

Substituent Effects on the Ground-State Properties of Naphthalene-Based Analogues of Salicylideneaniline in Solution

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Substituent effects on the ground-state properties of seven naphthalene analogues of salicylideneaniline (**1**–**7**) were studied by comparison of both steady-state and transient absorption spectra. The naphthalene-based salicylideneanilines **1**–**5** were prepared from 2-hydroxy-1-naphthaldehyde and corresponding amines. The UV absorption spectra of **1**–**5** were largely dependent on the substituent group on the amino group. Transient absorption studies revealed that the introduced naphthyl group in **1**–**5** prolonged the lifetime of $^1K_{trans}$ in non-polar solvent probably due to the formation of intramolecular hydrogen bonding between nitrogen and C–H hydrogen on the naphthyl group. In acetonitrile, the lifetimes of **1**–**6** are much longer than those in benzene, probably because the zwitterionic forms are stabilized in polar solvents, whereas the much shorter lifetime in ethanol suggests that $^1K_{trans}$ undergoes the solvent assisted re-enolizaiton reaction in ethanol.

Salicylideneaniline (SA), reported by Cohen et al. in 1960s, is one of the typical molecules exhibiting excited-state intramolecular proton (or hydrogen-atom) transfer (ESIPT).[1–3] The potential energy diagram of the ESIPT reaction of SAs has been proposed as depicted in Fig. 1. SA and its analogues usually exist in their enol form (^1E) in the ground state. On irradiation of SAs, ESIPT takes place from the excited-state enol form ($^1E^*$) to the excited-state *cis*-keto tautomer ($^1K_{cis}^*$), which deactivates by 1) non-radiative decay, 2) emission of large Stokes-shifted fluorescence, 3) intersystem crossing to the excited triplet species ($^3K_{cis}^*$), or 4) isomerization to produce the *trans*-isomer in the ground state ($^1K_{trans}$). The meta-stable $^1K_{trans}$ reverts back to the stable ^1E. These deactivation processes on the excitation of SAs have been studied both in their crystal form[4–6] and in solution.[7–9]

Substituent effects on the photochemistry of SA have been studied in solution.[10–16] Among many SAs, interesting features of naphthalene-based analogues of SA derivatives have been reported. When naphthalene is introduced in the A ring of SA (Fig. 2), the compounds exhibit solvatochromism, thermochromism, and photochromism, while naphthalene introduced in the B ring of SA exhibits only photochromism.[17] Despite their interesting features, there are few reports on the structure–property relationship of naphthalene-based SAs based on spectroscopic study in solution. In this paper, we explore the structure–property relationship of naphthalene-based SA analogues and hydrogen-atom transfer in the ground state. In order to investigate systematically, compounds **1**–**7** (Fig. 3) were prepared. In **1**–**5**, the naphthyl group is introduced in the A ring of SA, whereas the naphthyl group is introduced in the B ring in **6**. In addition, intramolecular hydrogen bonding is blocked in **7**. Briefly, the introduction of substituents in the B ring considerably influenced their absorption spectra in various solvents, when naphthalene was substituted in the A ring. Transient absorption studies revealed that the introduction of naphthyl groups in the A ring prolonged the lifetime of $^1K_{trans}$ in non-polar solvents, probably due to the formation of intramolecular hydrogen bonding between nitrogen and C–H hydrogen on the naphthyl group (Fig. 4).

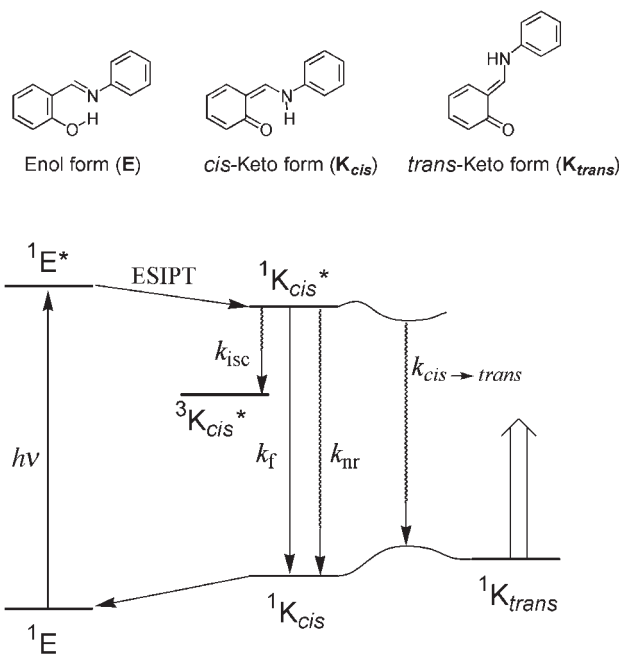

Fig. 1. The schematic diagram of the excited-state intramolecular hydrogen transfer (ESIPT) reaction of salicylideneaniline (SA) and its analogues: excitation of the enol primary structure ($^1\mathbf{E}$ and $^1\mathbf{E}^*$) give excited keto tautomer ($^1\mathbf{K}_{cis}^*$) by ESIPT, which undergoes intersystem crossing to the excited triplet species ($^3\mathbf{K}_{cis}^*$), photoisomerization to the ground-state *trans*-isomer ($^1\mathbf{K}_{trans}$), or decays to the ground state ($^1\mathbf{K}_{cis}$) with fluorescence emission or non-radiatively. The meta-stable $^1\mathbf{K}_{trans}$ undergoes thermal isomerization to $^1\mathbf{K}_{cis}$, which immediately reverts to $^1\mathbf{E}$ by proton-transfer reaction in the ground state.

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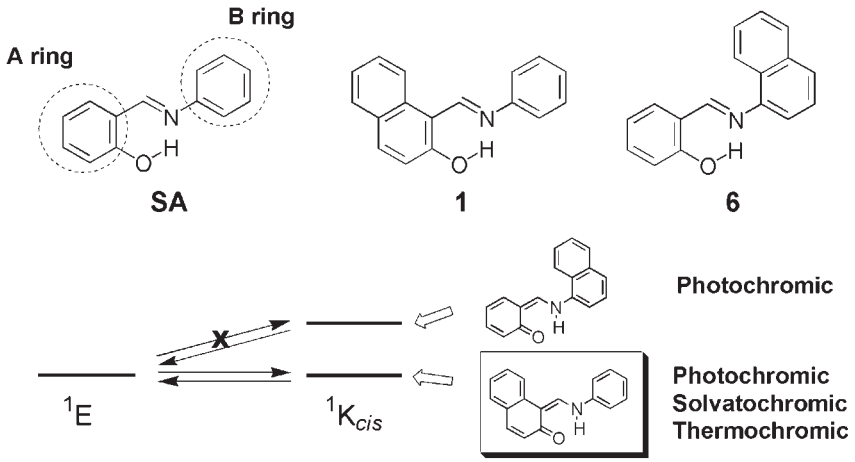
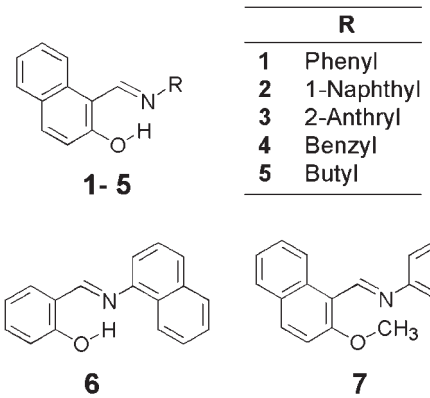


Fig. 2. Structures of naphthyl analogues of SAs. Naphthalene is introduced in the A ring of SA **1**, and in the B ring **2**. Compound **1** exhibits photochromism, solvatochromism, and thermochromism, whereas **2** shows photochromism only.



	R
1	Phenyl
2	1-Naphthyl
3	2-Anthryl
4	Benzyl
5	Butyl

Fig. 3. Chemical structures of naphthyl analogues of SAs **1**–**7**.

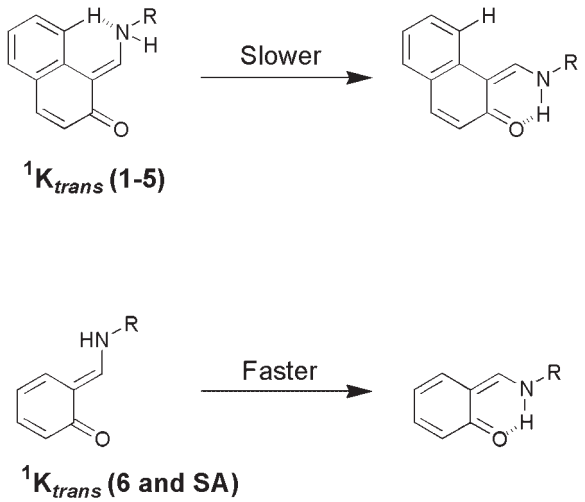


Fig. 4. Hydrogen bonding between nitrogen and hydrogen on naphthyl ring in $^1K_{trans}$ of **1**–**5**.

Experimental

Materials. Compounds **1**,[17] **2**,[17] **4**,[18] and **5**[19] have been reported previously; however, no NMR data were shown in Ref. 18 for **4** and Ref. 19 for **5**, respectively.

Typical Procedure: A mixture of a corresponding amine and aldehyde in ethanol was refluxed for 3 h. After evaporation, a crude mixture was purified by recrystallization twice from ethanol to give a pure product.

1-(Anthracen-2-yliminomethyl)naphthalen-2-ol (**3**) was prepared from 2-hydroxy-1-naphthaldehyde and 2-aminoanthracene in 72% yield: ^{1}H NMR ($CDCl_3$, 400 MHz, Me_4Si) δ 15.68 (1H, d, J = 4.4 Hz, OH), 9.53 (1H, d, J = 4.0 Hz, CH=N), 8.46 (2H, d, J = 3.2 Hz, ArH), 8.17 (1H, d, J = 8.3 Hz, ArH), 8.11 (1H, d, J = 9.1 Hz, ArH), 8.03–8.01 (2H, m, ArH), 7.92 (1H, d, J = 2.0 Hz, ArH), 7.82 (1H, d, J = 9.2 Hz, ArH), 7.74 (1H, d, J = 8.0 Hz, ArH), 7.60–7.54 (2H, m, ArH), 7.51–7.49 (2H, m, ArH), 7.36 (1H, t, J = 8.0 Hz, ArH) 7.13 (1H, d, J = 9.2 Hz, ArH). ^{13}C NMR ($CDCl_3$, 125 MHz, Me_4Si) δ 170.7, 154.0, 142.0, 136.9, 133.3, 132.4, 131.8, 131.7, 130.3, 130.2, 129,4, 128.3, 128.1, 128.0, 127.3, 126.5, 126.2, 126.0, 125.6, 123.6, 122.4, 119.8, 118.9, 117.2, 109.1. Elemental Analysis. Anal. Calcd for $C_{25}H_{17}NO$: C, 86.43; H, 4.93; N, 4.03%. Found: C, 86.15; H, 5.19; N, 3.95%.

1-(Benzyliminomethyl)naphthalen-2-ol (**4**) was prepared from 2-hydroxy-1-naphthaldehyde and benzylamine in 28% yield. ^{1}H NMR ($CDCl_3$, 400 MHz, Me_4Si) δ 14.78 (1H, s, OH), 8.88 (1H, d, J = 7.6 Hz, CH=N), 7.87 (1H, d, J = 8.0 Hz, ArH), 7.70 (1H, d, J = 9.2 Hz, ArH), 7.63 (1H, d, J = 8.4 Hz, ArH), 7.44–7.21 (7H, m, ArH), 6.96 (1H, d, J = 9.2 Hz, ArH), 4.83 (2H, s, CH_2Ar). Elemental Analysis. Anal. Calcd for $C_{18}H_{15}NO$: C, 82.73; H, 5.79; N, 5.36%. Found: C, 82.34; H, 5.78; N, 5.20%.

1-Butyliminomethylnaphthalen-2-ol (**5**) was prepared from 2-hydroxy-1-naphthaldehyde and *n*-butylamine in 82% yield. ^{1}H NMR ($CDCl_3$, 400 MHz, Me_4Si) δ 14.42 (1H, s, OH), 8.67 (1H, d, J = 8.0 Hz, CH=N), 7.83 (1H, d, J = 8.2 Hz, ArH), 7.67 (1H, d, J = 9.2 Hz, ArH), 7.59 (1H, dd, J = 6.8 and 1.2 Hz, ArH), 7.42 (1H, td, J = 6.8 and 1.2 Hz, ArH), 7.22 (1H, t, J = 8.2 Hz, ArH), 6.90 (1H, d, J = 9.2 Hz, ArH), 3.61 (2H, t, J = 6.8 Hz, CH_2), 1.75 (2H, m, CH_2), 1.47 (2H, m, CH_2), 0.99 (3H, t, J = 7.6 Hz, CH_3). Elemental Analysis. Anal. Calcd for $C_{15}H_{17}NO$: C, 79.26; H, 7.54; N, 6.10%. Found: C, 79.22; H, 7.53; N, 6.16%.

(2-Methoxynaphthalen-1-ylmethylene)phenylamine (**7**) was prepared from 2-methoxy-1-naphthaldehyde and aniline in 74% yield. ^{1}H NMR ($CDCl_3$, 400 MHz, Me_4Si) δ 9.52 (1H, d, J = 8.3 Hz, ArH), 9.25 (1H, s, CH=N), 7.92 (1H, d, J = 9.1 Hz, ArH), 7.77 (1H, d, J = 8.1 Hz, ArH), 7.57 (1H, m, ArH), 7.44–7.37 (3H, m, ArH), 7.31–7.21 (4H, m, ArH), 3.97 (3H, s, Me). ^{13}C NMR ($CDCl_3$, 125 MHz, Me_4Si) δ 159.4, 158.6, 153.4, 133.7, 131.9, 129.2, 129.1, 129.0, 128.4, 128.2, 125.8, 125.5, 124.2, 121.0,

117.1, 115.0, 112.5, 56.5. Elemental Analysis. Anal. Calcd for $C_{18}H_{15}NO$: C, 82.73; H, 5.79; N, 5.36%. Found: C, 82.73; H, 5.81; N, 5.41%.

Measurements. The ^1H and ^{13}C NMR spectra were measured with Bruker ARX-400 (400 MHz for ^1H NMR) and Bruker AVANCE 500 (125 MHz for ^{13}C NMR) spectrometers respectively, in solutions of $CDCl_3$ with tetramethylsilane as an internal standard. The UV absorption and fluorescence spectra were recorded on a Shimadzu UV-1600 UV–visible spectrophotometer and on a Hitachi F-4500 fluorescence spectrometer, respectively. Laser flash photolysis was performed by using an excimer laser (Lambda Physik LPX-100, 308 nm, 20 ns fwhm) as the excitation light source and a pulsed xenon arc (Ushio UXL-159) was used as a monitoring light source. A photomultiplier (Hamamatsu R-928) and a storage oscilloscope (Iwatsu TS-123) were used for detection.

Results

Steady-State Absorption and Fluorescence Spectra. Figure 5 shows the steady-state UV absorption spectra of the series of compounds **1**–**7** in various solvents under argon at room temperature. The similar absorption spectra of compounds **1**, **2**, and **6** have been reported previously.[17] Measurements were performed using methylcyclohexane and benzene as non-polar solvents, acetonitrile as a polar aprotic solvent, and ethanol as a polar protic solvent. Compounds **1**–**5**, where naphthalene was introduced in the A ring, exhibited solvatochromism. Thus, the absorption maximum shifted to the longer wavelength in the polar solvent and protic solvent. Compound **6** exhibited similar spectra to the parent compound SA in each solvent (Fig. 5f). The absorption spectra of compound **7** are almost the same in various solvents.

The fluorescence and fluorescence excitation (FLE) spectra for **1**–**6** were observed at low temperature. Figure 6 shows the fluorescence and fluorescence excitation spectra for **1**–**6** in methylcyclohexane and in ethanol at 77 K. The similar fluorescence spectra of **1**, **2**, and **6** have been reported previously.[17] The fluorescence excitation spectra for **1**–**5** in both solvents were similar to the absorption spectra in ethanol at room temperature (Figs. 6a–6e). The fluorescence and fluorescence excitation spectra of **6** in both methylcyclohexane and in ethanol at 77 K (Fig. 6f) are similar to the absorption spectra at room temperature.

Transient Absorption Spectra in Various Solvents. The transient absorption spectra for **1** and **6** have already been reported.[17] Figure 7a shows the transient absorption spectra of **2** (1.0×10^{-4} M) in benzene under argon at room temperature. The decay curve of the transient species peaking at 460 nm was not affected by oxygen and therefore is ascribed to $^1K_{trans}$ in the ground state. A weak peak at 550 nm was quenched by oxygen and was assigned to $^3K_{cis}$*. In ethanol, the transient absorption band for $^1K_{trans}$ of **2** could not be detected (Fig. 7b). Compound **1** exhibited a similar solvent effect to **2** on the transient absorption spectra. In the transient absorption spectra of **3** (1.0×10^{-4} M), only the shoulder of the transient absorption band for $^1K_{trans}$ was observed in benzene, acetonitrile, and ethanol, because it overlapped with the band for $^1K_{cis}$ in each solvent (Fig. 7c). Therefore, the lifetimes of $^1K_{trans}$ for **3** could not be determined. Figure 7d shows the transient absorption spectra of **4** (8.0×10^{-5} M) in benzene under argon at room temperature. The band attributed to $^1K_{trans}$ in the ground state was observed at 420 nm. The absorption band for $^1K_{trans}$ of **4** in ethanol (Fig. 7e) appeared in a different shape from that in benzene because **4** exists mainly as the $^1K_{cis}$ tautomer in ethanol and the absorption spectra of $^1K_{cis}$ is overlapped with that of $^1K_{trans}$. The transient absorption spectra of **5** in each solvent were very similar to those of **4**, indicating that the effects of benzyl and the alkyl

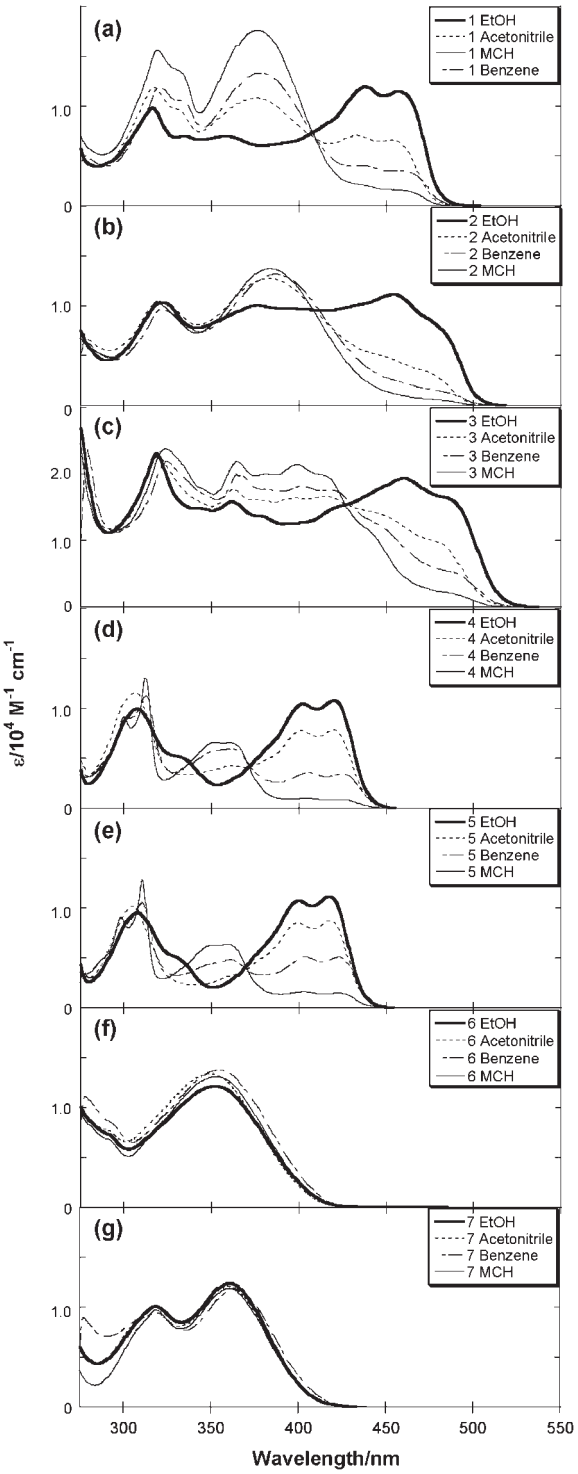


Fig. 5. UV absorption spectra of **1**–**7** in various solvents at room temperature under argon.

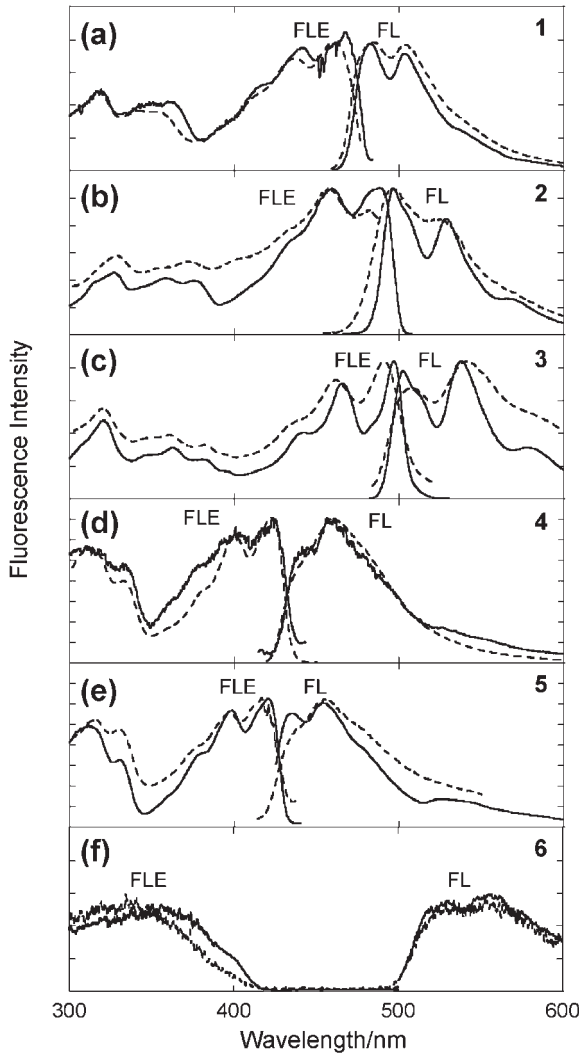


Fig. 6. Fluorescence (FL) and fluorescence excitation (FLE) spectra of compounds **1**–**6** in methylcyclohexane (dotted line) and in ethanol (solid line).

Table 1. The Absorption Maxima of the Transient Absorption Bands and Lifetimes for $^1K_{trans}$ of Compounds **1**, **2**, **4**–**6**, and **SA** in Various Solvents at Room Temperature under Argon

	In Benzene		In Acetonitrile		In Ethanol	
	λ_{max}/nm	τ/μs	λ_{max}/nm	τ/μs	λ_{max}/nm	τ/μs
1	430	220	450	1400	480	190
2	460	2300	—	—	—	—
4	420	760	440	1800	440	13
5	420	490	440	1900	440	63
6	460	28	460	630	470	55
SA	480	40	480	1500	—	—

group on the transient species are not significant. The lifetimes of the transient species ($^1K_{trans}$) of **1**–**6** in each solvent are summarized in Table 1.

Discussion

Effects of Naphthyl Group on the A Ring on Solvatochromic Behaviors. As shown in Figs. 5a–5e, compounds **1**–**5**, where naphthalene was introduced in the A ring, exhibited solvatochromism. Thus, the solvatochromic property of naphthyl analogues of salicylideneaniline at the A ring was always observed, regardless of the substituent at the B ring. The solvatochromic behavior of those compounds is based on the intramolecular hydrogen transfer in their ground states. Compounds **1**–**5** mainly exist as their enol form (^1E) in non-polar solvents and *cis*-keto form ($^1K_{cis}$) in polar solvents. A possible reason for showing solvatochromism in **1**–**5** is that the decrease of aromaticity caused by tautomerization to K_{cis} is not so large because the benzene ring is still present in the tautomer form of the naphthalene ring in **1**–**5**.[10]

Compound **6** exhibited similar absorption spectra to the parent compound SA in each solvent (Fig. 5f). In this case, the aromaticity is totally lost because a benzene ring instead of a naphthalene ring is present at the hydrogen-bonding part. These results give a clear indication that aromaticity in the A ring is important to give solvatochromic behavior in SA derivatives.

At low temperature, the fluorescence spectra of **1**–**6** were observed (Fig. 6). The peaks of the emission bands of **1**–**5** were largely dependent on the substituent groups in the B ring. On the other hand, the peaks of the emission bands in methylcyclohexane and in ethanol were not very different in each compound. These results indicate that the energy gap between the ground state and excited singlet state strongly depend on the substituent group in the B ring, whereas the solvent polarity does not strongly affect the energy gap. Compounds **1**–**5** exist as $^1K_{cis}$ in the ground state regardless of the solvents (methylcyclohexane or ethanol), which was revealed by fluorescence excitation spectra (Figs. 6a–6e), indicating that compounds **1**–**5** show thermochromism regardless of the substituents in the B ring.

Effects of Substituent in the B Ring on the Absorption Spectra. Here, we focus on the effect of each substituent group in the B ring on the absorption spectra. The substituent effect on the absorption spectra of naphthalene-based SAs was significant. Although there is no conjugation between A and B rings in the drawing of $^1K_{cis}$ in Fig. 1, the absorption spectra for $^1K_{cis}$ in polar solvents greatly changed upon the substituent groups in the B ring. With increasing the aromaticity of the aromatic ring from phenyl in **1** to the 2-anthryl in **3**, the bands for both ^1E and $^1K_{cis}$ shifted to the longer wavelength (Figs. 5a–5c), which indicates that the aromaticity is preserved throughout the whole range of molecules (**1**–**3**) in solution. On the other hand, a blue-shift of the absorption spectra was observed in compounds **4** and **5**, having benzyl or alkyl groups in the B ring. It is noted that the introduction of the naphthyl group only in the B ring of SA did not affect the absorption spectra as shown in Fig. 5g. Thus, one can say that the chromophore in the B ring largely affects the steady-state absorption spectra when naphthalene is introduced in the A ring of SA. Compound **7** did not show solvatochromism (Figs. 5g and 5h) as expected. The relevant absorption data of **1**–**7** in each solvent are shown in Table 1.

Substituent Effects on the Transient Absorption Spectra of $^1K_{trans}$. In ethanol, only depletion of the absorption spectra was observed. Compound **2** predominantly exists as a tautomer $^1K_{cis}$ in the ground state and the absorption band of $^1K_{cis}$ reaches 500 nm. If we compare the absorption spectra (Fig. 5b)

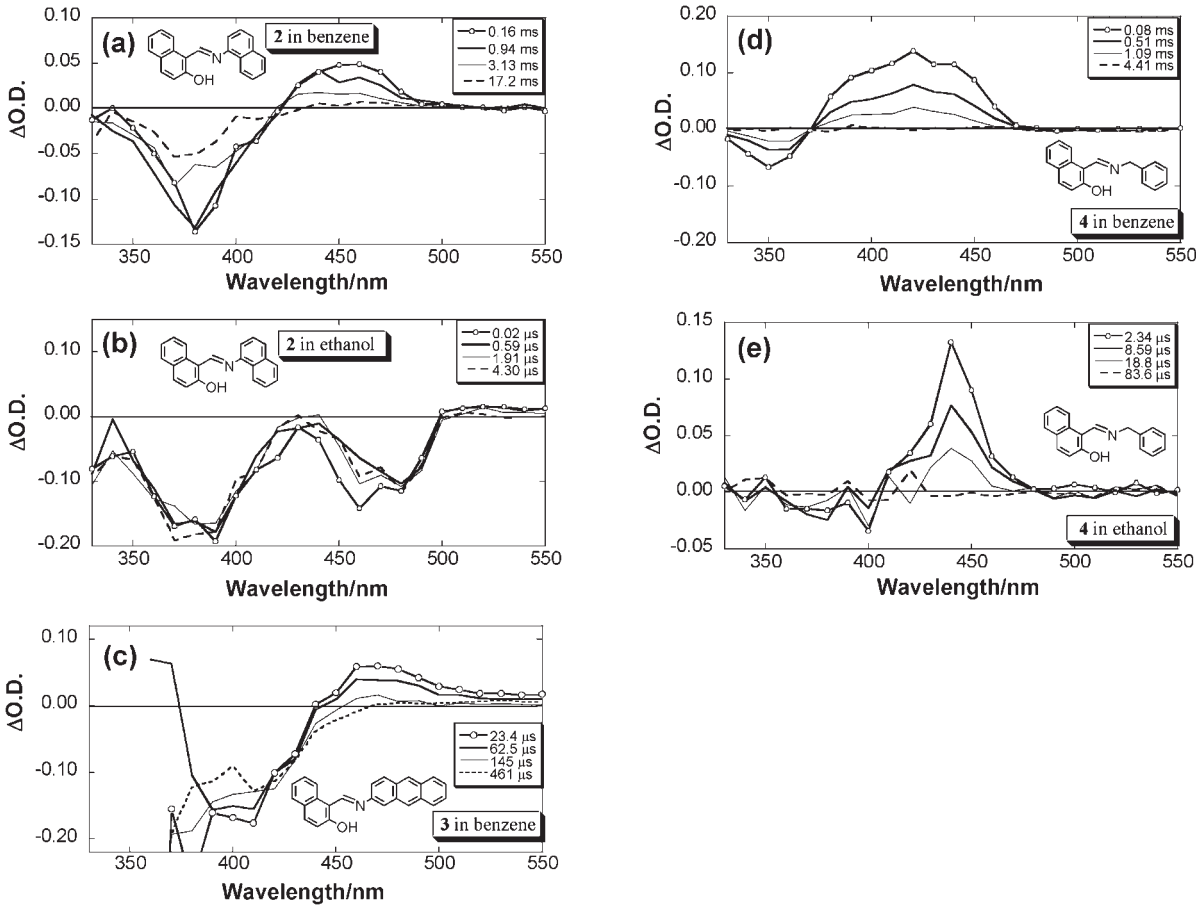


Fig. 7. The transient absorption spectra ($\lambda_{ex} = 308$ nm) of **2** in benzene (a), **2** in ethanol (b), **3** in benzene (c), **4** in benzene (d), and **4** in ethanol (e).

and the transient absorption spectra (Fig. 7b), we can estimate that the absorption maximum for $^1K_{trans}$ of **2** comes around 450 nm. The absorption band for $^1K_{trans}$ comes at a shorter wavelength than that for $^1K_{cis}$ in **2**.

On the other hand, the transient spectra peaking at 440 nm was observed for **4**, indicating that the absorption maximum for $^1K_{trans}$ of **4** exists at around 440 nm. These results indicate that the absorption spectra of $^1K_{trans}$ are not strongly affected by substituents in the B ring of SA, whereas those of both ^1E and $^1K_{cis}$ are strongly influenced in the naphthalene-based SAs **1**–**5**.

Effects of Naphthyl Group in the A Ring on the Lifetime of Transient $^1K_{trans}$. As depicted in Fig. 1, the ground-state $^1K_{trans}$ species is produced by excitation of ^1E. The following ESIPT takes place because in the excited singlet state, $^1K_{cis}$* is more stable than ^1E*. The photoisomerization of $^1K_{cis}$* gives the meta-stable $^1K_{trans}$. The lifetimes of $^1K_{trans}$ for **1**–**6** and **SA** in different solvents are summarized in Table 1. The lifetimes for **1**, **2**, and **6** have been reported previously.[17] The results suggest the existence of hydrogen bonding between nitrogen and C–H hydrogen atoms on the naphthalene ring in $^1K_{trans}$ of **1**–**5** (Fig. 4). In benzene, the lifetimes of $^1K_{trans}$ for **1**–**5** were much longer than those of **6** and SA, which may be explained by the effect of the intramolecular hydrogen bonding between nitrogen and CH hydrogen in the naphthalene ring. This intramolecular hydrogen bonding is not present in acetonitrile or ethanol, which may lead to the similar lifetimes of $^1K_{trans}$ among **1**–**6** and SA in these solvents. In acetonitrile, the lifetimes of $^1K_{trans}$ for **1**–**6** were much longer than in benzene. The keto forms $^1K_{trans}$ should be more polar than enol forms, which may result in longer lifetimes in polar solvents than in non-polar solvents. In addition, the contribution of the zwitterionic form is suggested in acetonitrile (Fig. 8).[20] On the other hand, the lifetimes in ethanol are much shorter than in acetonitrile, or even in benzene for **1**–**5**. The results indicate that the re-enolization process is considerably promoted by the intramolecular proton transfer assisted by the solvent[21] to give $^1E'$, which may immediately rotate back to the stable conformation of ^1E (Fig. 9). Thus, the introduction of a naphthyl group in the A ring of SA not only changed the ground-state properties, but also influenced the properties of transient species produced by photoinduced reaction.

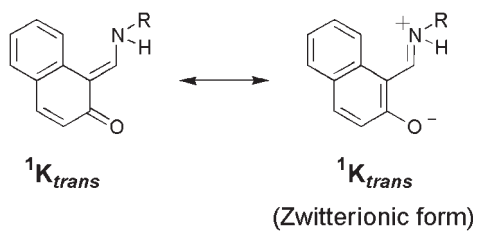


Fig. 8. Formation of zwitterion of $^1K_{trans}$ in acetonitrile.

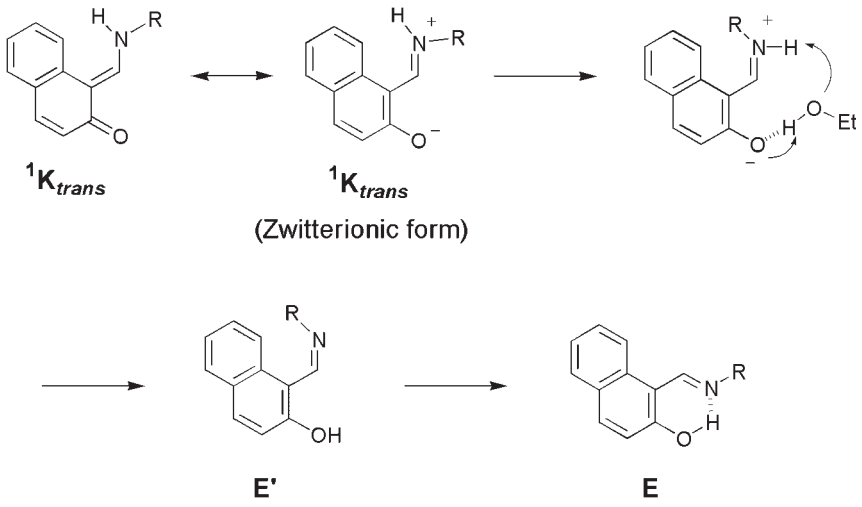
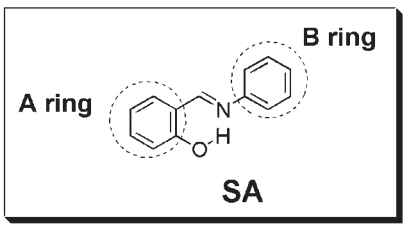


Fig. 9. Possible mechanism of solvent assisted re-enolization reaction in ethanol.



Introduced naphthyl group on

A ring

B ring

1-5

6

Absorption spectra	strongly dependent on **R**	similar to **SA**
Photochromic	Yes	Yes
Solvatochromic	Yes	No
Lifetime of $^1K_{trans}$ in benzene	much longer	similar to **SA**

Fig. 10. Comparison of the chemical properties of naphthyl analogues of SAs.

Conclusion

We described the substituent effects on the ground-state properties of naphthalene analogues of salicylideneaniline by comparison of both steady-state and transient absorption spectra of the series of compounds **1–6** (Fig. 10). The generality of the solvatochromic property of naphthyl analogues of SA has been shown by the steady-state absorption spectra. The introduction of substituents in the B ring considerably influenced their absorption spectra in various solvents, when naphthalene was substituted in the A ring. Transient absorption studies revealed that the introduction of naphthyl groups in the A ring prolonged the lifetime of $^1K_{trans}$ in non-polar solvents, probably due to the formation of intramolecular hydrogen bonding between nitrogen and C–H hydrogen on naphthyl groups. In acetonitrile, the lifetimes of **1–6** are much longer than those in benzene probably because the zwitterionic forms are stabilized in the polar solvent, whereas the much shorter lifetime in ethanol suggests that $^1K_{trans}$ undergoes the solvent assisted re-enolizaiton reaction in ethanol.

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