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Journal of Photochemistry and Photobiology A: Chemistry 162 (2004) 473–479

Journal of Photochemistry and Photobiology A:Chemistry

www.elsevier.com/locate/jphotochem

Photochromism, thermochromism, and solvatochromism of naphthalene-based analogues of salicylideneaniline in solution

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Received 4 August 2003; received in revised form 9 September 2003; accepted 20 September 2003

Abstract

A series of salicylideneaniline (**SA**) derivatives **1–3** were prepared to study the substituent effect on the chromic properties. Compounds **1** and **3**, introduced naphthyl group on the A ring in the parent compound **SA**, exhibited the solvatochromism, thermochromism and photochromism, while compound **2**, introduced the naphthyl group on the B ring in **SA**, did not show the solvatochromism or thermochromism. The discussion in this study is mainly focused on the behaviour of compounds **1** and **2**. The UV absorption spectra of **1** dramatically changed with increasing the solvent polarity as well as cooling temperature, suggesting that **1** takes *Z*-NH form in polar solvents at room temperature or even in non-polar solvents at below 120 K. Compound **1** emits fluorescence (FL) only at low temperature with small Stokes shift, while **2** exhibits fluorescence emission with considerably large Stokes shift. On UV irradiation, **1** in benzene underwent intramolecular hydrogen atom transfer to give *E*-NH form. The large Stokes shift observed in **2** indicates that **2** takes *E*-OH form even at low temperature in any solvents, and underwent intramolecular hydrogen atom transfer in the excited state to give *Z*-NH form, which emits the fluorescence at low temperature, or undergoes isomerization around C=C double bond to give *E*-NH form. The potential energy diagrams for **1** and **2** were also determined.

Keywords: Schiff bases; Salicylideneanilines; Solvatochromism; Thermochromism; Photochromism

1. Introduction

Photochemical behaviour of salicylideneaniline (**SA**) and its related compounds have been thoroughly investigated especially in the solid state [1–7].

We have recently reported that salicylideneanilines and salicylidene benzylamines exhibited chromic behaviour depending on the substituent as well as solvent, temperature and light [8,9]. Among the compounds, the parent compound salicylideneaniline exhibited photochromic behaviour in the excited triplet state. Furthermore, in salicylidene benzylamine we have observed T–T absorption spectra as well as phosphorescence spectra and propose the occurrence of hydrogen atom transfer in the excited triplet state of these compounds.

In the course of our studies, we have prepared a series of related compounds having different aromatic ring and studied their photochemical behaviours in addition to that of solvatochromic and thermochromic properties. In this paper we focus our discussion on these properties of naphthyl analogues of salicyclideneaniline. The effects of solvent and temperature on the absorption spectra for these compounds have already been reported [10–23], but the photochromic behaviours have not been reported so far. In this respect we have performed laser transient spectroscopic experiments as well as fluorescence (FL) studies of naphthyl analogues of salicyclideneaniline. We wish to report here colour change depending upon the solvent, substituent pattern, temperature and light irradiation. We studied the behaviour of compounds **1–3** (Scheme 1), but the discussion is mainly focused on the behaviour of compounds **1** and **2**.

2. Experimental

2.1. Preparation of compounds 1–3

Typical procedure: a corresponding aldehyde and amine (1.0 eq.) were dissolved to ethanol and the solution was refluxed for 3 h. After evaporation, a crude product was recrystallized twice from ethanol to give a pure product. Compounds **1** and **2** have been reported in previous paper [11,14,16].

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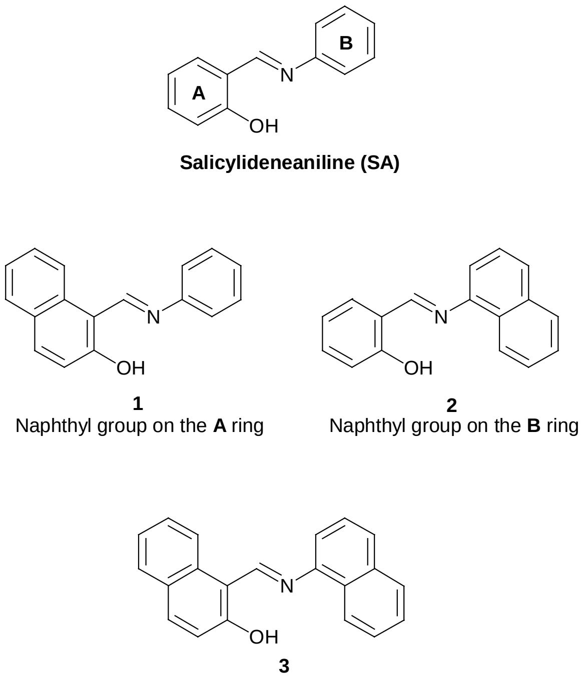
doi:10.1016/S1010-6030(03)00388-5



Salicylideneaniline (SA)

1
Naphthyl group on the **A** ring

2
Naphthyl group on the **B** ring

3
Naphthyl groups on the both **A** and **B** rings

Scheme 1.

2.1.1. 1-Phenyliminomethyl-naphthalen-2-ol (1)

1-Phenyliminomethyl-naphthalen-2-ol **(1)** was prepared from 2-hydroxy-1-naphthaldehyde and aniline in 61% yield. δ^{H} (400 MHz, $CDCl_3$, Me_4Si) 15.48 (1H, s, OH), 9.33 (1H, s, HC=N), 8.09 (1H, d, *J* 9.2 Hz, ArH), 7.80 (1H, d, *J* 9.2 Hz, ArH), 7.66 (1H, d, *J* 9.2 Hz, ArH), 7.54–7.44 (3H, m, ArH), 7.38–7.27 (4H, m, ArH), 7.08 (1H, d, *J* 9.2 Hz, ArH); Anal. Calcd. for $C_{17}H_{13}NO$: C, 82.57; H, 5.30; N, 5.66. Found: C, 82.51; H, 5.43; N, 5.64.

2.1.2. 2-(Naphthalen-1-yliminomethyl)-phenol (2)

2-(Naphthalen-1-yliminomethyl)-phenol **(2)** was prepared from 2-hydroxybenzaldehyde and 1-naphthylamine in 40% yield. δ^{H} (400 MHz, $CDCl_3$, Me_4Si) 13.37 (1H, s, OH), 8.71 (1H, s, HC=N), 8.27–8.25 (1H, m, ArH), 7.90–7.86 (1H, m, ArH), 7.78 (1H, d, *J* 8.8 Hz, ArH), 7.57–7.42 (5H, m, ArH), 7.18 (1H, d, *J* 7.2 Hz, ArH), 7.10 (1H, d, *J* 8.0 Hz, ArH), 6.98 (1H, t, *J* 7.6 Hz, ArH); Anal. Calcd. for $C_{17}H_{13}NO$: C, 82.57; H, 5.30; N, 5.66. Found: C, 82.34; H, 5.49; N, 5.28.

2.1.3. 1-(Naphthalen-1-yliminomethyl)-naphthalen-2-ol (3)

1-(Naphthalen-1-yliminomethyl)-naphthalen-2-ol **(3)** was prepared from 2-hydroxy-1-naphthaldehyde and 1-naphthylamine in 20% yield. δ^{H} (400 MHz, $CDCl_3$, Me_4Si) 15.68 (1H, s, OH), 9.47 (1H, s, HC=N), 8.33 (1H, d, *J* 7.6 Hz, ArH), 8.14 (1H, d, *J* 8.0 Hz, ArH), 7.89–7.83 (2H, m, ArH), 7.79–7.73 (2H, m, ArH), 7.58–7.50 (4H, m, ArH), 7.36–7.32 (2H, m, ArH), 7.19 (1H, d, *J* 9.6 Hz, ArH); Anal. Calcd. for $C_{21}H_{15}NO$: C, 84.82; H, 5.08; N, 4.71. Found: C, 84.71; H, 5.13; N, 4.72.

2.2. Measurements

Absorption spectra were measured on a Shimazu UV-1600. Fluorescence spectra were measured on a Hitachi F-4500. Laser flash photolysis was performed by using an excimer laser (Lambda Physik LPX-100, 308 nm 20 ns fwhm) as an 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-8123) were used for the detection.

3. Results and discussion

3.1. Absorption and fluorescence spectra

Fig. 1 shows the absorption spectra of **1**–**3** in various solvents. Similar absorption spectra of **1** were observed in the previous papers [15,16]. Compounds **1** exhibited the absorption maxima at 380 and 320 nm with small shoulder at 460 and 440 nm in methylcyclohexane. The spectral profile dramatically changed with the solvent polarity. The absorbance at longer wavelength region increased with increasing the solvent polarity with concomitant decrease of the absorbance at 380 nm. The peak at 430–460 nm becomes the highest in ethanol. However, the profile of the absorption spectra of **2** is almost unchanged in all the solvents examined. Compound **3**, where two naphthyl groups are introduced on the both A and B rings in the parent compound **SA** showed the solvatochromism. The results indicate that **1** and **3** may exist as a mixture of *trans*-enol form (*E*-OH form) and *cis*-keto form (*Z*-NH form) (Scheme 2) [14]. The equilibrium between the two tautomers shifted to the *Z*-NH form in highly polar solvents and in protic solvents. Table 1 summarised the absorption maximum of **1–3** observed in various solvents. Compounds **1** and **3**, where the naphthyl group is introduced in the aromatic ring A (Scheme 1), exhibited similar solvatochromic effect.

Fluorescence and fluorescence excitation (FLE) spectra for 1^{16}–**3** were observed at low temperature (77 K). Fig. 2 shows the fluorescence and fluorescence excitation spectra for **1–3** in methylcyclohexane and in ethanol. The fluorescence excitation spectra of **1** in both solvents were similar to the absorption spectra in ethanol at room temperature and in methylcyclohexane at low temperature. The fluorescence and fluorescence excitation spectra of **3** were similar to those of **1**. The fluorescence excitation spectra of **2** in both solvents are similar to the absorption spectra at room

Table 1
Absorption maxima (λ_{max} nm^{-1}) of **1–7** in various solvents

Compounds	MCH	Benzene	Acetonitrile	Ethanol
1	376	378, 433, 457	376, 433, 453	438, 457
2	353	355	349	352
3	384	386	382	455

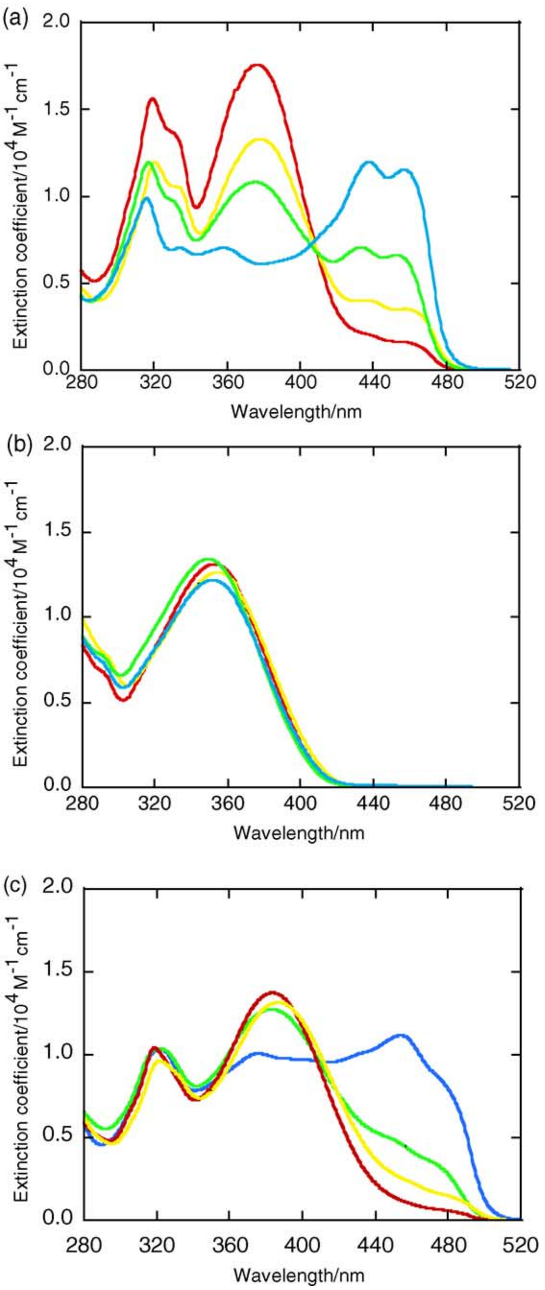
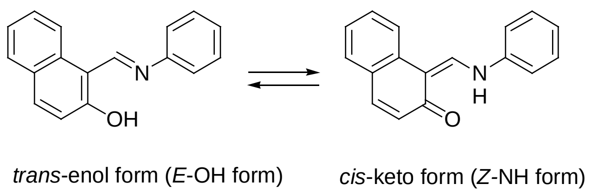


Fig. 1. UV absorption spectra of compound (a) **1**, (b) **2** and (c) **3** in methylcyclohexane (red), in benzene (yellow), in acetonitrile (green) and in ethanol (blue), respectively.



Scheme 2.

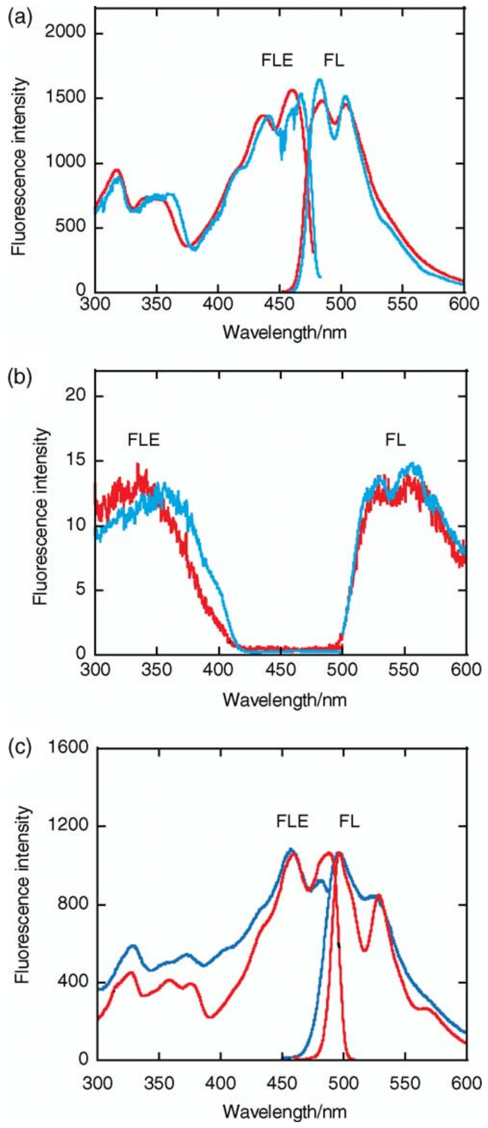


Fig. 2. Fluorescence (FL) and fluorescence excitation (FLE) spectra of compound (a) **1**, (b) **2** and (c) **3** in methylcyclohexane (red) and in ethanol (blue) at 77 K.

temperature. The Stokes shift between the maximum wavelength of fluorescence spectrum and the fluorescence excitation spectrum was only 1100 cm^{-1} for **1** and 540 cm^{-1} for **3**, respectively but is as large as 10,000 cm^{-1} for **2** (see Table 2). These results indicate that **1** and **3**, which exists as the *Z*-NH form at low temperature, absorbs light and emits fluorescence without changing the conformation, while **2,**

Table 2
Stokes shift of **1–3** in methylcyclohexane and ethanol

Compounds	Stokes shift (cm^{-1})	
	MCH	Ethanol
1	1100	670
2	11000	9200
3	540	370

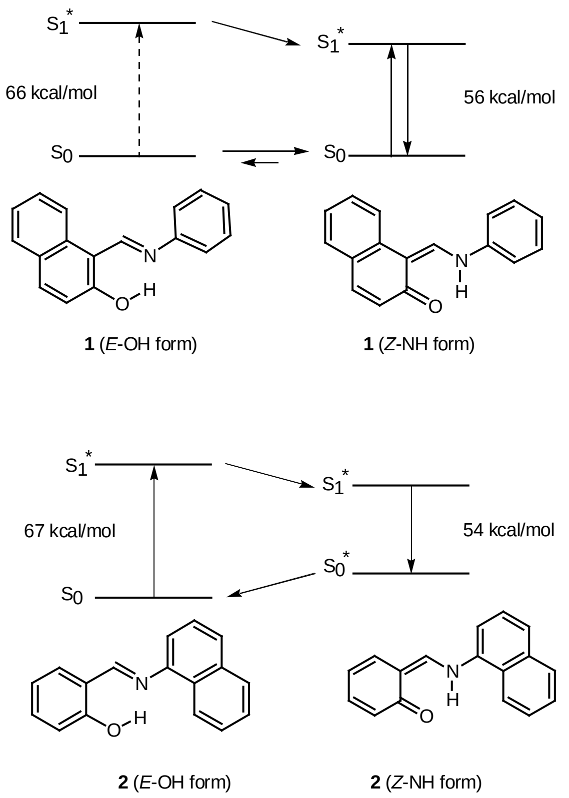


Fig. 3. Potential energy diagram of compounds **1** and **2** at 77 K.

existing as the *E*-OH form at low temperature, absorbs light and undergoes intramolecular hydrogen atom transfer to produce the *Z*-NH form in the excited singlet state, which gives the fluorescence emission. The potential energy diagrams of **1** and **2** are shown in Fig. 3.

Fig. 4 shows the change in the absorption spectra of **1** in methylcyclohexane depending on temperature. Cooling the solution causes a shift of the equilibrium between *E*-OH and *Z*-NH toward the *Z*-NH form [16]. The spectra largely changed between 200 and 140 K as shown in the inset in Fig. 4 [21]. The sudden change of the absorption spectra at 160–180 K cannot be explained by the simple mechanism of equilibration between the *E*-OH form and *Z*-NH form, but can be explained by the sudden aggregation of the **1** at this temperature range to induce tatomerizm in the ground state as proposed by Ogawa et al. [21].

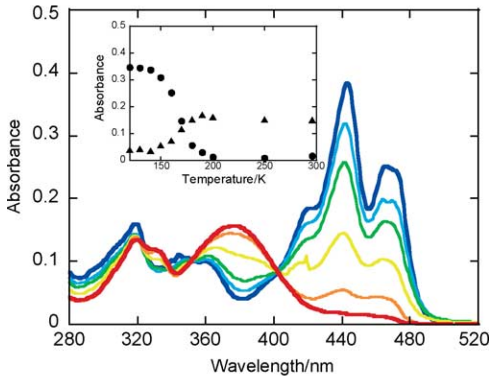


Fig. 4. Temperature dependence of absorption spectra of **1** in methylcyclohexane: 298 K (red), 180 K (orange), 170 K (yellow), 160 K (green), 150 K (light blue) and 120 K (blue). Inset shows temperature vs. absorbance at (●) 443 nm and (▲) 377 nm.

3.2. Transient absorption spectra

On 308 nm laser excitation, **1** exhibited the transient absorption spectra at room temperature under argon atmosphere as shown in Fig. 5. Compound **1** showed a new absorption peaking at 400–500 nm with the depletion at 350–400 nm in each solvent. The transient species appeared

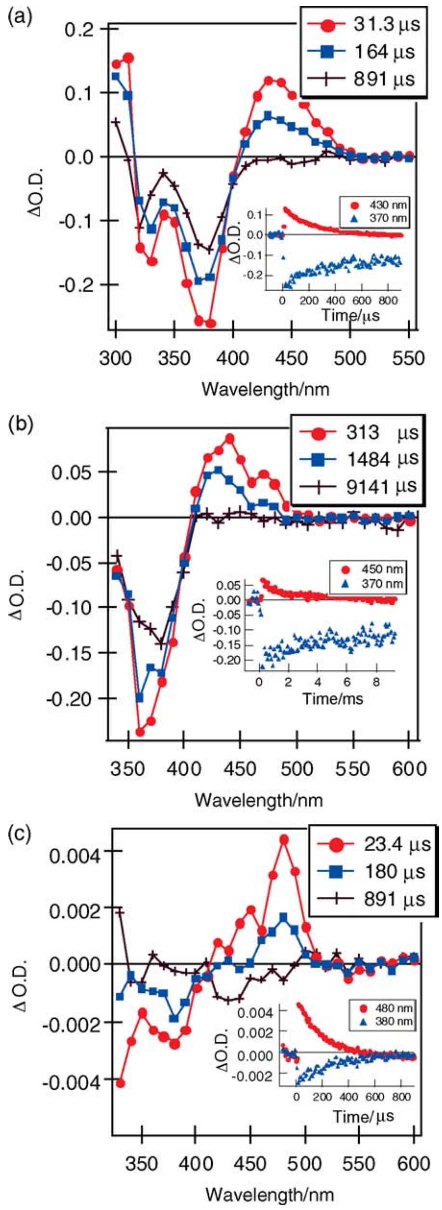


Fig. 5. Transient absorption spectra of **1** in (a) benzene, (b) in acetonitrile and (c) in ethanol, respectively on excitation at 308 nm under argon. Inset shows the time profile at certain wavelength.

at 400–500 nm in each solvent was not quenched by oxygen, so that it can be ascribed to the ground state tautomer, either *Z*-NH form or *E*-NH form. The transient absorption spectra observed at 400–500 nm in benzene and in acetnitrile are similar to the absorption spectrum observed in ethanol as a *Z*-NH form (Fig. 1a). Therefore, the observed ground state species by laser flash photolysis could be assigned to the *Z*-NH form (Fig. 5a and b). However, in ethanol, the long lifetime of the observed ground state tautomer indicates that the transient spectra can be assigned to *E*-NH form because of the new absorption peaking at 480 nm which is longer wavelength than that of ground state of *Z*-NH form of **1** in ethanol (Fig. 5c). The decay curves for the transient peaking at 400–500 nm fit the single exponential function to give the time constant of 220 μs in benzene, 1400 μs in acetonitrile and 190 μs in ethanol, respectively (inset in Fig. 5). After the decay of the signal at 400–500 nm, the signals of depletion at 350–400 nm remain in the time range of ms in benzene and in acetonitrile (inset in Fig. 5a and b) [8], while the signal at 350–400 nm in ethanol decreased simultaneously with that at 400–500 nm (inset in Fig. 5c). In addition, we could not observe any permanent change of the absorption spectra by conventional UV-Vis spectroscopy. Therefore, the seemingly permanent change at shorter wavelength region observed in benzene and acetonitrile is ascribable to the *Z*-OH isomer, given by *E*–*Z* photochemical isomerization around the C=N double bond from the excited state of *E*-OH form. On the other hand, **1** in ethanol apparently takes *Z*-NH form in the ground state. Since the photo-excitation of **1** in ethanol did not give the long lifetime species (*Z*-OH form), the excited state of *Z*-NH form is more stable than that of *E*-OH form.

Fig. 6 shows the transient absorption spectra of **2** in ethanol. Unlike the transient spectra of **1** in ethanol, compound **2** showed the signal of depletion at 380 nm with the lifetime of milliseconds after the decay of the signals at 400–500 nm. The transient absorption spectra of **2** in benzene and in acetonitrile were similar to that in ethanol.

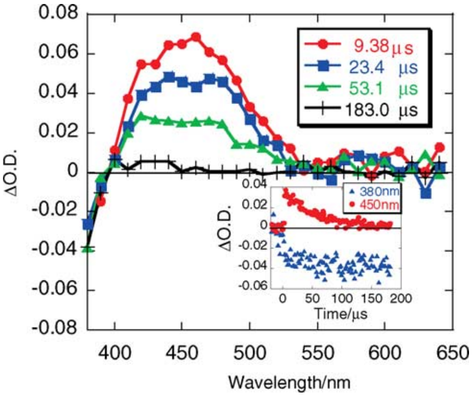


Fig. 6. Transient absorption spectra of **2** in ethanol on excitation at 308 nm under argon. Inset shows the time profile at certain wavelength.

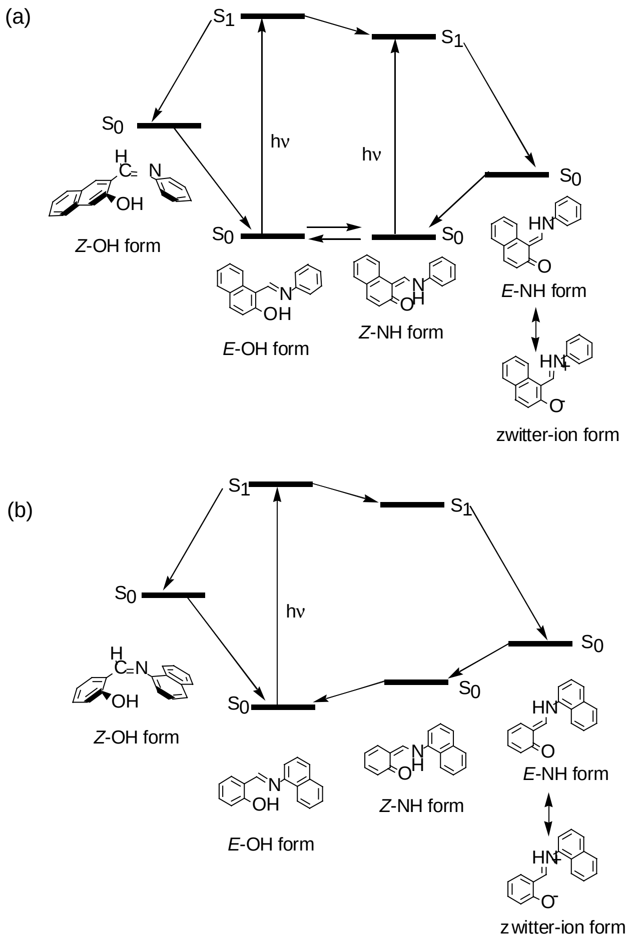


Fig. 7. Potential energy diagram of (a) **1** and (b) **2** at room temperature.

3.3. Potential energy diagram

In addition to Fig. 3, the transient spectroscopies suggest the potential energy diagram of hydrogen atom transfer of **1** and **2** at room temperature as shown in Fig. 7.

At room temperature **1** exist as a mixture of *E*-OH form and *Z*-NH form and the equilibrium is shifted to the *Z*-NH form with increasing the solvent polarity and in protic solvent. In the excited singlet state *Z*-NH form is more stable than that of *E*-OH form from so that the long lifetime peaking at 350–400 nm, which was ascribed to *Z*-OH form, was observed in benzene and in acetonitrile but not in ethanol. Compound **2** exists as the *E*-OH form regardless of the solvent properties probably because the ground state of *Z*-NH form is less stable than that of *E*-OH form in **2**. This is in agreement with the results shown in Fig. 3.

Fluorescence spectra were not observed at room temperature for any compounds in any solvent. However, at 77 K fluorescence emission peaking at 485 and 494 nm for **1** was observed in methylcyclohexane and in ethanol, respectively. The considerably small Stokes shift indicates that **1** exists as the *Z*-NH form in the ground state at 77 K even in methylcyclohexane and the fluorescence is emitted from the same

NH-form for **1**. The observation of the fluorescence emission, peaking at 530 and 560 nm with large Stokes shift, indicates that **2** exists as a *E*-OH form in the ground state even at 77 K and the hydrogen atom transfer should take place in the excited singlet state to produce the *Z*-NH form, which emits fluorescence with large Stokes shift.

Both **1** and **2** gave fluorescence emission of the *Z*-NH form indicating that in the excited singlet state the *Z*-NH form is more stable than the *E*-OH form for both **1** and **2**. In the ground state, **1** exists as a mixture of *E*-OH form and the *Z*-NH form depending on the solvent and temperature, while **2** exists as the *E*-OH form irrespective of the solvent and temperature.

3.4. Comparison with the parent compound, salicylideneaniline

The photochemical properties of **1–3** were compared with **SA**. Three points are noteworthy:

(i) **SA** and **2** did not show the solvatochromism, while **1** and **3**, introduced naphthyl group on the A ring of **SA** (Scheme 1), showed the change in absorption spectra depending on the solvent polarity, suggesting the *Z*-NH tautomer in high polar solvents was stabilized by the naphthyl group on the A ring of **SA**.

(ii) **SA** and **2** did not show the thermochromism. The absorption spectra of **1** in methylcyclohexane dramatically changed with cooling temperature especially in the range from 200 to 140 K, which indicates that the ground state of the *Z*-NH form of **1** is more stable than that of *E*-OH form below 140 K.

(iii) The absorption maximum and the lifetime of transient absorption on excitation at 308 nm light in benzene were 480 nm and 40 ms for **SA**, 460 nm and 28 ms for **2**, 440 nm and 220 ms for **1**, and 440 nm and 2200 ms for **3**, respectively. Thus, the introduction of naphthyl group on the A ring elongated the transient lifetime due to the *E*-NH form.

The observed differences of the photochemical properties in **SA**, **1**, and **3** were mostly due to the effect of the introduction of naphthyl group on the A ring of **SA** to give the similar ground state energy between *E*-OH form and *Z*-NH form in **1** as shown in Fig. 7. The introduction of naphthyl group on the B ring in **2** scarcely affect the photochemical properties of **SA**. The ground state energy of *Z*-NH form in **SA** and in **2** should be higher than that of *E*-OH form and therefore, **SA** or **2** did not show the solvatocromism or thermocromism in the ground state.

4. Conclusion

Compounds **1** and **3**, introduced naphthyl group on the A ring in the parent compound salicylideneaniline, exhibited the strong substituent effect on their photochemical properties. In this study we explored **1** as the typical example, which showed the solvatochromism, thermochromism and photochromism. The UV absorption spectra of **1** dramatically changed with increasing the solvent polarity as well as cooling temperature, suggesting that **1** takes *Z*-NH form in polar solvents at room temperature or even in non-polar solvents at below 120 K. Compound **1** emits fluorescence only at low temperature with small Stokes shift. The transient lifetime of **1** was much longer than that of **SA** in benzene. Compound **2**, on the other hand, introduced the naphthyl group on the B ring in **SA**, did not show the solvatochromism or thermochromism. The Stokes shift of **2** was much larger than that of **1**, indicates that **2** takes *E*-OH form even at low temperature in any solvents, and underwent intramolecular hydrogen atom transfer in the excited state to give *Z*-NH form, which emits the fluorescence at low temperature, followed by isomerization around C=C double bond to give *E*-NH form.

Acknowledgements

This work was supported by a Grant-in-Aid for Scientific Research on Priority Areas (417) and the 21 Century COE Program from the Ministry of Education, Culture, Sports, Science and Technology (MEXT) of the Japanese Government and by the Asahi Glass Foundation.

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