from Aldrich Chemical Co.  $\alpha$ -CD and  $\beta$ -CD were used without further purification and ACBP was purified by repetitive recrystallization from ethanol. The melting point of ACBP (120–121 °C) is in good agreement with the reported value [12]. All the organic solvents used were spectroscopic grade and further purified as described elsewhere [13].

The absorption spectra were measured on a Varian Cary 3 spectrophotometer. The steady-state fluorescence spectra were recorded on a scanning SLM-AMINCO 4800 spectrofluorometer which makes it possible to obtain the corrected spectra using Rhodamine B as a quantum counter. The solutions were accurately diluted with the proper solvent to have optical densities of less than or equal to 0.6 at the excitation wavelength to eliminate possible self-absorption effects. All the sample solutions were deaerated by 3-4 freeze-pumpthaw cycles under 10<sup>-4</sup> Torr before the measurements. Fluorescence lifetimes of ACBP in various solutions were measured by a time-correlated single photon counting (TCSPC) system. This system employed a dual jet dye laser (Coherent, model 702) synchronously pumped by a mode-locked Ar ion laser (Coherent, Innova 200) as described in detail previously [8-11]. The cavity-dumped beam had a 1 ps pulse width, average power of ca. 40 mW at 3.8 MHz dumping rate, and tunability of 560-620 nm when Rhodamine 6G for gain dye and DODCI (diethoxydicyanine iodide) for the saturable absorber were used. To excite the sample, the dye laser pulses were frequency doubled by a  $\beta$ -BBO ( $\beta$ -barium borate) crystal. All the standard electronics used for the TCSPC were from EG&G Ortec. This method allows a time resolution of about 20 ps after deconvolution.

## 3. Results and discussion

#### 3.1. Solvent effects

Fig. 1 shows the absorption spectra of ACBP in various solvents at room temperature. They show absorption maxima around 272 nm in non-polar solvents. The molar extinction coefficient is very high ( $\sim 10^4 \, \mathrm{M}^{-1} \, \mathrm{cm}^{-1}$ ), and the absorption maximum shifts slightly from 275 nm to 285 nm as the solvent polarity increases. These results imply that this band is attributed to the ( $\pi$ , $\pi^*$ ) transition of biphenyl moiety. The (n, $\pi^*$ ) transition of the carbonyl group is hidden by an extended  $\pi$ -conjugation of the biphenyl rings to the neighboring carbonyl group. This is supported by the observation that the absorption maximum of ACBP is significantly red shifted compared with that of unsubstituted biphenyl or 4-biphenylacetic acid (4-BPAA) (250 nm) [8].

Fig. 2 shows the fluorescence spectra of ACBP in various solvents. In contrast to the weak solvent-dependence of the

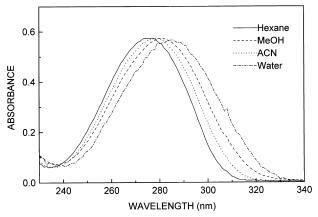


Fig. 1. Absorption spectra of ACBP in various solvents. The spectra are normalized arbitrarily.

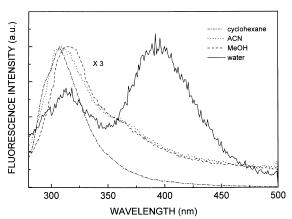


Fig. 2. Fluorescence emission spectra of ACBP  $(1.0 \times 10^{-5} \text{ M})$  in various solvents. The emission spectra were measured with an excitation at 260 nm and normalized arbitrarily.

absorption maximum, the steady-state emission properties of ACBP are strongly solvent dependent, indicating a possibility of a change in the character of the electronic state, possibly through the solvent relaxation process in the excited state. In non-polar solvents, the fluorescence spectrum of ACBP with a maximum at 310 nm has a vibrational structure with a nonmirror image to the absorption spectrum as in the case of unsubstituted biphenyl [8,10]. This indicates that the molecular geometry change takes place in the excited <sup>1</sup>L<sub>b</sub> state with minor polarity [14]; the pre-twisted Franck-Condon (FC) conformation of the biphenyl moiety is changed into the coplanar conformation on excitation by internal rotation around the central C-C bond. Thus, most of the normal fluorescence is from the coplanar conformation. However, as the solvent polarity increases, the 310 nm emission band becomes structureless and slightly red shifted in parallel with the red shift of the absorption maximum, indicating that the normal fluorescent state is changed from the <sup>1</sup>L<sub>b</sub> state to the polar <sup>1</sup>L<sub>a</sub> state which is more solvent dependent. The structureless spectrum suggests that the excited-state geometry change toward coplanar conformation is more or less hindered in the <sup>1</sup>L<sub>a</sub> state through a molecule–solvent interaction with the subsequent solvent relaxation. In other words, the biphenyl moiety of the excited ACBP in the polar solvent would remain as twisted as in the ground state. Such solvent effects on the fluorescence spectra were not observed for unsubstituted biphenyl and 4-BPAA [8], in which the steric hindrance of the hydrogen atoms in the ortho positions is known to be overcome by the enhanced resonance interaction between the two phenyl rings in the excited state with the subsequent intramolecular twisting relaxation toward coplanar conformation [15]. Considering these facts, the extended  $\pi$ -conjugation of the phenyl moiety to acetyl group seems to play an important role in the interaction with polar solvent to hinder the internal rotation of the biphenyl moiety in the excited state of ACBP. The extended  $\pi$ -conjugation would induce an excited-state resonance contribution of the acetyl group to the benzene ring, resulting in the increased polarity to facilitate the interaction with polar solvent. Hence protonation of the acetyl group coplanar with the benzene ring should also be facilitated in the excited state. Consequently this would be a barrier to enhance the resonance interaction between the two phenyl rings, inhibiting the internal rotation toward coplanarity in protic polar solvents.

It should also be pointed out that as the solvent polarity increases a new broad emission shoulder appears around 380 nm with a large Stokes shift (ca. 10 000 cm<sup>-1</sup>) in addition to the slightly red-shifted blue emission (around 310 nm) (Fig. 2). This indicates that the excited ICT state is formed by the relaxation from the initially excited <sup>1</sup>L<sub>a</sub> state, which is as in the case of 4-BPCA or 2-BPCA [8–10]. It is noteworthy that in contrast to the large Stokes shifted broad ICT emission in other protic polar solvents, the ICT emission in water is further red shifted to 400 nm, even though the maximum of the normal emission around 310 nm is nearly independent of the hydrogen-bonding ability of solvents. This dual fluores-

cence behavior was not observed for 4-cyanobiphenyl which shows only normal fluorescence from the  $^1L_a$  state or a mixed state of  $^1L_a$  and  $^1L_b$  depending on the solvent polarity [14]. These observations suggest that the ICT process of ACBP in water seems to be influenced by the enhanced intermolecular hydrogen-bonding of the acetyl group with water in the excited state. Supporting this, the fluorescence spectrum in the aqueous solution is changed significantly on addition of dioxane, showing a dual emission and an isoemissive point in dioxane–water mixture (Fig. 3). These results are similar to those of other ICT molecules such as 2-N-arylamino-6-naphthalenesulfonate [16], in which dioxane inhibits a solute–solvent interaction and the charge transfer process.

It is also noteworthy that the fluorescence spectrum of ACBP in water shows a different feature depending on the excitation wavelength (260 and 300 nm) (Fig. 4). In the case of 260 nm excitation, the emission spectrum exhibits the dual emission (310 and 400 nm). However, with an excitation at 280 nm or 300 nm, the ICT emission at 400 nm is dominant. This excitation-wavelength dependence of the dual emission is similar to the typical red edge effect [17,18] observed in the twisted ICT (TICT) fluorescence which is usually observed under the restrictive molecular mobility environment like the polymer system [19,20]. When the

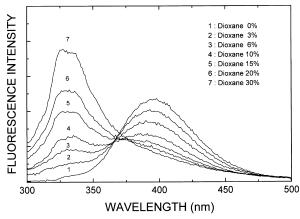


Fig. 3. Fluorescence emission spectra of ACBP in dioxane–water mixed solvents. The excitation wavelength was 300 nm.

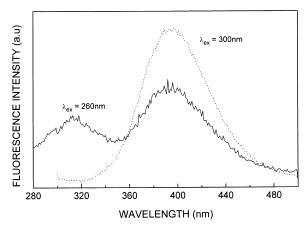


Fig. 4. Fluorescence emission spectra of ACBP measured with different excitation wavelengths, 260 and 300 nm, in aqueous solution.

conformational change is restricted, a number of independent conformers absorbing different wavelengths exist within the time-scale of the excited-state lifetime. A pre-twisted conformer in the ground state can be excited by the red edge absorption and undergoes a facile conversion to the TICT state because the necessary angle for the twist relaxation is smaller. Actually, ACBP in the ground state has been reported to have a dihedral angle of 25° between the two phenyl moieties [10]. In non-polar solvents, the non-planar biphenyl moiety becomes coplanar on excitation. However, as stated above, the conformational change is restricted in protic polar solvents, especially in water, through the strong hydrogenbonding of the carbonyl group with solvent. Under this condition, there can exist another pre-twisted conformer which has a dihedral angle larger than 25°. Thus it can be speculated that the enhanced 400 nm emission should originate from the TICT-like state. Supporting this implication, the excitation spectra exhibit the monitoring wavelength dependence as shown in Fig. 5. The excitation spectrum for the 400 nm emission is distinctly different from that for the 310 nm emission. These results suggest that the TICT-like state may be formed either by a further twist of the normal conformer (aform) in the locally excited state or directly by excitation of the highly pre-twisted conformer (b-form) as shown in Scheme 1. As Twine and Bursey [21] reported that ACBP has a large dipole moment even in the ground state (in addition to the normal pre-twisted conformer), the highly pretwisted state is expected to exist as an intermolecular complex (ACBP-H<sub>2</sub>O) in the ground state by the strong hydrogen-

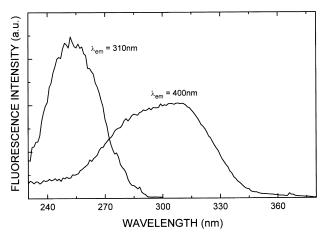


Fig. 5. Fluorescence excitation spectra of ACBP in aqueous solution for the 310 and 400 nm emission bands.

bonding interaction with water. These hydrogen-bonding complexes may have slightly different absorption maxima compared with the normal conformer. This interaction should induce a structural change in ACBP and thus change its absorption bandwidth. Actually, it was observed that the absorption spectrum of ACBP in water is broad compared with those in other organic solvents (Fig. 1), and it was fitted by two Gaussian functions (Fig. 6), resolved into two absorption bands having maxima at 270 and 290 nm respectively.

In order to understand further the excited-state geometry change and ICT process of ACBP, we measured the picosecond time-resolved fluorescence decays of ACBP in various solvents at 350 and 400 nm emissions. Fig. 7 shows the typical fluorescence decay profiles of ACBP in water and methanol monitored at 400 nm, together with the instrument response function (IRF). The analyzed decay times in various solvents are listed in Table 1. In a non-polar solvent such as hexane, the decay of the 350 nm emission was too short to be measured and it is estimated to be close to the instrument response time, ca. 10 ps. This is much shorter than that expected for the coplanar fluorescent state which is usually about 1 or 2 ns [8,15]. Probably the longer decay component can be resolved when the fluorescent decay is detected around 310 nm. However, unfortunately, it was impossible to meas-

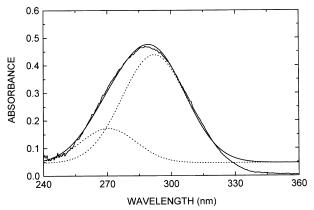


Fig. 6. The absorption spectrum of ACBP in aqueous solution fitted by two Gaussian functions.

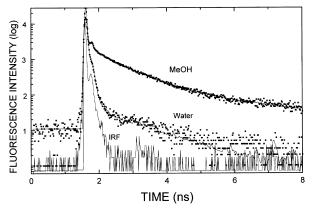


Fig. 7. Fluorescence decay profiles at 400 nm of ACBP in water and methanol. The excitation wavelength was 290 nm.

Table 1 Fluorescence decay times (ps) of ACBP in various solvents and aqueous  $\alpha$ -CD solution at room temperature

Medium	Monitoring wavelength	
	350 nm	400 nm
n-Hexane	≈10 (1.00) <sup>a</sup>	_
Acetonitrile	20 (0.92)	30 (0.80)
	730 (0.08)	780 (0.16)
		3100 (0.04)
Methanol	20 (0.94)	30 (0.69)
	650 (0.06)	670 (0.22)
		2540 (0.09)
Water	26 (1.00)	34 (1.00)
α-CD (8 mM)	_	31 (0.98)
		590 (0.02)

<sup>&</sup>lt;sup>a</sup> The values in parentheses represent the pre-exponential factor. Measurement error limits are less than 10% of the listed values.

ure the fluorescent decay around 310 nm owing to the overlap with a large scattering signal under our experimental conditions. Thus, it is likely that the 350 nm emission can be attributed to a portion of a pre-twisted conformer rather than a coplanar conformer, and the short decay time might be due to the rapid rotational relaxation from the pre-twisted form to the coplanar form on excitation. Actually, in hexane a portion of the short fluorescent decay time of the pre-twisted 4-BPCA has been detected in addition to the coplanar fluorescent decay time [8]. However, in organic polar solvents such as methanol and acetonitrile, the decay times at 350 nm emission are resolved into the two decay components. The lifetime of the major component is ca. 20 ps and that of the minor component is ca. 700 ps, supporting the idea that the excited-state rotational relaxation toward coplanarity is more or less inhibited in polar solvents. For the 400 nm emission, similar decay times were observed with an additional long decay component of ca. 3 ns lifetime. This long decay time is similar to that of the TICT emission observed for 2-BPCA [8], indicating that the large Stokes shift emission of ACBP in polar solvents originates from the TICT-like state. However, the ICT emission in water exhibits a single exponential decay with a very short lifetime (34 ps). The remarkable change in the decay behavior of the ICT emission in water may be attributable to the strong hydrogen-bonding interaction between the carbonyl group and water. The strong hydrogen-bonding interaction probably induces the exciplex formation, 1(ACBP/H<sub>2</sub>O)\*, which may be facilitated through the ICT state. The energy of the exciplex is even lower than that of the ICT state, as demonstrated by the further red shift of the ICT emission in water.

## 3.2. Cyclodextrin effects

In order to substantiate the hydrogen-bonding effect on ICT, the absorption and fluorescence spectra were measured in aqueous cyclodextrin (CD) solutions. The absorption

spectra of 4-ACBP were measured in aqueous solution containing different concentrations of  $\alpha$ -CD. As the concentration of  $\alpha$ -CD increases, the absorption maximum at 284 nm is slightly red shifted to 290 nm with a little decrease in molar extinction coefficient, indicating the formation of ACBP–CD inclusion complexes.

In contrast to the weak dependence of the absorption spectra on CD concentration, the fluorescence spectra exhibit a significant change as the concentration of  $\alpha$ -CD increases, as shown in Fig. 8. The 400 nm emission in water is decreased on addition of  $\alpha$ -CD, and at the same time a new emission band at 330 nm appears. This indicates that ACBP becomes more sensitive to the microenvironment of CD on excitation, supporting again the idea that ACBP forms the inclusion complex with  $\alpha$ -CD. In order to determine the stoichiometry of the inclusion complex, the dependence on  $\alpha$ -CD of the ACBP fluorescence was analyzed using the Benesi–Hildebrand equation [22,23] for the 1:1 complex (Eq. (1)) and 2:1 complex (Eq. (2)) between ACBP and  $\alpha$ -CD as shown below:

$$\frac{1}{I_{\rm f}^0 - I_{\rm f}} = \frac{1}{I_{\rm f}^0 - I_{\rm f}'} + \frac{1}{K(I_{\rm f}^0 - I_{\rm f}')[\alpha - \text{CD}]}$$
(1)

$$\frac{1}{I_{\rm f}^0 - I_{\rm f}} = \frac{1}{I_{\rm f}^0 - I_{\rm f}'} + \frac{1}{K(I_{\rm f}^0 - I_{\rm f}')[\alpha - \text{CD}]^2}$$
(2)

where K is the formation constant,  $I_f^0$  the initial fluorescence intensity of free ACBP at 400 nm,  $I_f'$  the fluorescence intensity of the  $\alpha$ -CD inclusion complex, and  $I_f$  the observed fluorescence intensity. According to Eq. (1), a plot of 1/ $(I_f^0 - I_f)$  vs.  $1/[\alpha$ -CD] gives an upward curve as shown in Fig. 9(a). However, a plot of  $1/(I_f^0 - I_f)$  vs.  $1/[\alpha$ -CD]<sup>2</sup> reveals a linear relationship (Fig. 9(b)). This analysis reflects the formation of 2:1 inclusion complexes between ACBP and  $\alpha$ -CD as in the case of 4-BPCA [10] (see Scheme 2). This is consistent with the observation of two isoemissive points (Fig. 8).

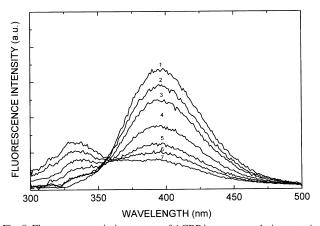


Fig. 8. Fluorescence emission spectra of ACBP in aqueous solution containing different amounts of  $\alpha$ -CD. The concentrations of  $\alpha$ -CD were changed from 0 to 16 mM along the direction from 1 to 7. The excitation wavelength was 300 nm.

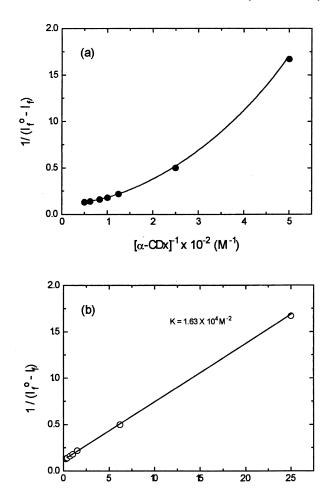
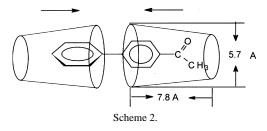


Fig. 9. The Benesi–Hilderbrand plots for the  $\alpha$ -CD/ACBP complex: (a) the plot of  $1/(I_{\rm f}^0-I_{\rm f})$  vs. [CD] $^{-1}$  and (b) the plot of  $1/(I_{\rm f}^0-I_{\rm f})$  vs. [CD] $^{-2}$ .

 $[\alpha - CDx]^{-2} \times 10^{-4} (M^{-2})$ 



As shown in Scheme 2, ACBP in the 2:1 inclusion complex is entrapped in the  $\alpha$ -CD cavity, and the hydrogen-bonding interaction is inhibited. Therefore, the fluorescence decay time of ACBP in 8 mM  $\alpha$ -CD aqueous solution was observed to be similar to that measured in methanol, except for the absence of a long-lived component attributed to the ICT state (Table 1). If this is the case, the excitation wavelength dependent ratio of the ICT emission to the normal emission in water should also be affected by the  $\alpha$ -CD concentration where the 2:1 complex is formed. Fig. 10 shows the relative ratios of the two emissions measured on excitation at two different wavelengths as a function of  $\alpha$ -CD concentration. When the excitation wavelength was 300 nm, the large rela-

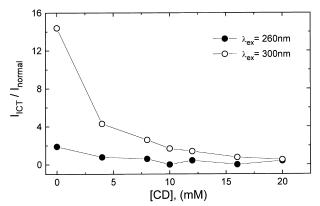


Fig. 10. The plot of the ratio of the normal emission intensity to the ICT emission intensity of ACBP as a function of  $\alpha$ -CD concentration with different excitation wavelengths.

tive ratio in water was decreased and became similar to the value for excitation at 260 nm. In other words, the excitation wavelength dependence of the relative ratio between the normal emission and the ICT emission is not effective any more in the 2:1 complex, as in the cases of organic polar solutions. As discussed above, the highly pre-twisted ACBP, which is more sensitive to hydrogen-bonding than the normally pre-twisted ACPB, absorbs 300 nm light to form species to exhibit the ICT emission directly. Thus, if the hydrogen-bonding is inhibited as in the CD inclusion complex, the formation of the ICT state by direct excitation at 300 nm should be inhibited. These results again support the idea that the ICT state



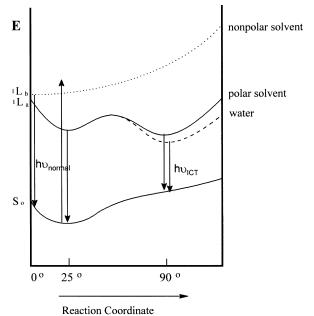


Fig. 11. A proposed schematic diagram of the potential energy surface for ACBP undergoing an excited-state geometry change and ICT state formation depending on the solvent polarity and hydrogen-bonding ability.

in water is further stabilized through exciplex formation between ACBP and water.

## 4. Conclusion

Our experimental observations suggest that the excitedstate potential surface connecting the twisted conformation and the coplanar conformation of ACBP is dependent on the solvent polarity and the hydrogen-bonding ability as shown in Fig. 11. In non-polar solvents, the normally pre-twisted conformation with 25° dihedral angle is changed into the coplanar conformation rapidly on excitation into the less polar <sup>1</sup>L<sub>b</sub> state, as in the case of unsubstituted biphenyl (the dotted line in Fig. 10). In polar solvents, however, the excited-state internal rotation of the biphenyl moiety in the <sup>1</sup>L<sub>a</sub> state is partially hindered by the solute–solvent interaction, and further twist relaxation takes place to form a TICTlike state partially as demonstrated by a dual emission in polar solvents. In particular, the ICT state in water is stabilized by exciplex formation, \(^1(ACBP/H\_2O)^\*\), through the strong hydrogen-bonding between the acetyl group and water.

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