

Time-resolved fluorescence of α -(9-anthryl)- ω -(1-naphthyl)-oligosilanes: intramolecular electronic energy and charge transfer through π - π and σ - π interactions

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Received 26 August 2003; accepted 16 December 2003

Abstract

The photophysical properties of a series of α , ω -diaryloligosilanes: (9-anthryl)-(SiMe₂)_n-(1-naphthyl) (**1–4**, and **6**), $n = 1, 2, 3, 4$, and **6**, were investigated. For $n \geq 2$, the intramolecular excimer emission was observed in cyclohexane. For the stationary and the time-resolved fluorescence (TR-FL) measurements, the excitation of the naphthyl moiety showed a very efficient intramolecular energy transfer to the anthryl moiety, thus only fluorescence from the anthryl moiety was observed. In the case of **4** and **6**, a charge-transfer (CT) emission was observed in acetonitrile. Then TR-FL of **6** revealed that the intramolecular-energy transfer from the naphthyl to anthryl moieties is faster than the CT.

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Keywords: Organosilicon oligomer; Energy transfer; Charge transfer; Time-resolved fluorescence

1. Introduction

Photophysical processes of aryldisilanes have been extensively investigated in order to elucidate the interactions between the aryl group and disilane unit. The origin of this interaction is proposed as a σ - π interaction between the π -orbital of the aryl group and the Si-Si σ -orbital or 3d- π -orbital [1–4]. Research is still continuing to elucidate such a mechanism.

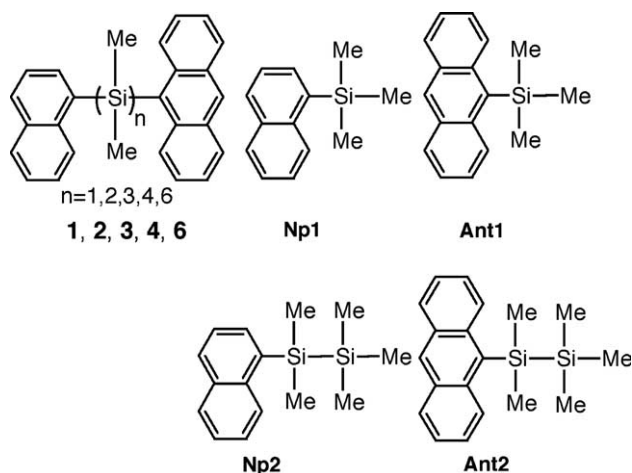
Our interests include making σ - π conjugated systems using selected aryl groups such as naphthyls and/or anthryls and an oligosilane linker unit, and to elucidate photophysical processes such as excimer/excimer formations, intramolecular charge-transfer (CT), and electronic energy transfer processes, and comparing those to the carbon analogues. There is a series of reports on α , ω -dipyrenyloligosilanes concerning excimer formation [5,6]. Our preliminary report focused on two series of oligosilanes, (1-naphthyl)-(SiMe₂)_n-(1-naphthyl) and

(9-anthryl)-(SiMe₂)_n-(9-anthryl) [7]. These compounds showed a π - π interaction between two aromatic groups and a σ - π interaction between the aromatics and silicon chain unit. In these cases, intramolecular excimer emission was observed in cyclohexane when $n \geq 2$. The strongest excimer emission of $n = 2$ is in contrast to the Hirayama-rule ($n = 3$) proposed for carbon analogues [8]. 1,3-Di(9-anthryl)propane derivatives produce an efficient intramolecular [4+4] cycloaddition, and therefore, they produce no excimer fluorescence [9,10], however, the 1, n -di(9-anthryl)oligosilanes showed a strong excimer fluorescence indicating that the intramolecular cycloaddition is a minor process. Only di(9-anthryl)dimethylsilane is reported to produce a unique intramolecular [2+4] cycloadduct [11–13]. We also reported that the CT abilities are higher for the naphthyl than anthryl oligosilanes, therefore, CT emission was observed for the α , ω -di(1-naphthyl)oligosilanes, $n \geq 4$ and α , ω -di(9-anthryl)oligosilanes, $n \geq 6$, in acetonitrile.

In this study, we would like to report the photophysical properties of a series of α , ω -diaryloligosilanes, **1**, **2**, **3**, **4**, and **6**; (9-anthryl)-(SiMe₂)_n-(1-naphthyl),

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Scheme 1. Structures of compounds examined in this study.

$n = 1, 2, 3, 4,$ and 6 . This especially includes the intramolecular electronic energy and charge transfer processes through π - π and σ - π interactions for these compounds (see Scheme 1).

2. Experimental

2.1. Materials

All reactions were carried out under nitrogen, using oven-dried glassware. Tetrahydrofuran (THF) and diethyl ether were dried over sodium under nitrogen and distilled just before use. Dichlorodimethylsilane and 1,2-dichlorotetramethyldisilane were purchased (Shin-Etsu and Aldrich, respectively), while the other dichloro-oligosilanes ($n = 3, 4, 6$) were synthesized as already reported [14].

1-(9-anthryl)-1-(1-naphthyl)dimethylsilane (**1**). 1-Bromonaphthalene (1.55 g, 7.5 mmol) in a THF/diethyl ether mixture (8:5) was cooled in an acetone/dry-ice bath and 6.1 ml of a 1.6 M solution of *n*-butyllithium in hexane was slowly added to the solution. The solution was stirred at -50 °C. A 0.97 g (7.5 mmol) sample of dichlorodimethylsilane in THF (5 ml) was then slowly added to the reaction mixture, and the solution was stirred for 6 h at room temperature. A solution of 9-lithioanthracene prepared from 9-bromoanthracene (1.89 g, 73 mmol) was cooled in an acetone/dry-ice bath, and a solution of (1-naphthyl) chlorodimethylsilane was added. The mixture was stirred for overnight at room temperature. After workup, the product was separated by flash chromatography (hexane/benzene = 1/1) and purified by recrystallization in hexane. **2**, **3**, **4**, and **6** were prepared in a similar way using 1,*n*-dichloro-oligosilanes ($n = 2, 3, 4,$ and 6). The yields based 9-bromoanthracene were 5–17%. The data for sample characterization are described below (using JEOL SX-400 NMR spectrometer, Perkin-Elmer PE-2400

elemental analysis instrument, and JEOL JMS-HX110 mass spectrometer).

1: M.p. 150–151 °C. Anal. Calc. for $C_{26}H_{22}Si_1$: C, 86.14; H, 6.12%. Found: C, 85.84; H, 6.00%. 1H -NMR (400 MHz, $CDCl_3$) $\delta = 1.04$ (6H, s, $-CH_3$), 7.13 (1H, t, $J = 6.9$ Hz, Ar-H), 7.25–7.41 (5H, m, Ar-H), 7.50 (1H, t, $J = 6.8$ Hz, Ar-H), 7.80 (1H, d, $J = 7.7$ Hz, Ar-H), 7.82 (1H, d, $J = 7.5$ Hz, Ar-H), 7.88 (1H, d, $J = 8.2$ Hz, Ar-H), 7.95 (1H, d, $J = 6.8$ Hz, Ar-H), 8.01 (1H, d, $J = 8.2$ Hz, Ar-H), 8.43 (1H, d, $J = 8.9$ Hz, Ar-H), 8.52 (1H, s, Ar-H). ^{29}Si -NMR ($CDCl_3$): $\delta = -8.95$.

2: M.p. 180–181 °C. Anal. Calc. for $C_{28}H_{28}Si_2$: C, 79.94; H, 6.71%. Found: C, 79.79; H, 6.70%. 1H -NMR (400 MHz, $CDCl_3$) $\delta = 0.52$ (6H, s, $-CH_3$), 0.70 (6H, s, $-CH_3$), 7.12 (1H, t, $J = 7.6$ Hz, Ar-H), 7.19–7.52 (6H, m, Ar-H), 7.62 (1H, d, $J = 6.9$ Hz, Ar-H), 7.81 (1H, d, $J = 7.2$ Hz, Ar-H), 7.83 (1H, d, $J = 8.0$ Hz, Ar-H), 7.87 (1H, d, $J = 8.5$ Hz, Ar-H), 7.96 (2H, d, $J = 8.0$ Hz, Ar-H), 8.20 (2H, d, $J = 9.0$ Hz, Ar-H), 8.41 (1H, sz, Ar-H). ^{29}Si -NMR ($CDCl_3$): $\delta = -19.28, -21.69$.

3: M.p. 115–116 °C. Anal. Calc. for $C_{30}H_{34}Si_3$: C, 75.25; H, 7.16%. Found: C, 75.14; H, 7.13%. 1H -NMR (400 MHz, $CDCl_3$) $\delta = 0.27$ (6H, s, $-CH_3$), 0.37 (6H, s, $-CH_3$), 0.56 (6H, s, $-CH_3$), 7.23–7.42 (8H, m, Ar-H), 7.71 (1H, t, $J = 8.2$ Hz, Ar-H), 7.77 (1H, d, $J = 8.2$ Hz, Ar-H), 7.89 (1H, d, $J = 8.2$ Hz, Ar-H), 7.95 (2H, d, $J = 8.5$ Hz, Ar-H), 8.14 (2H, d, $J = 8.9$ Hz, Ar-H), 8.37 (1H, s, Ar-H). ^{29}Si -NMR ($CDCl_3$) $\delta = -18.75, -19.84, -43.03$.

4: M.p. 89–90 °C. Anal. Calc. for $C_{32}H_{40}Si_4$: C, 71.57; H, 7.51%. Found: C, 71.59; H, 7.56%. 1H -NMR (400 MHz, $CDCl_3$) $\delta = 0.01$ (6H, s, $-CH_3$), 0.02 (6H, s, $-CH_3$), 0.48 (6H, s, $-CH_3$), 0.68 (6H, s, $-CH_3$), 7.35–7.47 (7H, m, Ar-H), 7.57 (1H, d, $J = 6.8$ Hz, Ar-H), 7.80 (1H, d, $J = 8.0$ Hz, Ar-H), 7.82 (1H, d, $J = 7.9$ Hz, Ar-H), 7.92 (1H, d, $J = 7.7$ Hz, Ar-H), 7.97 (2H, d, $J = 8.0$ Hz, Ar-H), 8.24 (2H, d, $J = 9.0$ Hz, Ar-H), 8.40 (1H, s, Ar-H). ^{29}Si -NMR ($CDCl_3$): $\delta = -18.31, -19.38, -40.4, -42.57$.

6: M.p. 72–73 °C. Anal. Calc. for $C_{36}H_{52}Si_6$: C, 66.18; H, 8.02%. Found: C, 65.58; H, 8.07%. HRMS (FAB, *m*-nitrobenzyl alcohol was used as matrix). Calcd *m/z* for $C_{36}H_{52}Si_6$: 652.2685. Found: 652.2696. 1H -NMR (400 MHz, $CDCl_3$) $\delta = -0.09$ (6H, s, $-CH_3$), -0.04 (6H, s, $-CH_3$), 0.14 (6H, s, $-CH_3$), 0.20 (6H, s, $-CH_3$), 0.54 (6H, s, $-CH_3$), 0.75 (6H, s, $-CH_3$), 7.26–7.48 (H, m, Ar-H), 7.61 (1H, d, $J = 6.9$ Hz, Ar-H), 7.80–7.85 (1H, m, Ar-H), 7.94–8.00 (1H, m, Ar-H), 8.28–8.30 (H, m, Ar-H), 8.42 (1H, s, Ar-H). ^{29}Si -NMR ($CDCl_3$) $\delta = -18.34, -19.23, -38.32, -38.40, -39.46, -41.75$. No contamination of siloxane was confirmed by the spectroscopy.

1-Trimethylsilylnaphthalene (**Np1**), 9-trimethylsilylanthracene (**Ant1**), 1-pentamethyldisilanyl naphthalene (**Np2**), and 9-pentamethyldisilanyl anthracene (**Ant2**). **Np1**, **Ant1**, **Np2**, and **Ant2** were prepared as previously reported [1,15].

2.2. Spectroscopy

The absorption spectra and fluorescence spectra were measured in spectroscopic grade c-hexane or acetonitrile (Dotite) using a JASCO V-570 spectrophotometer and an FP-6600 spectrofluorimeter, respectively. For the fluorescence measurements, sample solutions were passed through and purged with dry argon for at least 15 min prior to the measurement.

The fluorescence decay curves and time-resolved fluorescence spectra were measured with a picosecond pulse laser and a single-photon timing apparatus at Hokkaido University [16,17]. The laser system was a mode-locked Ti:sapphire laser (Coherent, Mira 900) and pumped by an argon ion laser (Coherent, Innova 300) combined with a pulse picker (Coherent, model 9200, repetition rate of 2.9 MHz). The third harmonics (280 nm) generated by an ultrafast harmonic system (Inrad, model 5-050) were used as the excitation light source. The decay curve was obtained by monitoring the emission with a microchannel-plate photomultiplier (Hamamatsu R2809U-01). The pulse width of the instrumental response function was 30 ps (fwhm). The picosecond time-resolved fluorescence spectra were obtained from the decay curves monitored at different wavelengths ranging from 300 to 600 nm with a 0.625 nm step. The time-resolved spectrum was constructed from a series of decay curves by plotting the fluorescence intensities at the given delay times as a function of the emission wavelength.

3. Results and discussion

Figs. 1(a) and (b) show the absorption spectra of **1–4** and **6** in cyclohexane and acetonitrile, respectively. The red shifts (5 nm) of absorption maxima were observed for the band around 300–400 nm when the silicon-linker lengths became 1–2, however, no significant shift was observed by further elongation. This shift is explained by the σ - π conjugation between the π -orbital of the naphthalene or anthracene ring and the σ -orbital of the Si-Si bond. In addition, increases in the molar extinction coefficient (ϵ) were observed around 275 nm when $n = 4$ and 6. This is due to the appearance of the absorption of the silicon-linker unit by the overlapping absorption of the naphthalene unit. The absorptions of the linker unit shifted to longer wavelengths by increasing the chain lengths by σ -conjugation [7].

Figs. 2(a) and (b) show the fluorescence spectra of **1–4** and **6** in argon-purged cyclohexane by excitation of an anthryl chromophore at 375 nm and an excitation of a naphthyl chromophore at 290 nm, respectively. All spectra were normalized to their intensities at the maxima for ease of comparison. Their relative intensities

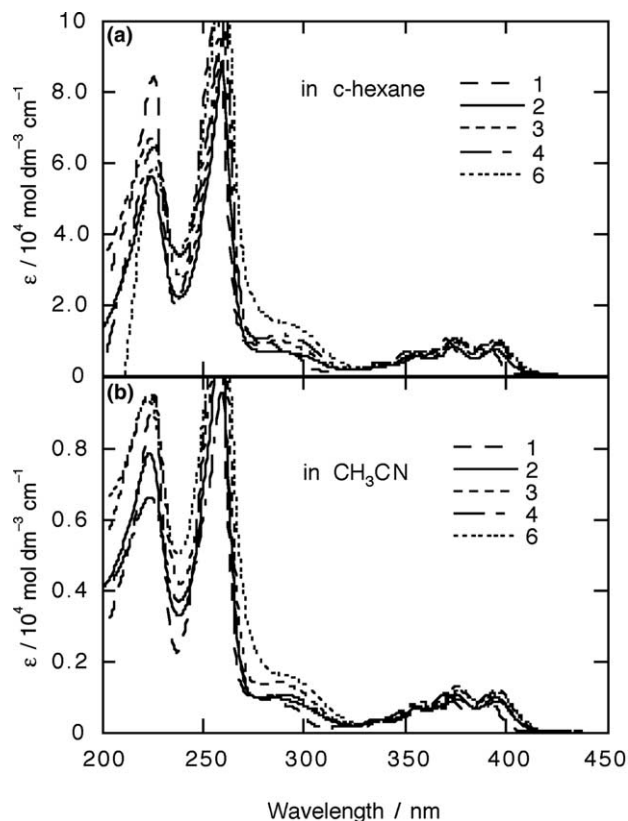


Fig. 1. Absorption spectra of **1–4**, and **6** in c-hexane (a) and acetonitrile (b).

were easily recognized by the fluorescence quantum yields (Φ_f) as shown in Table 1.

When the excitation is performed at the naphthyl moiety, it is surprising that the energy transfer efficiently occurred from the naphthyl to anthryl moiety, therefore, the fluorescence spectra were observed only from the anthryl moiety and not from the naphthyl moiety. The spectra obtained by the excitation at 290 nm were identical to the spectra obtained by the excitation at 375 nm. It is surprising that the energy transfer efficiencies from the naphthyl to anthryl moiety are almost unity within the experimental uncertainty for all the examined compounds.

This phenomenon is not only the case observed by conventional spectroscopy but also observed by TR-FL. Figs. 3(a)–(c) show the TR-FL obtained for **1**, **3**, and **6** by the excitation at 280 nm in argon-purged c-hexane. These spectra were identical to the spectra obtained by the excitation at 375 nm. Only the fluorescence from the anthryl moiety was observed just after the laser pulse for even **6**, which has the longest linker unit, and no emission from the naphthyl moiety was observed. The carbon chain analogue of **6** showed an emission from the naphthyl moiety [16,17], and a detailed study indicates that the energy transfer occurs by a through-bond interaction. The reason for this fast energy transfer for the aryloligosilanes system is under consideration by

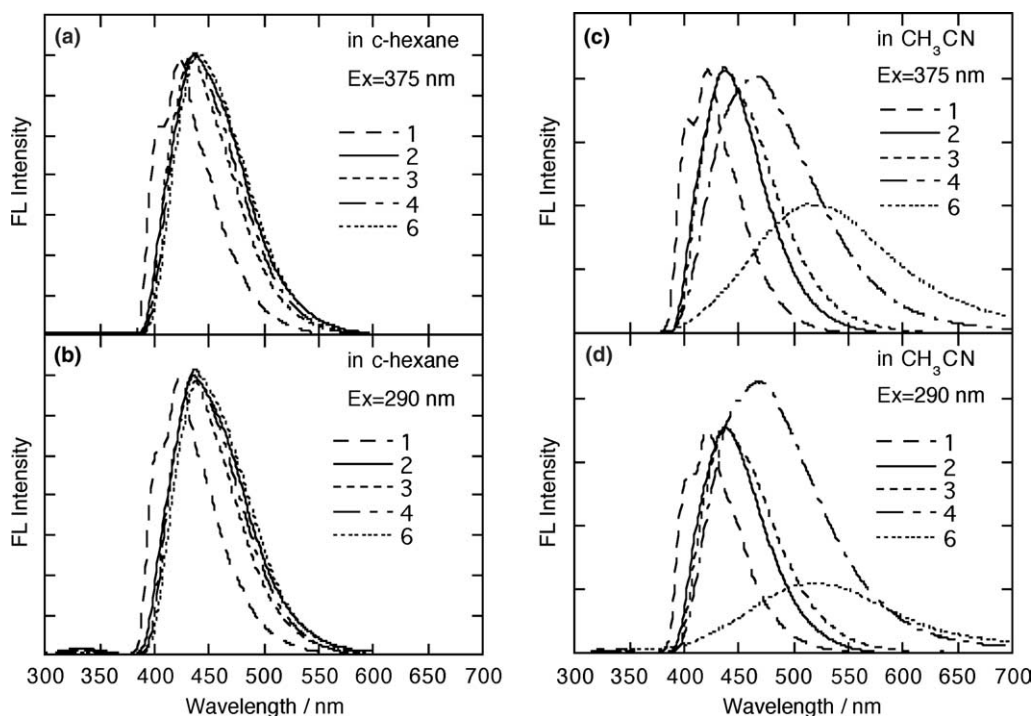


Fig. 2. Fluorescence spectra of **1–4**, and **6** excited at the anthryl moiety (375 nm, a and c) and naphthyl moiety (290 nm, b and d) in argon-purged c-hexane (a and b) or acetonitrile (c and d).

Table 1

Excitation-wavelength and solvent dependences of fluorescence quantum yield^a under argon

Compound	In c-hexane		In CH ₃ CN	
	Φ_f^{375b}	Φ_f^{290b}	Φ_f^{375b}	Φ_f^{290b}
1	0.72	0.74	0.62	0.55
2	0.67	0.61	0.21	0.20
3	0.66	0.59	0.17	0.12
4	0.65	0.51	0.080	0.073
6	0.50	0.49	0.017	0.012

^a Φ_f was determined using 1,2-benzanthracene ($\Phi_f = 0.19$ and 0.22 in nonpolar and polar solvent, respectively) as standard [18].

^b Superscript indicates the excitation wavelength.

comparing the proposed intramolecular energy transfer mechanisms [19], however, one possibility is the strong interaction between one of the aryl groups and silane linker unit (which is also acting as a chromophore) conjugate in the LUMO orbital. Such theoretical investigations are now underway.

The fluorescence quantum yields decrease by increasing the silicon chain length. This may be caused by the increasing vibrational deactivation paths due to the increasing silicon chain lengths, and further investigation is now in progress.

The fluorescence spectra of **1** in either solvent were identical but were different from the others (**2–4**, **6**) having the maxima at 15 nm shorter wavelengths with a shoulder of vibrational fine structure (Fig. 2).

To clarify the origin of the longer shift and lost fine structure of the fluorescence spectra for **2–4** and **6**, 1-trimethylsilyl- and 1-pentamethyldisilyl-naphthalene (**Np1** and **Np2**, respectively), and 9-trimethylsilyl- and 9-pentamethyldisilyl-anthracene (**Ant1** and **Ant2**, respectively) were examined. The absorption spectra of the four model compounds are shown in Fig. 4(a). The absorption band of A_u symmetry at 220 and 255 nm shifted a couple nanometers, and the B_u symmetry at 285 and 390 nm shifted about 10 nm to the longer wavelengths by the σ - π conjugation.

The fluorescence spectra of the model compounds **Np1** and **Ant1** were compared with **1** as shown in Fig. 4(b). Also, the spectra of **Np2** and **Ant2** were compared with **2** as shown in Fig. 4(c). **Np1** and **Np2** have absorptions of much shorter wavelengths than **Ant1** and **Ant2**. The fluorescence of **1** is essentially the same as that of **Ant1** indicating the origin is the local emission (LE) of the anthrylsilyl moiety. However, the fluorescence from **2** is different from the emission of **Ant2**. The emission from **2** was broad and was composed of the LE emission similar to **Ant2** and the exciplex-emission which has a maximum at 475 nm (estimated by the differential spectrum obtained by [spectrum of **2**]-[spectrum of **Ant2**]). These exciplex emissions were also observed for **3**, **4**, and **6**, however, the Stokes shifts were small and the shapes were similar to each other (Figs. 2(a) and (b)). These characteristics were in contrast to the reported behaviors of the α,ω -di(1-naphthyl)oligosilanes and α,ω -di(9-anthryl)oligosilane

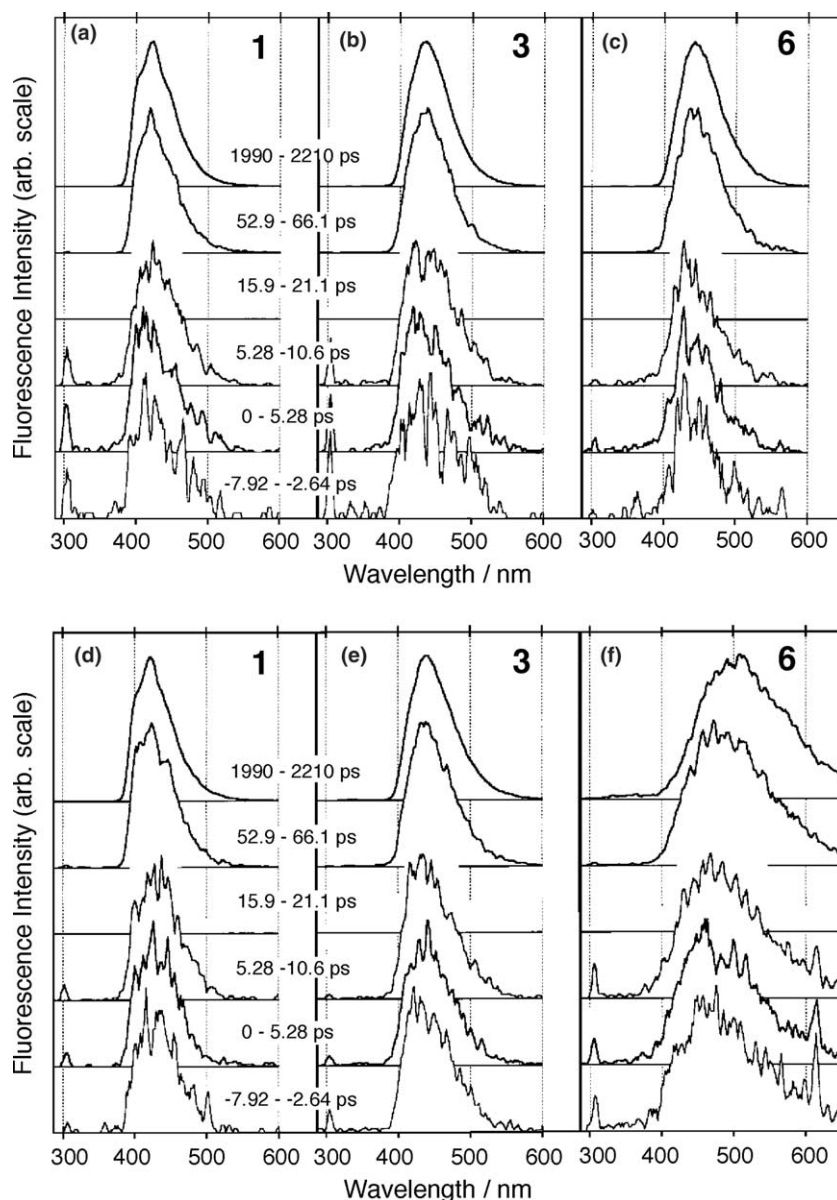


Fig. 3. TR-FL spectra of **1**, **3**, and **6** in argon-purged *n*-hexane (a–c) and acetonitrile (d–f). The excitation was performed at 280 nm.

[7]. The carbon chain analogues of **2**, **3**, and **4** also showed a weak and small shifted exciplex emission, indicating the inefficient stacking of the anthryl and naphthyl moieties [20].

Figs. 2(c) and (d) show the fluorescence spectra of **1–4** and **6** in argon-purged acetonitrile by excitation at 375 and 290 nm, respectively. In our previous report on the (1-naphthyl)-(SiMe₂)_n-(1-naphthyl) and (9-anthryl)-(SiMe₂)_n-(9-anthryl), a CT emission was observed for $n \geq 3$ for the α, ω -di(1-naphthyl)oligosilane, and $n \geq 4$ for the α, ω -di(9-anthryl)oligosilane [7], therefore, the naphthyl moiety has a higher ability of CT-interaction between silicon linker unit than the anthryl moiety. In the series of α -anthryl- ω -naphthyloligosilanes examined in this experiment, the oligosilanes, $n \geq 3$, are expected

to have the ability to produce a CT emission. However, only **4** and **6** had CT emissions. This indicates that the excitation of the naphthyl moiety did not induce the CT interaction immediately (Fig. 5(a)). However, very fast energy transfer occurred from the naphthyl to anthryl moieties, then charge transfer occurred from the silicon linker to the anthryl moiety (Fig. 5(b)). This was also supported by the fluorescence quantum yields. The Φ_f values in acetonitrile were much smaller than those in cyclohexane even for compound **1**. The yield suddenly drops one-third when the linker length $n = 2$, then the values gradually decrease by elongation of the chain length. The Φ_f^{290} values are almost identical to Φ_f^{375} , and it is reasonably explained by the mechanism that CT emission comes from after the efficient intramolecular

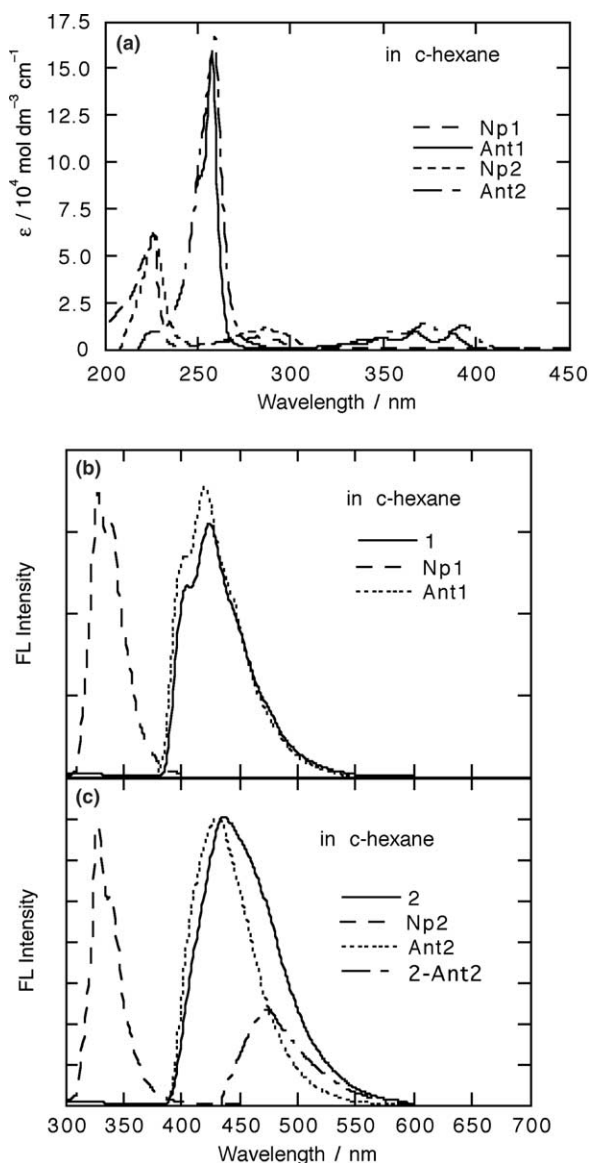


Fig. 4. Absorption spectra of Np1, Np2, Ant1, and Ant2 in c-hexane (a) Fluorescence spectra of 1, Np1, and Ant1 (b) and 2, Np2, Ant2 and 2-Ant2 (c) in argon purged c-hexane.

energy transfer from naphthyl to anthryl moieties (the efficiencies are estimated 73–95% by $\Phi_f^{290} / \Phi_f^{375}$).

This mechanism can be supported by the TR-FL. Figs. 3(d)–(f) show the TR-FL obtained for 1, 3, and 6 by the excitation at 280 nm in argon-purged acetonitrile, and the spectra were identical to those obtained by the excitation at 375 nm. The behavior vs. time for 6 is different from 1, 3, and also 6 in c-hexane. The emission just after the laser pulse mainly comes from the anthryl moiety, and the fluorescence maximum shifts to the longer wavelength showing the formation of the CT state. This clearly indicates that the CT emission comes after the energy transfer from the naphthyl to anthryl moieties (Fig. 5(b)). The behavior by time for 1 and 3 are similar to each other, and is also similar to those in c-hexane.

The origin of the CT-interaction is still under investigation. Both the mechanism of σ – π interaction proposed initially by Pitt et al. [4] and extended to the OICT mechanism by Sakaurai et al. [3] and the alternating $2p\pi^* \rightarrow 3d\pi$ mechanism proposed by Shizuka et al. [1,2] which was based on the CT mechanism of naphthyl-trimethylsilanes proposed by Evans et al. [21] are not contradictory to the experimental results in this study at this moment.

4. Conclusions

α -(9-Anthryl)- ω -(1-naphthyl)oligosilanes undergo efficient intramolecular electronic energy transfer from the naphthyl to anthryl moiety in both cyclohexane and acetonitrile. For $n = 4$ and 6 in acetonitrile, the excitations of both the naphthyl and anthryl moieties induce an intramolecular charge-transfer process producing CT fluorescence. The TR-FL measurement reveals that the intramolecular electronic energy transfer process is faster than the CT process, therefore, the CT ability is mainly governed by the property of the anthryl moiety and Si chain unit.

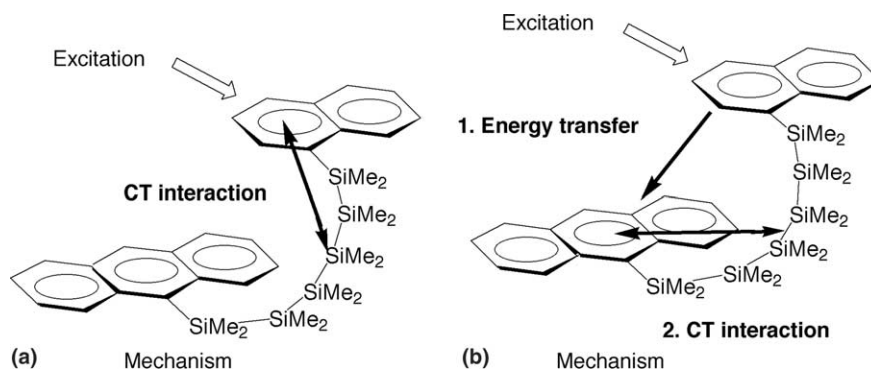


Fig. 5. Proposed mechanism of energy transfer and CT-interaction of 6 after excitation at 280 nm. Mechanism a, direct CT interaction between the naphthyl and hexasilanyl moieties; mechanism b, CT interaction after the energy transfer between the anthryl and hexasilanyl moieties which can be rationalized by TR-FL measurement.

Acknowledgements

The authors thank Prof. Tatsuo Arai (University of Tsukuba) for useful discussion, and Analytical Center (Chiba University) for the measurements of HRMS and elemental analysis. This work was supported by a Grant-in-Aid for Scientific Research on Priority Areas (417, No. 14050022) and Scientific Research (No. 14550786) from the Ministry of Education, Culture, Sports, Science, and Technology (MEXT) of the Japanese Government, to T.K.

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