Supporting Information for

Solvent Dependent Intramolecular Excimer Emission of Di(1-pyrenyl)silane and Di(1-pyrenyl)methane Derivatives

Shin-ichi Kondo*[†‡], Yuka Taguchi[†], and Yi Bie[†]

[†]*Department of Material and Biological Chemistry, Faculty of Science, Yamagata University, Yamagata 990-8560, Japan*

[‡]*Institute for Regional Innovation, Yamagata University, Kanakame, Kaminoyama, Yamagata 999-3101, Japan*

E-mail: kondo@sci.kj.yamagata-u.ac.jp

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General

All reagents used were of analytical grade. Tetrahydrofuran was dried over Na/benzophenone. NMR spectra were measured on a JEOL ECA-500 (500 MHz) spectrometer. UV-vis spectra were recorded on JASCO V-560 and Shimadzu UV-2500PC spectrometers with a thermal regulator (±0.5 ºC). Fluorescence spectra were recorded on Hitachi F-7000 and Jobin Yvon Horiba Fluoromax-3 spectrofluorometers under aerobic condition. HRMS were measured in $CHCl_3$: MeOH = 3: 1 at 150–200 ºC on a ABI Sciex TripleTOF 4600. Column chromatography was performed by using Silica Gel 60N from Kanto Reagents. Melting points were determined with a Yanagimoto MP-J3 micro melting point apparatus and are uncorrected. Di(1-pyrenyl)silanediol was prepared according to the previously reported method.[1]

Synthesis of di(1-pyrenyl)bis(trimethylsiloxy)silane (1b)

Into a solution of di(1-pyrenyl)silanediol (0.10 g, 0.22 mmol) in THF, pyridine (0.50 ml, 6.2 mmol) and chlorotrimethylsilane (0.11 ml, 0.86 mmol) were added and the mixture was stirred for 3 h under argon atmosphere at r.t. The mixture was extracted with chloroform/dil. HCl and the organic layer was dried over anhydrous sodium sulfate. After evaporation under reduced pressure, the residue was chromatographed on silica gel column chromatography ($CHCl_3$: hexane = 1 : 1, *v*/*v* as an eluent) to give the product as colorless needles. Yield 0.11 g, 83%. M. p. 221.2–222.2 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.48 (d, 2H, *J* = 9.0 Hz), 8.46 (d, 2H, *J* = 7.5 Hz), 8.19 (d, 2H, *J* = 7.5 Hz), 8.16 (d, 2H, *J* = 7.5 Hz), 8.10 (d, 2H, *J* = 8.8 Hz), 8.09 (d, 2H, *J* = 7.5 Hz), 8.08 (d, 2H, *J* = 8.8 Hz), 7.95 (t, 2H, *J* = 7.5 Hz), 7.89 (d, 2H, *J* = 9.0 Hz), 0.08 (s, 18H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 135.78, 133.12, 132.78, 132.69, 131.21, 130.69, 128.46, 128.16, 127.55, 127.01, 125.70, 125.18, 125.04, 124.75, 124.58, 124.25, 1.84. ^{29}Si NMR (99 MHz, $CDCl_3$) δ 10.8, −21.8. HRMS (ESI, positive mode): Calcd for $C_{38}H_{36}NaO_2Si_3$ $[M+Na]^+$, 631.1915. Found 631.1872.

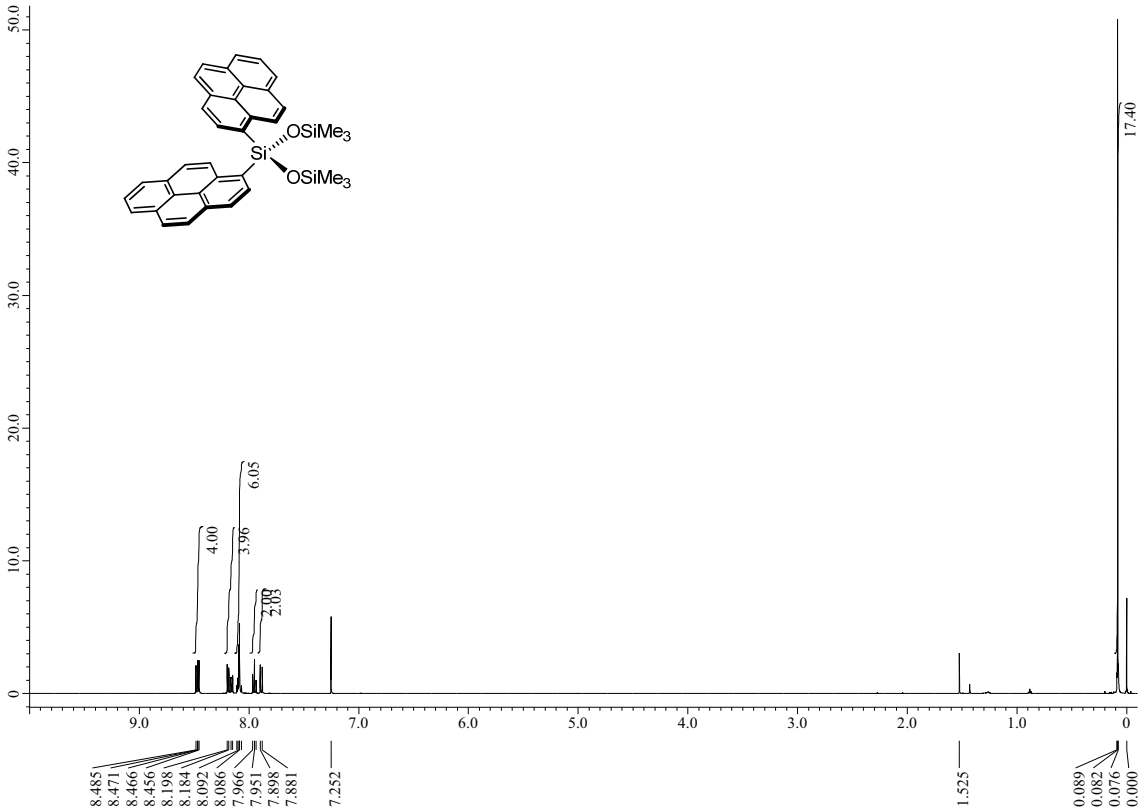

Fig. S1. 500 MHz ^{1}H NMR spectrum of di(1-pyrenyl)di(trimethylsiloxy)silane in $CDCl_3$.

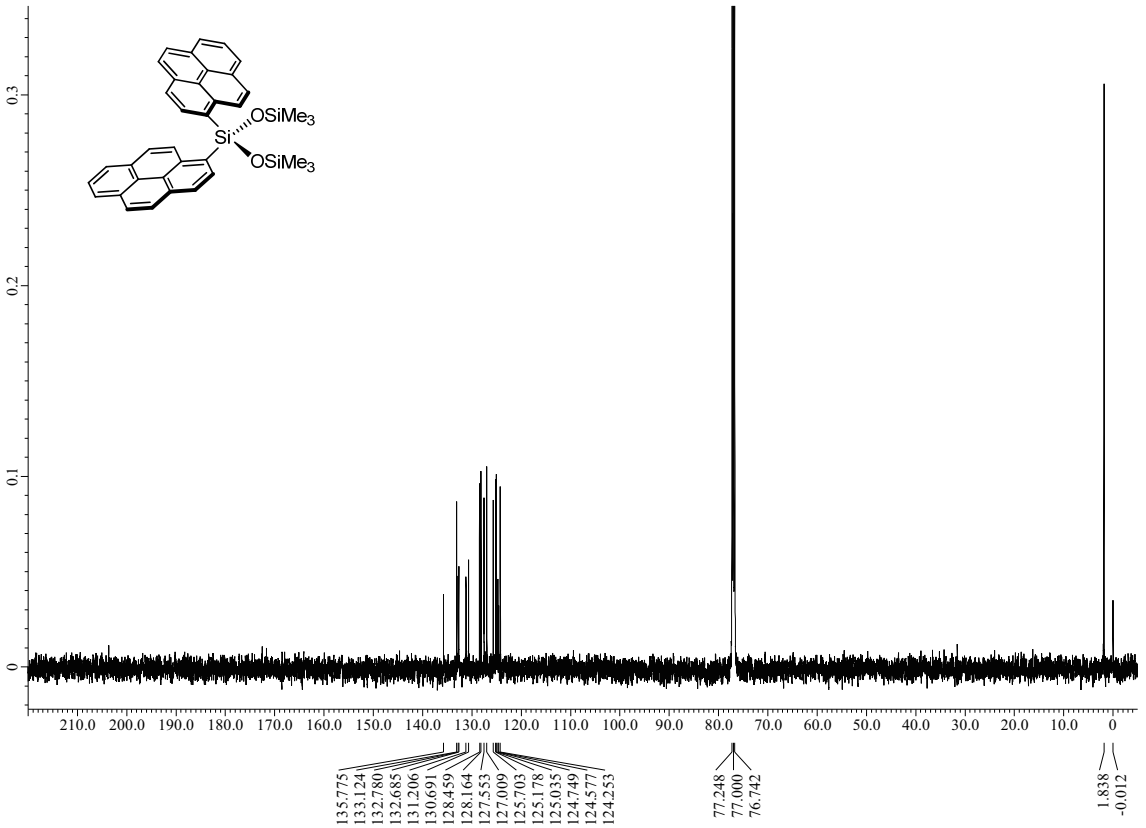

Fig. S2. 126 MHz ^{13}C NMR spectrum of di(1-pyrenyl)di(trimethylsiloxy)silane in $CDCl_3$.

Synthesis of dimethyldi(1-pyrenyl)silane (1c)

Into a solution of 1-bromopyrene (1.00 g, 3.56 mmol) in THF (10 mL), was added *n*-BuLi/hexane (1.64 M, 2.60 mL, 4.27 mmol) at −78 ºC under argon atmosphaere. The mixture was stirred at −78 ºC for 1 h. Then tetrachlorosilane (0.204 mL, 1.78 mmol) was slowly added into the solution and the mixture was stirred at −78 ºC for 30 min, then stirred at −40 ºC for additional 30 min. After the mixture was cooled again to −78 ºC, freshly prepared MeMgI from iodomethane (ca. 2.6 M, 5.5 ml, 8 equiv) was added in the solution. The mixture was allowed to warm to room temperature and stirred overnight. Aqueous ammonium chloride (sat.) was added to the mixture, and the mixture was extracted with ether (50 mL × 2). The combined organic layer was washed with brine and dried over anhydrous sodium sulfate. After evaporation under reduced pressure, the residue was purified by column chromatography on silica gel with hexane as an eluent to give the product (191 mg, 23%) as colorless solid: M. p. 251.2–252.0 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41 (d, 2H, *J* = 7.5 Hz), 8.22 (d, 2H, *J* = 9.2 Hz), 8.21 (d, 2H, *J* = 9.2 Hz), 8.15 (d, 2H, *J* = 7.5 Hz), 8.09 (s, 4H), 8.06 (d, 2H, *J* = 7.5 Hz), 7.93 (t, 2H, *J* = 7.5 Hz), 7.81 (d, 2H, *J* = 9.2 Hz), 1.09 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 135.84, 134.43, 132.77, 132.42, 131.24, 130.58, 128.06, 127.75, 127.51, 127.18, 125.78, 125.21, 125.09, 124.80, 124.75, 124.42, 0.60. HRMS (ESI, positive mode): Calcd for $C_{34}H_{24}NaSi$ $[M+Na]^+$, 483.1539. Found 483.1503.

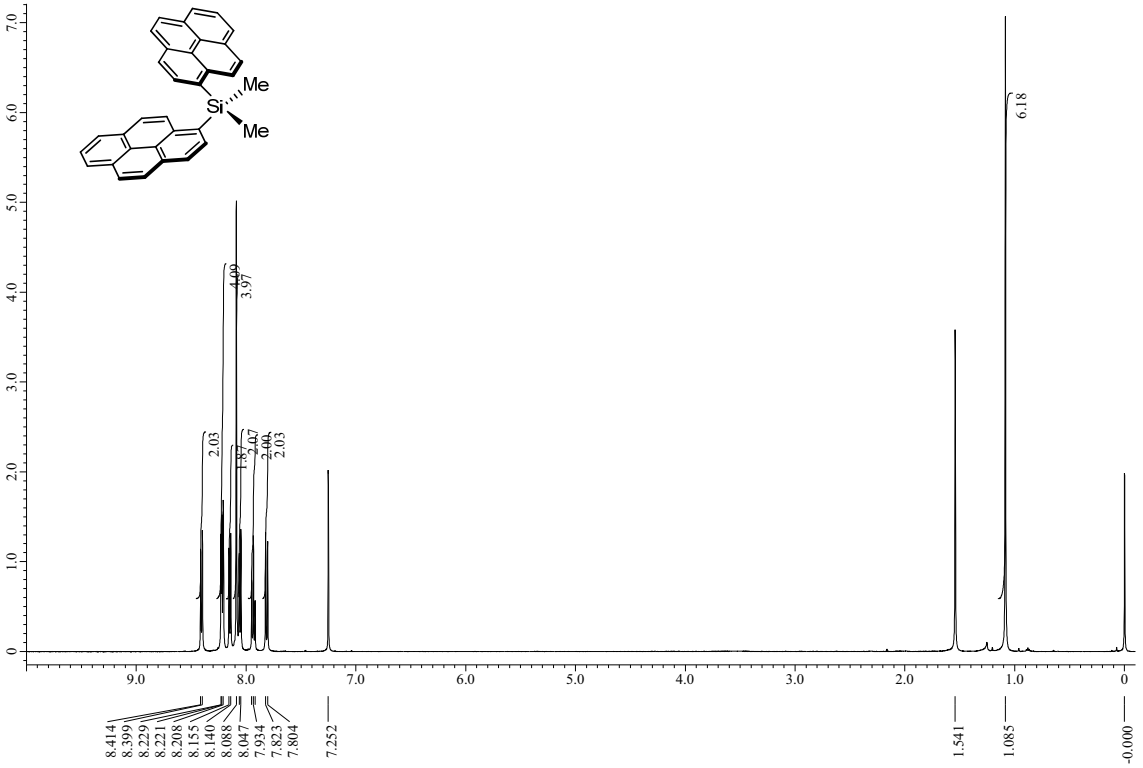

Fig. S3. 500 MHz ^{1}H NMR spectrum of dimethyldi(1-pyrenyl)silane in $CDCl_3$.

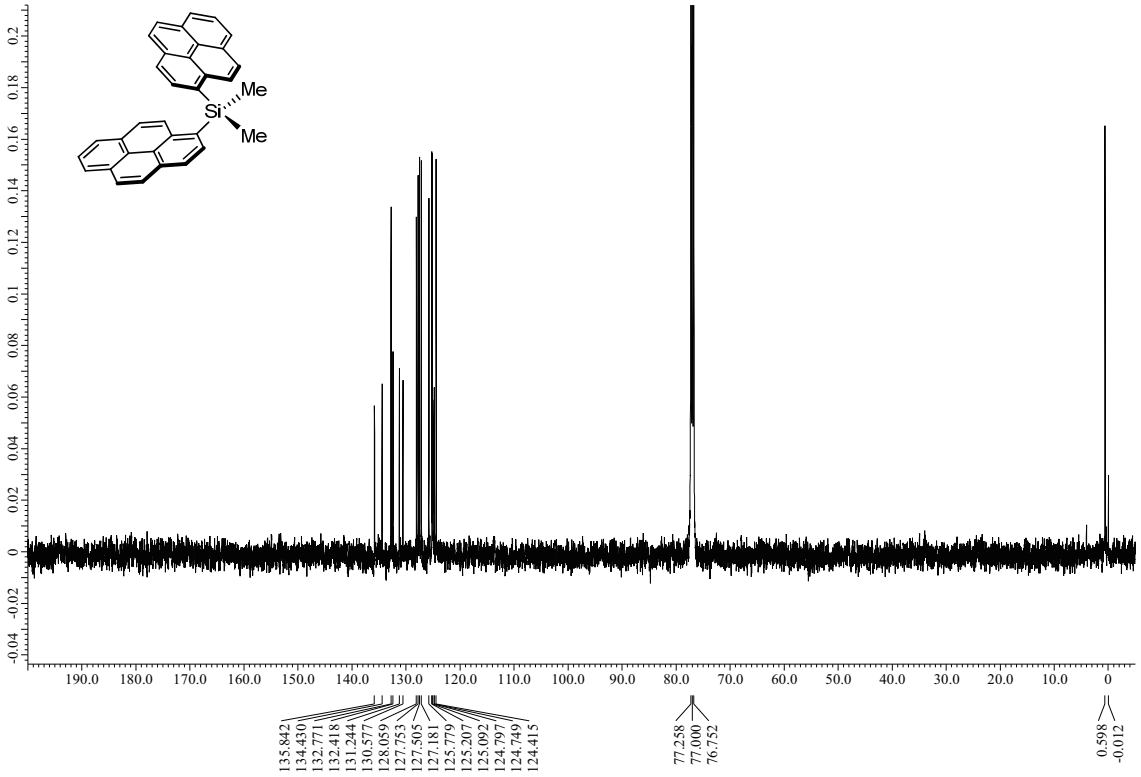

Fig. S4. 126 MHz ^{13}C NMR spectrum of dimethyldi(1-pyrenyl)silane in $CDCl_3$.

Synthesis of dimethylphenyl(1-pyrenyl)silane (2)

Into a solution of 1-bromopyrene (0.75 g, 2.67 mmol) in THF (10 mL), was added *n*-BuLi/hexane (1.64 M, 1.95 mL, 3.20 mmol) at −78 ºC under argon atmosphere. The mixture was stirred at −78 ºC for 1 h. Then phenyltrichlorosilane (0.428 mL, 2.67 mmol) was slowly added into the solution and the mixture was stirred at −78 ºC for 30 min, then stirred at −40 ºC for additional 30 min. After the mixture was cooled again to −78 ºC, freshly prepared MeMgI from iodomethane (ca. 2.6 M, 8.4 mL, 8 equiv) was added in the solution. The mixture was allowed to warm to room temperature and stirred overnight. Aqueous ammonium chloride (sat.) was added to the mixture, and the mixture was extracted with ether (45 mL × 2). The combined organic layer was washed with brine and dried over anhydrous sodium sulfate. After evaporation under reduced pressure, the residue was purified by column chromatography on silica gel with hexane as an eluent to give the product (364 mg, 41%) as pale yellow solid: M. p. 167.5–168.0 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.23 (d, 1H, J = 7.5 Hz), 8.19 (d, 1H, J = 9.2 Hz), 8.17 (d, 2H, J = 7.5 Hz), 8.14 (d, 1H, J = 7.5 Hz), 8.09 (d, 1H, J = 9.2 Hz), 8.06 (d, 1H, J = 9.2 Hz), 7.98 (t, 1H, J = 7.5 Hz), 7.96 (d, 1H, J = 9.2 Hz), 7.58 (dd, 2H, J_1 = 7.7, J_2 = 1.7 Hz), 7.36 (tt, 1H, J_1 = 7.4 Hz, J_2 = 1.7 Hz), 7.34–7.31 (m, 2H), 0.83 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 139.12, 135.90, 134.23, 133.13, 132.97, 132.44, 131.25, 130.59, 129.08, 128.06, 128.04, 127.94, 127.50, 127.05, 125.81, 125.24, 125.11, 124.80, 124.65, 124.09, −0.57. HRMS (ESI, positive mode): Calcd for $C_{24}H_{20}NaSi$ $[M+Na]^+$, 359.1226. Found 359.1195.

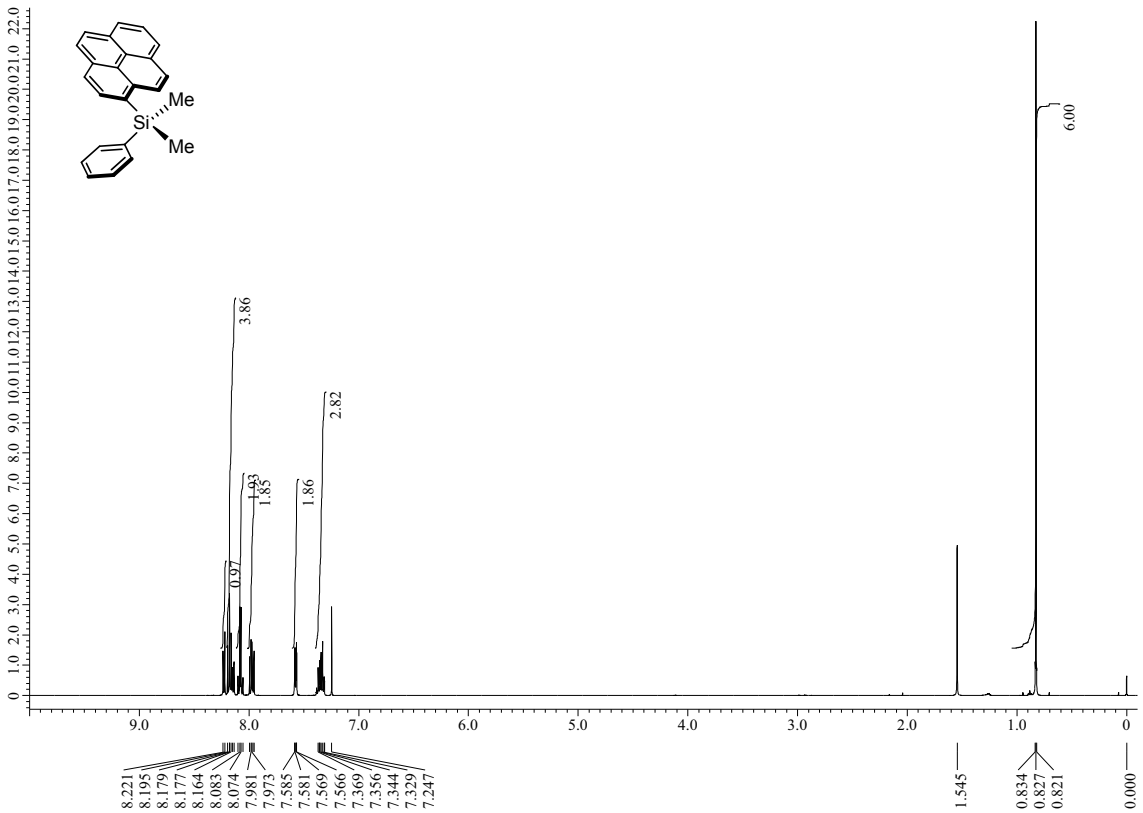

Fig. S5. 500 MHz ^{1}H NMR spectrum of dimethylphenyl(1-pyrenyl)silane in $CDCl_3$.

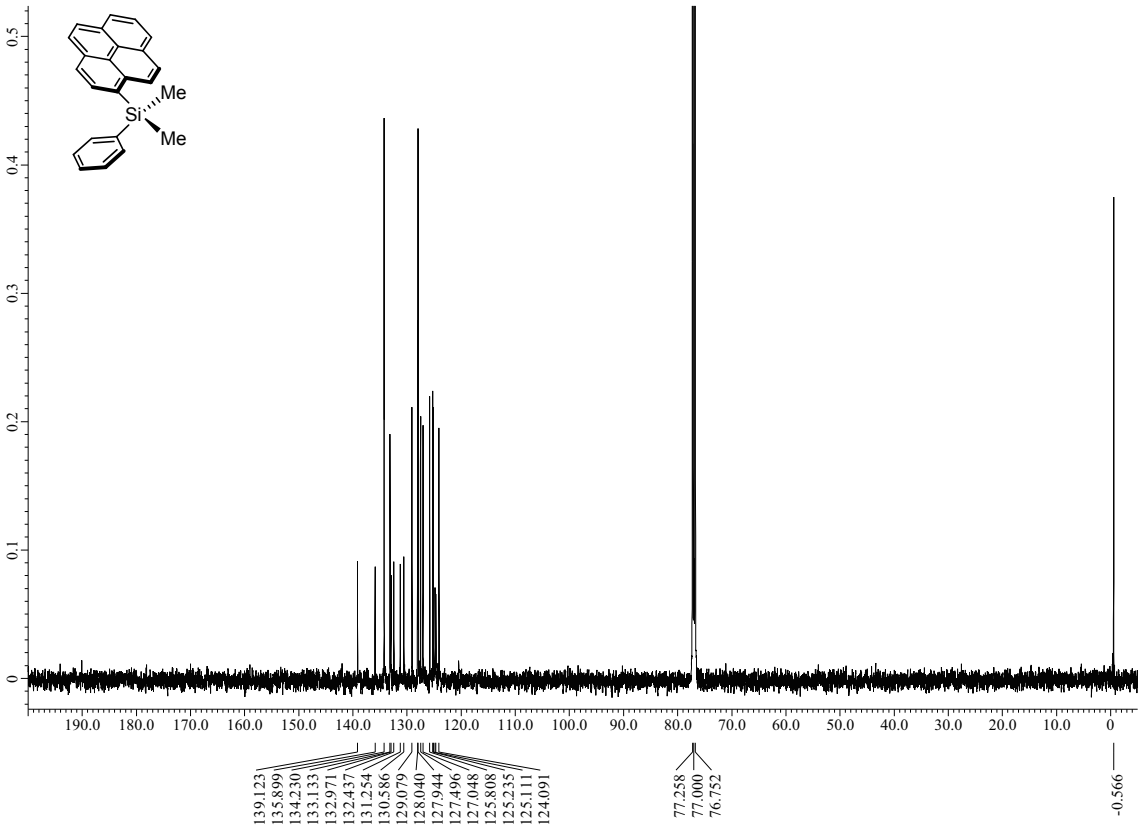

Fig. S6. 126 MHz ^{13}C NMR spectrum of dimethylphenyl(1-pyrenyl)silane in $CDCl_3$.

Synthesis of di(1-pyrenyl)methanol (3a)

Into a solution of 1-bromopyrene (0.50 g, 1.78 mmol) in THF (5 mL), was added *n*-BuLi/hexane (1.64 M, 1.30 mL, 2.13 mmol) at −78 ºC under argon atmosphere. The mixture was stirred at −78 ºC for 1 h. Then 1-pyrenecarboxyaldehyde (0.41 g, 1.78 mmol) in 5 mL of THF was slowly added into the solution and the mixture was stirred at −78 ºC for 30 min. The mixture was allowed to warm at room temperature and stirred overnight. Water (10 mL) was added to the mixture, and ether (10 mL) was also added to the mixture. The produced solid was filtered and the solid was washed with ether to give the product (0.33 g, 43%) as pale yellow solid: M. p. 248.5–249.5 ºC (decomp, lit.[2] 254 ºC). ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.49 (dd, 2H, J_1 = 9.2, J_2 = 2.3 Hz), 8.29 (d,2H, J = 7.5 Hz), 8.25 (d, 2H, J = 6.9 Hz), 8.24 (d, 2H, J = 7.5 Hz), 8.15 (m, 6H), 8.06 (t, 2H, J = 7.5 Hz), 8.04 (d, 2H, J = 6.9 Hz), 7.82 (d, 1H, J = 4.0 Hz), 6.55 (d, 1H, J = 4.0 Hz). ^{13}C NMR (126 MHz, DMSO-d_6) δ 138.41, 130.83, 130.18, 130.10, 127.72, 127.56, 127.42, 127.15, 126.25, 125.38, 125.35, 125.16, 124.74, 124.11, 124.00, 123.42, 68.33. HRMS (ESI, positive mode): Calcd for $C_{33}H_{20}NaO$ $[M+Na]^+$, 455.1406. Found 455.1376.

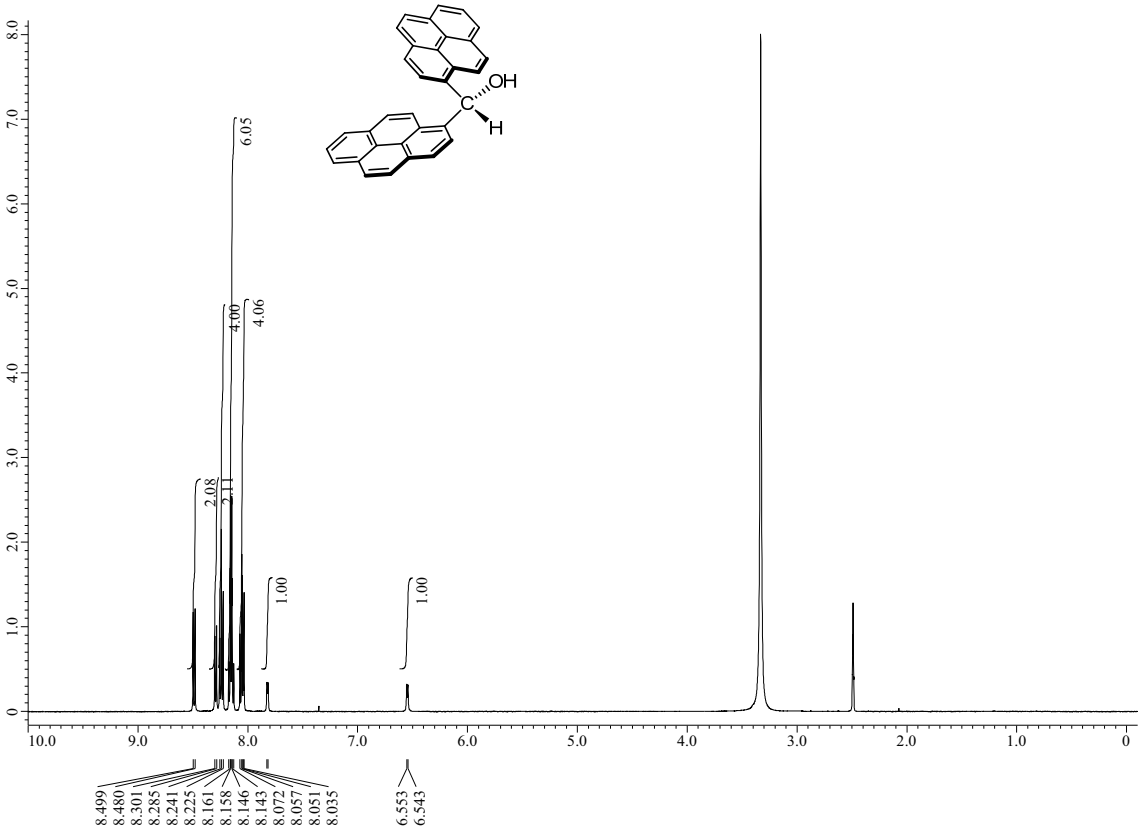

Fig. S7. 500 MHz ^{1}H NMR spectrum of di(1-pyrenyl)methanol in DMSO-d_6.

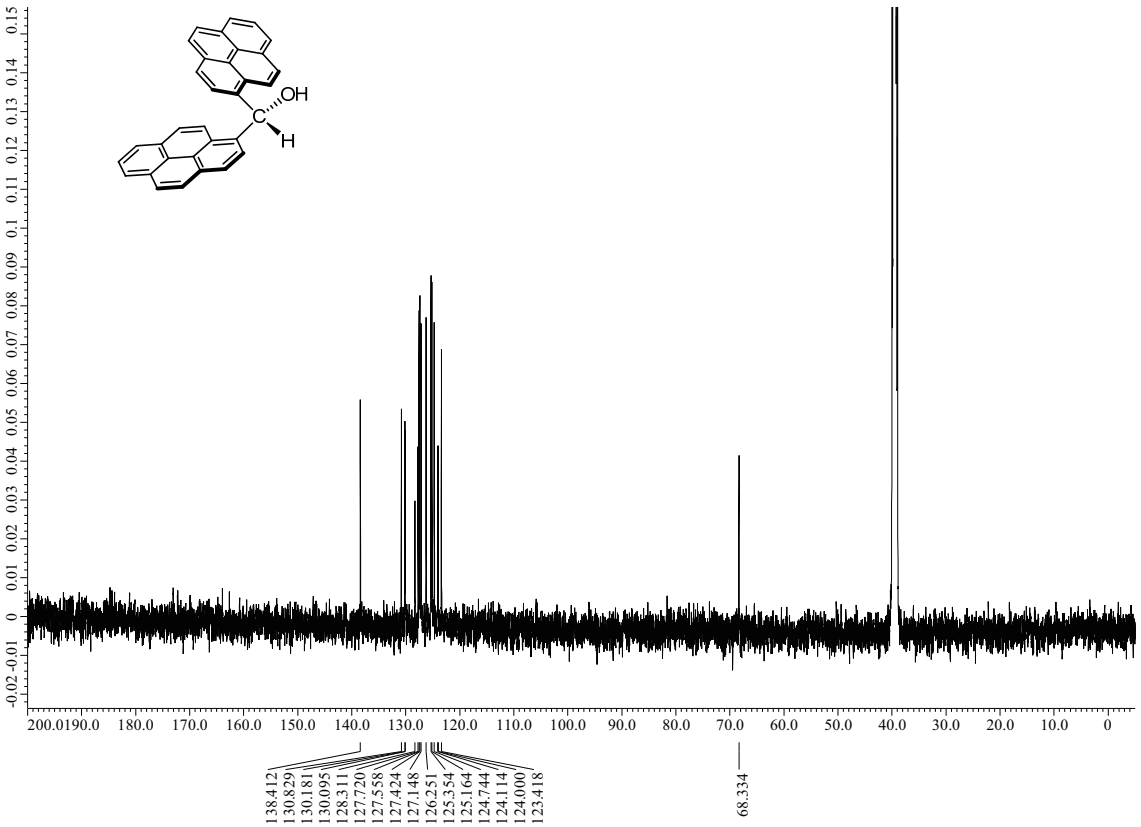

Fig. S8. 126 MHz ^{13}C NMR spectrum of di(1-pyrenyl)methanol in DMSO-d_6.

Synthesis of di(1-pyrenyl)methanone

Into a suspension of pyridinium chlorochromate (0.199 g, 0.925 mmol) and Hyflo Super-Cel (0.199 g) in dichloromethane (100 mL), was slowly added di(1-pyrenyl)methanol (0.20 g, 0.462 mmol) at r.t. under argon atmosphere. The mixture was stirred at r.t. for 2 h and column chromatographed on silica gel (CH_2Cl_2) to give the product (0.195 g, 98%) as yellow solid. M. p. 260.0–262.0 ºC (lit.[3] 234–235 ºC).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.82 (d, 2H, J = 9.5Hz), 8.28 (d, 2H, J = 8.0Hz), 8.26 (d, 2H, J = 8.0Hz), 8.21 (d, 2H, J = 8.6Hz), 8.17 (d, 2H, J = 9.8Hz), 8.12–8.08(m, 6H), 8.08 (t, 2H, J = 8.0Hz) ; ^{13}C NMR (126 MHz, $CDCl_3$) δ 200.72, 134.39, 133.91, 131.27, 130.77, 130.51, 129.71, 129.59, 129.14, 127.35, 126.56, 126.43, 126.27, 125.07, 124.54, 124.01.

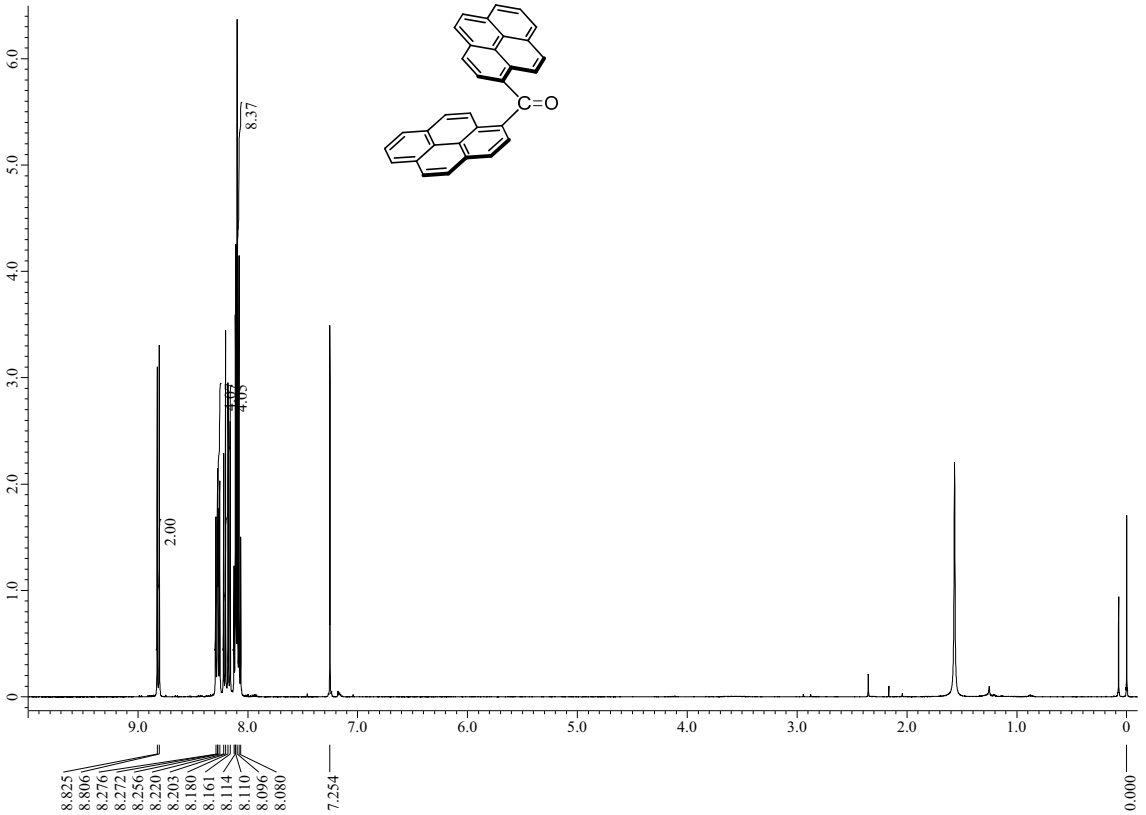

Fig. S9. 500 MHz ^{1}H NMR spectrum of di(1-pyrenyl)methanone in $CDCl_3$.

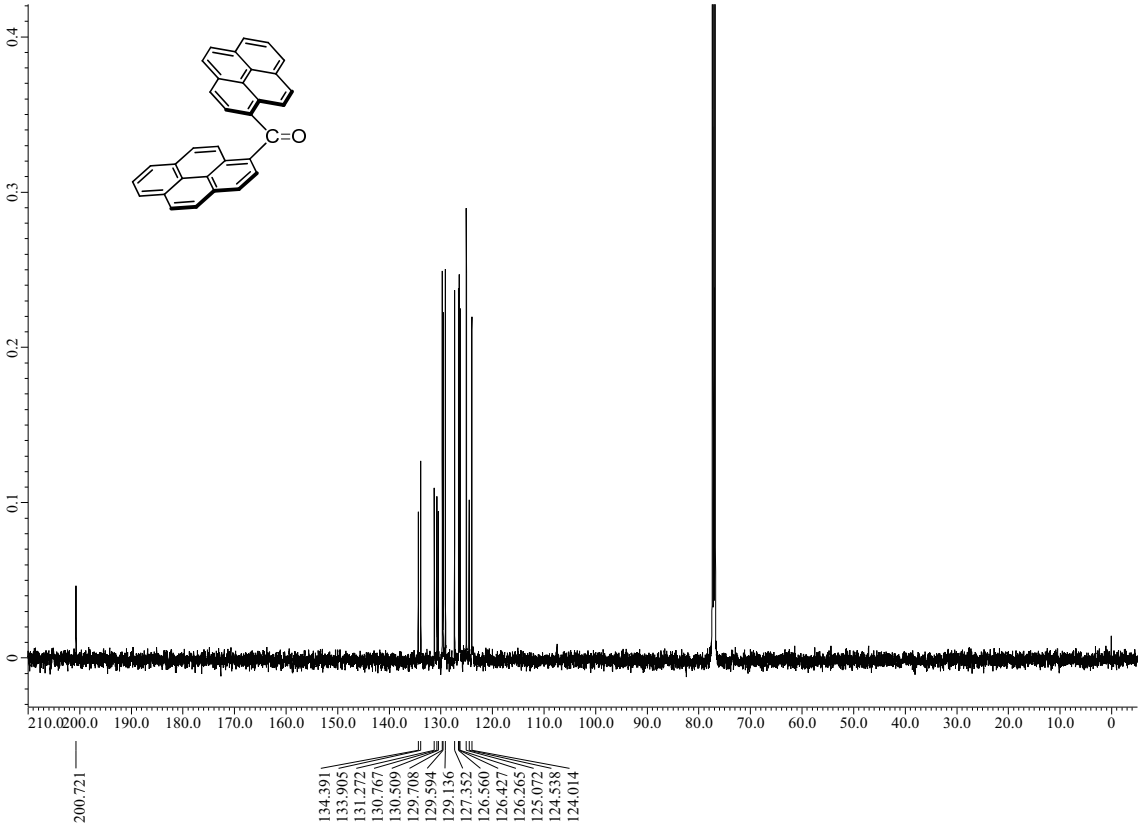

Fig. S10. 126 MHz ^{13}C NMR spectrum of di(1-pyrenyl)methanone in $CDCl_3$.

Synthesis of di(1-pyrenyl)methane (3b)

A suspension of di(1-pyrenyl)methanone (0.10 g, 0.232 mmol), hydrazine monohydrate (0.031 g, 0.627 mmol), and potassium hydroxide (0.102 g, excess) in triethylene glycol (2 mL) was stirred at 180 ºC overnight under argon atmosphere. The mixture was filtered with suction and the filtrate was extracted with chloroform (10 mL×2) and water. The combined organic layer was twice washed with water and dried over anhydrous sodium sulfate. After filtration, the solution was evaporated under reduced pressure. The residue was purified by column chromatography on silica gel with $CHCl_3$:hexane = 1:1 (*v*/*v*) as an eluent to give the product (19 mg, 20%) as pale yellow solid: M. p. 226.4–228.0 ºC (decomp. lit.[2] 235 ºC).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.36 (d, 2H, *J* = 9.8Hz), 8.20 (d, 2H, *J* = 7.4Hz), 8.18 (d, 2H, *J* = 7.4Hz), 8.10 (d, 2H, *J* = 7.4Hz), 8.06 (d, 2H, *J* = 8.6Hz), 8.05–8.02(m, 6H), 8.01 (t, 2H, *J* = 7.4Hz), 7.66 (d, 2H, *J* = 8.0Hz), 5.47 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 134.44, 131.45, 130.92, 130.16, 129.16, 127.81, 127.72, 127.54, 126.91, 125.93, 125.14, 125.08, 125.00, 124.97, 124.93, 123.42, 36.47.

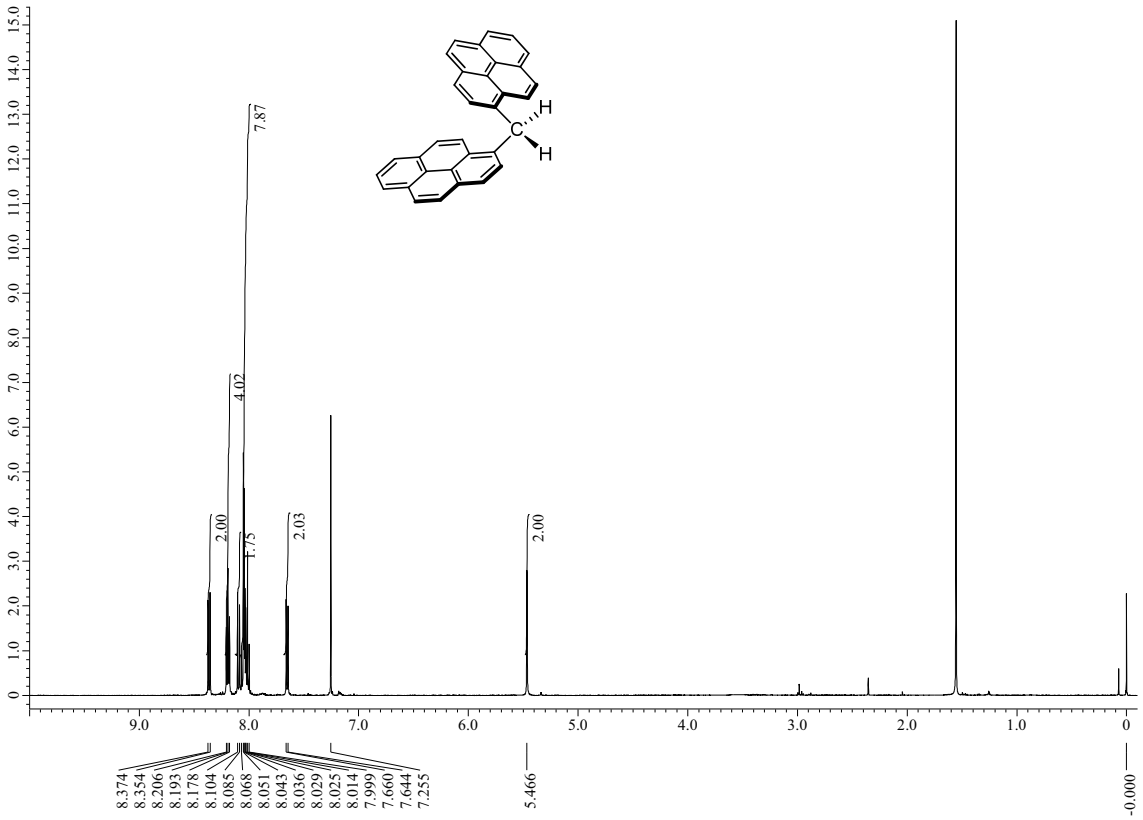

Fig. S11. 500 MHz ^{1}H NMR spectrum of di(1-pyrenyl)methane in $CDCl_3$.

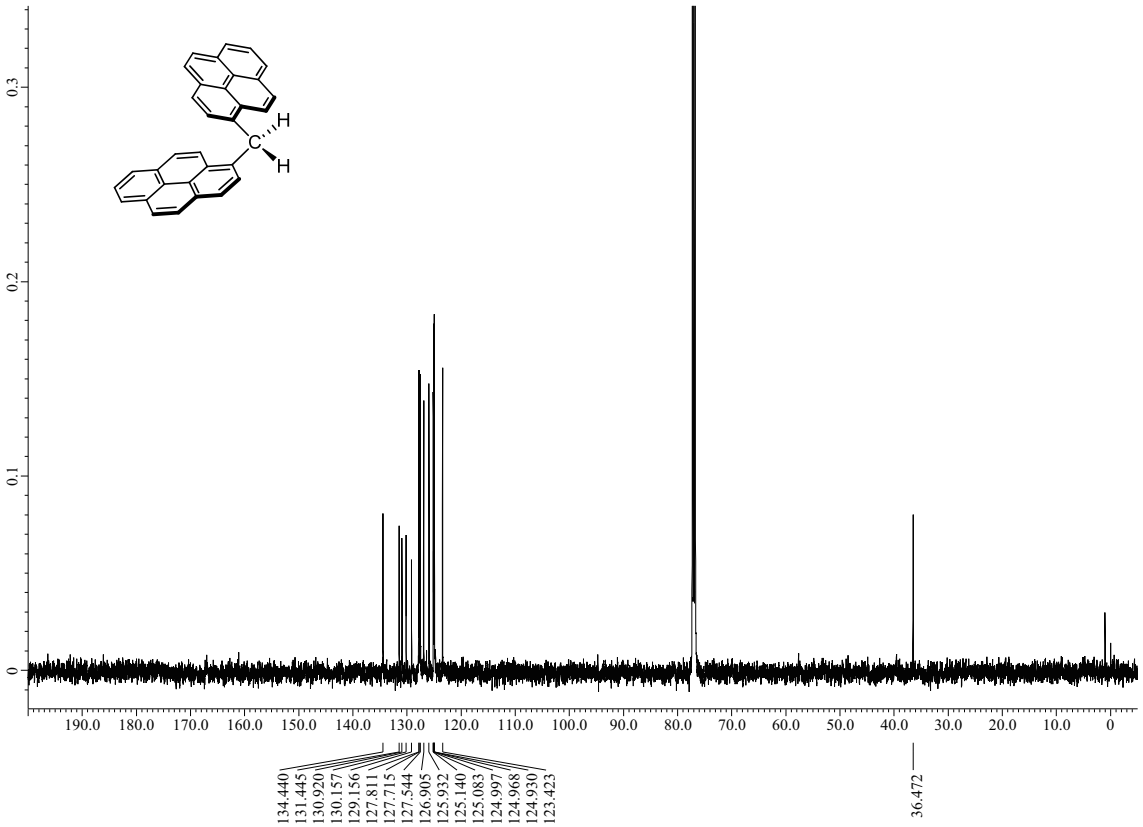

Fig. S12. 126 MHz ^{13}C NMR spectrum of di(1-pyrenyl)methane in $CDCl_3$.

Synthesis of phenyl(1-pyrenyl)methanol (4)[4]

Into a solution of 1-bromopyrene (1.00 g, 3.56 mmol) in THF (7 mL), was added *n*-BuLi/hexane (1.64 M, 2.60 mL, 4.27 mmol) at −78 ºC under argon atmosphere. The mixture was stirred at −78 ºC for 1 h. Then freshly distilled benzaldehyde (0.453 g, 4.27 mmol) in 5 mL of THF was slowly added into the solution and the mixture was stirred at −78 ºC for 10 min. The mixture was allowed to warm at room temperature and stirred overnight. Water was added to the mixture, and the mixture was extracted with $CHCl_3$ (10 mL×2). After evaporation under reduced pressure, the residue was chromatographed on silica gel ($CHCl_3$ as an eluent) to give the product (505 mg, 46.0%) as pale yellow solid: M. p. 126.2–127.2 ºC (lit.[2] 126 ºC)

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.31 (d, 1H, *J* = 10.0Hz), 8.18–8.17 (m, 3H), 8.15 (d, 1H, *J* = 7.5Hz), 8.05–8.04 (m, 3H), 7.99 (t, 1H, *J* = 7.5Hz), 7.44 (d, 2H, *J* = 7.5Hz), 7.32 (t, 2H, *J* = 7.5Hz), 7.27 (t, 1H, *J* = 7.5Hz), 6.87 (d, 1H, *J* = 3.3Hz) , 2.54 (d, 1H, *J* = 3.5Hz).

^{13}C NMR (126 MHz, $CDCl_3$) δ 143.60, 136.57, 131.30, 131.00, 130.59, 128.55, 128.07, 127.78, 127.60, 127.46, 127.43, 126.96, 125.97, 125.34, 125.17, 125.01, 124.88, 124.78, 124.68, 123.04, 73.57.

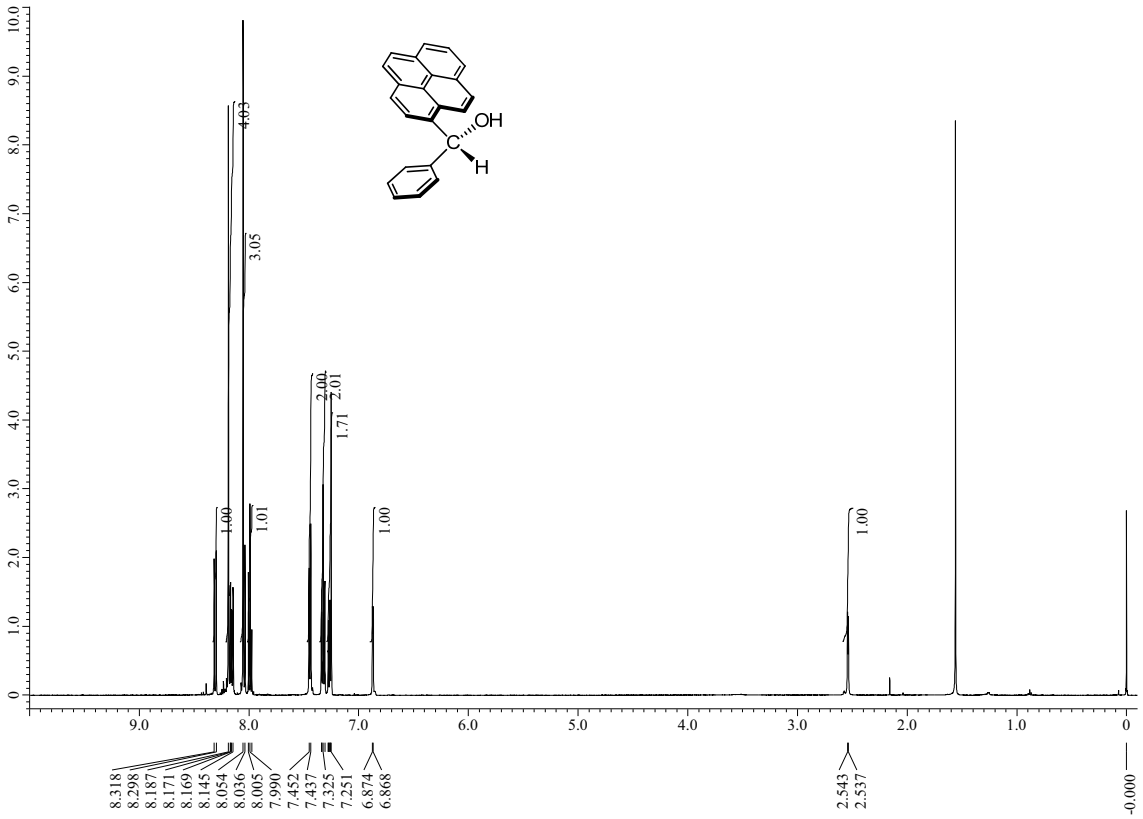

Fig. S13. 500 MHz ^{1}H NMR spectrum of phenyl(1-pyrenyl)methanol in $CDCl_3$.

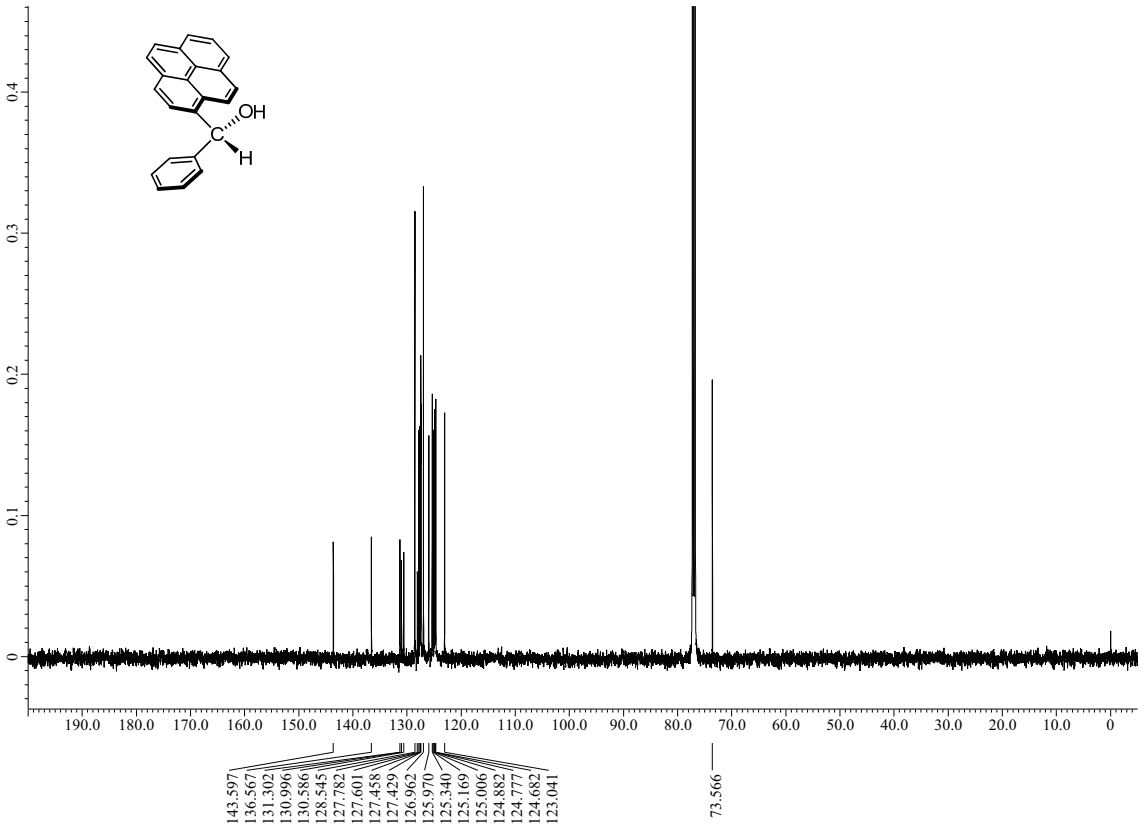

Fig. S14. 126 MHz ^{13}C NMR spectrum of phenyl(1-pyrenyl)methanol in $CDCl_3$.

Measurements of UV-vis spectra of probes

Into a solvent (3 mL) in a quarts cell, an aliquots (5.0 μL) of the stock solution of probes (2.5×10^{-3} mol dm^{-3} for **1a–c**, **2**, **3b**, and **4** in chloroform, 2.5×10^{-3} mol dm^{-3} for **3a** in DMSO, and 2.5×10^{-4} mol dm^{-3} for **1c** in chloroform for measurements in ethylene glycol) was added with a microsyringe and a UV-vis spectrum of the solution was measured at 298 ± 0.5 K. The process was repeated for additional two times (the concentrations of the all probes were up to 1.25×10^{-5} mol dm^{-3} except for **1c** in ethylene glycol, 2.0×10^{-6} mol dm^{-3}). The molar absorption coefficients at each wavelength were calculated from the slope of the absorbance against the concentration of the probes by linear regressions. The solubility of **1b** in ethylene glycol is too low to determine the molar absorption coefficients. The results are shown in Fig. S15.

Measurements of fluorescence spectra of probes

Into a solvent (3 mL) in a quarts cell, an aliquots (8.0 μL) of the stock solution of probes (2.5×10^{-4} mol dm^{-3} for **1a–c**, **2**, **3b**, and **4** in chloroform and 2.5×10^{-4} mol dm^{-3} for **3a** in DMSO) was added with a microsyringe and a fluorescence spectrum of the solution excited at 348 nm was measured at 298 ± 0.5 K. The process was repeated for additional two times. The concentrations of the all probes were up to 2.0×10^{-6} mol dm^{-3}. The fluorescence intensities at each wavelength were calculated from the slope of the intensity against the concentration of the probes by linear regressions. The results are summarized in Figs. 1 and S17.

Determination of quantum yields

Fluorescence quantum yields were determined from the derived fluorescence spectrum of each species using quinine sulfate as standard (Φ_{QS} = 0.546 in 0.5 mol dm^{-3} H_2SO_4 at 298 K) and were corrected for solvent refractive index. Into an appropriate solvent (3 mL) in a quarts cell, an aliquots of the stock solution of probes was added with a microsyringe and UV-vis and fluorescence spectra (excited at 348 nm) were measured at 298 ± 0.5 K. The process was repeated for additional at least four times. The integrated fluorescence intensities of the probes were plotted against the UV-vis absorbance at each concentrations to give the slope F/A, in which F and A are the integrated fluorescence intensity and the absorbance of the probes, respectively. The same procedure was performed for quinine sulfate in 0.5 mol dm^{-3} H_2SO_4 at 298 ± 0.5 K to give F_{QS}/A_{QS}, in which F_{QS} and A_{QS} are the integrated fluorescence intensity and the absorbance of the quinine sulfate, respectively. The quantum yield of the probes Φ was calculated according to the equation;

$$\Phi = \Phi_F \frac{F/A}{F_{QS}/A_{QS}} \left(\frac{n}{n_{QS}}\right)^2$$

where n and n_{QS} are the refractive index of the measured solvent and 0.5 mol dm^{-3} H_2SO_4, respectively.

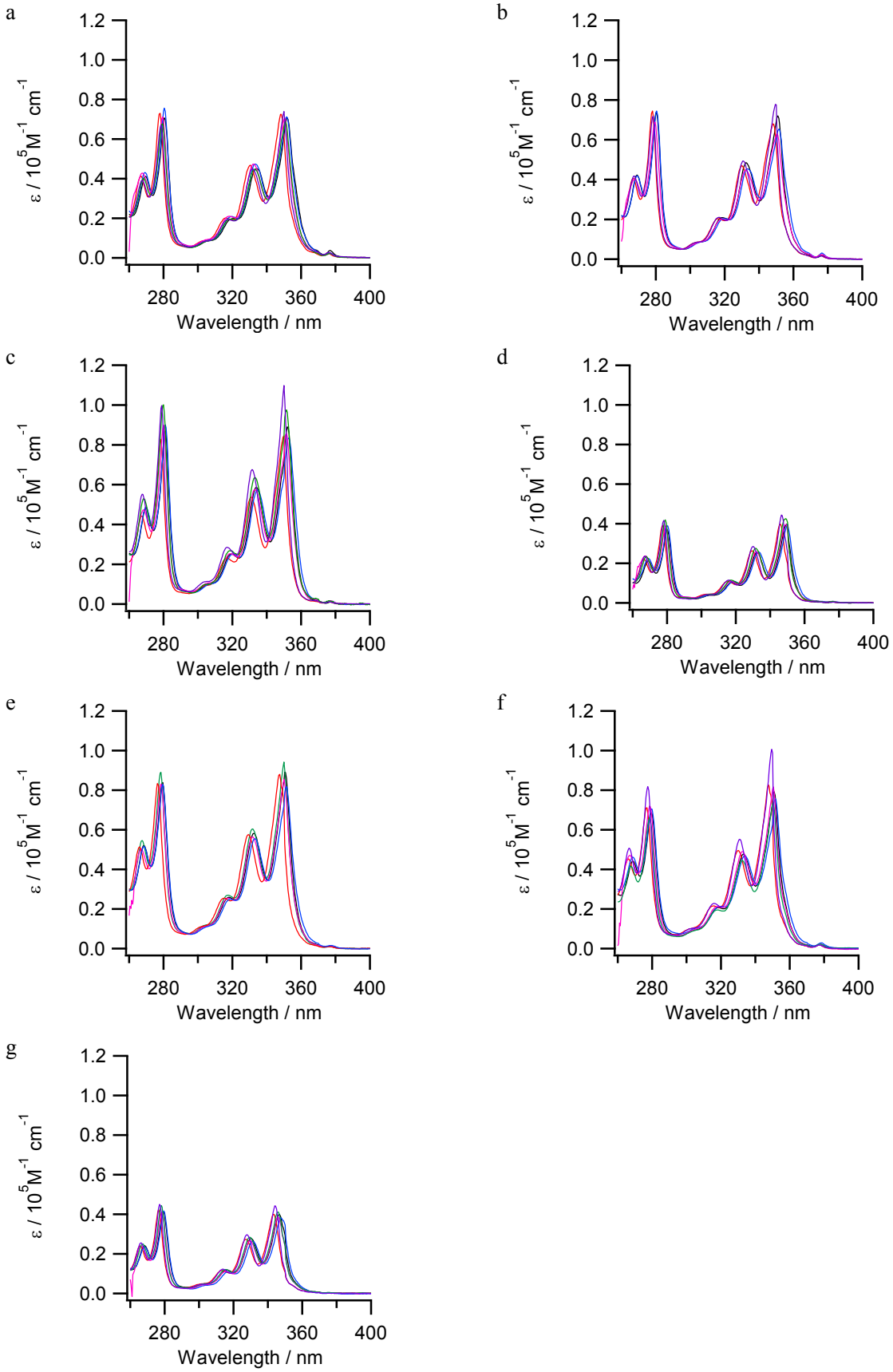

Fig. S15. UV-vis spectra of **1a** (a), **1b** (b), **1c** (c), **2** (d), **3a** (e), **3b** (f), and **4** (g) in DMSO (—), ethylene glycol (—), DMF (—), MeCN (—), $CHCl_3$ (—), and cyclohexane (—). A UV-vis spectrum of **1b** in ethylene glycol is not shown due to the low solubility of **1b**.

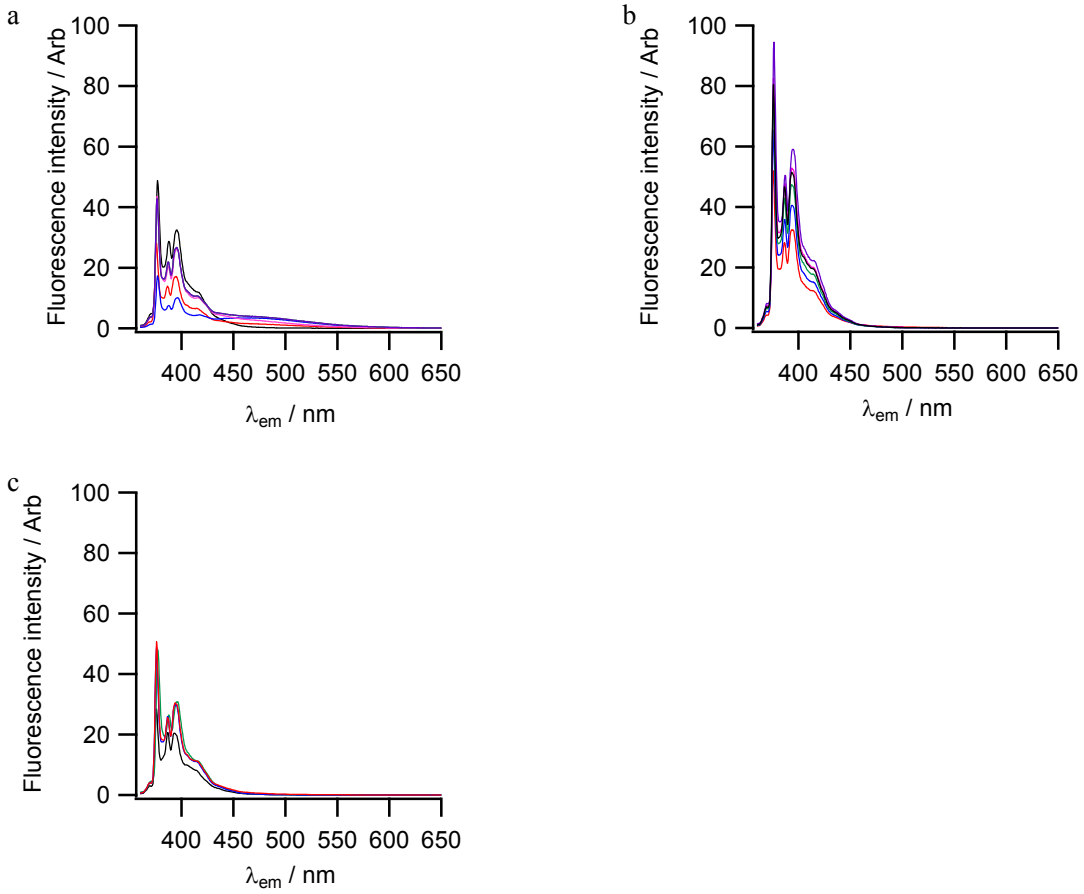

Fig. S16. Fluorescence spectra of **1c** (a) in DMSO (—), ethylene glycol (—), DMF (—), MeCN (—), $CHCl_3$ (—), and cyclohexane (—); (b) in EtOH (—), 2-PrOH (—), 1-BuOH (—), MeOH (—), Et_2O (—), and THF (—); and (c) in acetone (—), ethyl acetate (—), toluene (—) , and hexane (—). λ_{ex} = 348 nm at 298 K.

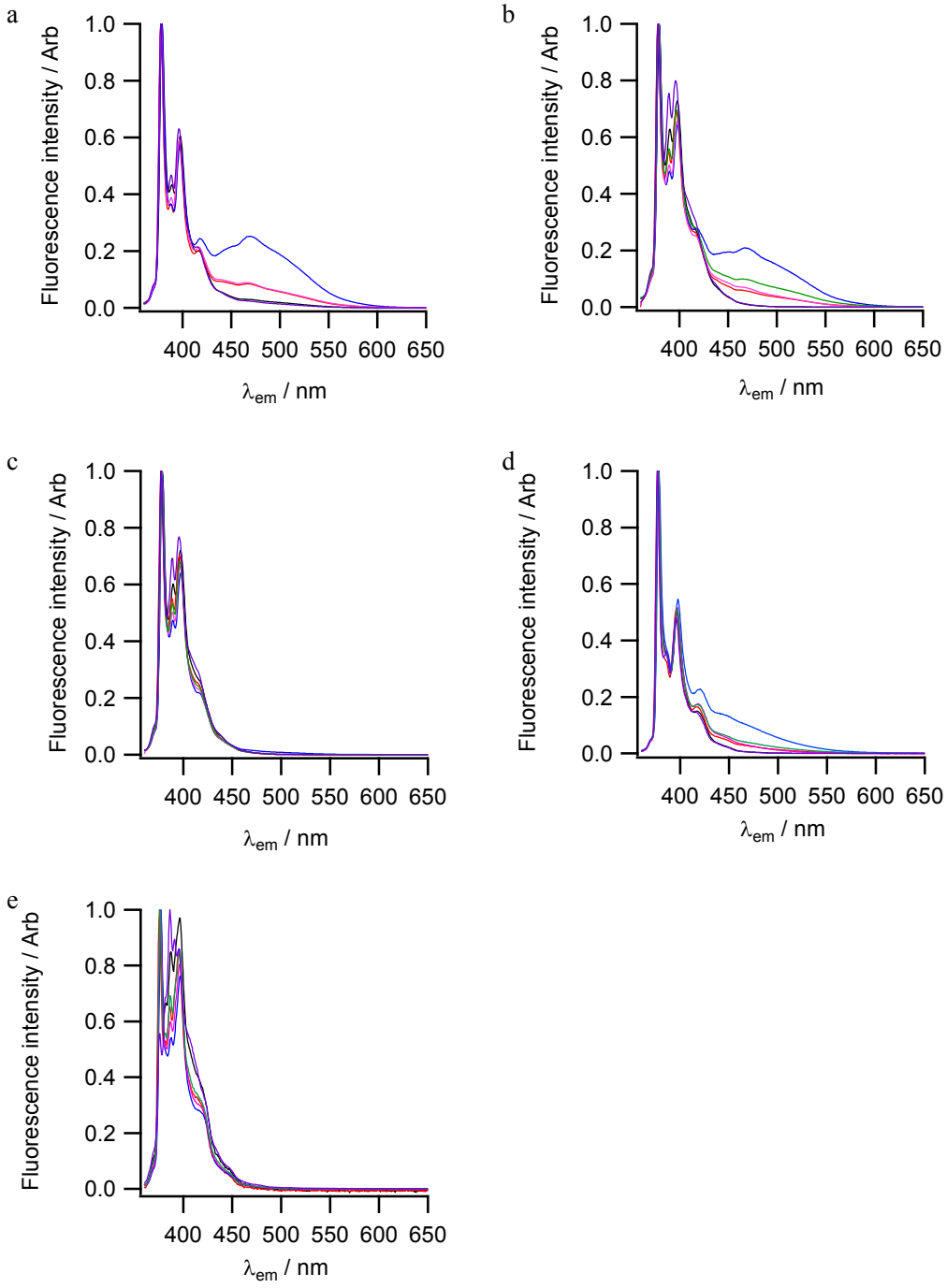

Fig. S17. Normalized fluorescence spectra of **1b** (a), **1c** (b), **2** (c), **3b** (d), and **4** (e) in DMSO (—), ethylene glycol (—), DMF (—), MeCN (—), $CHCl_3$ (—), and cyclohexane (—). A fluorescence spectrum of **1b** in ethylene glycol is not shown due to the low solubility of **1b**. λ_{ex} = 348 nm at 298 K.

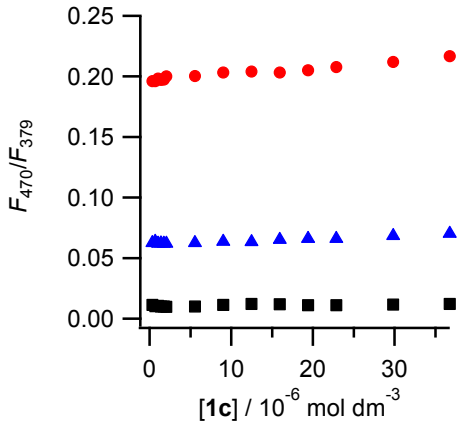

Fig. S18. Effect of concentration of **1c** on the ratio of fluorescence intensities at 470 and 379 nm in DMSO (●), MeCN (▲), and $CHCl_3$ (■). λ_{ex} = 348 nm at 298 K.

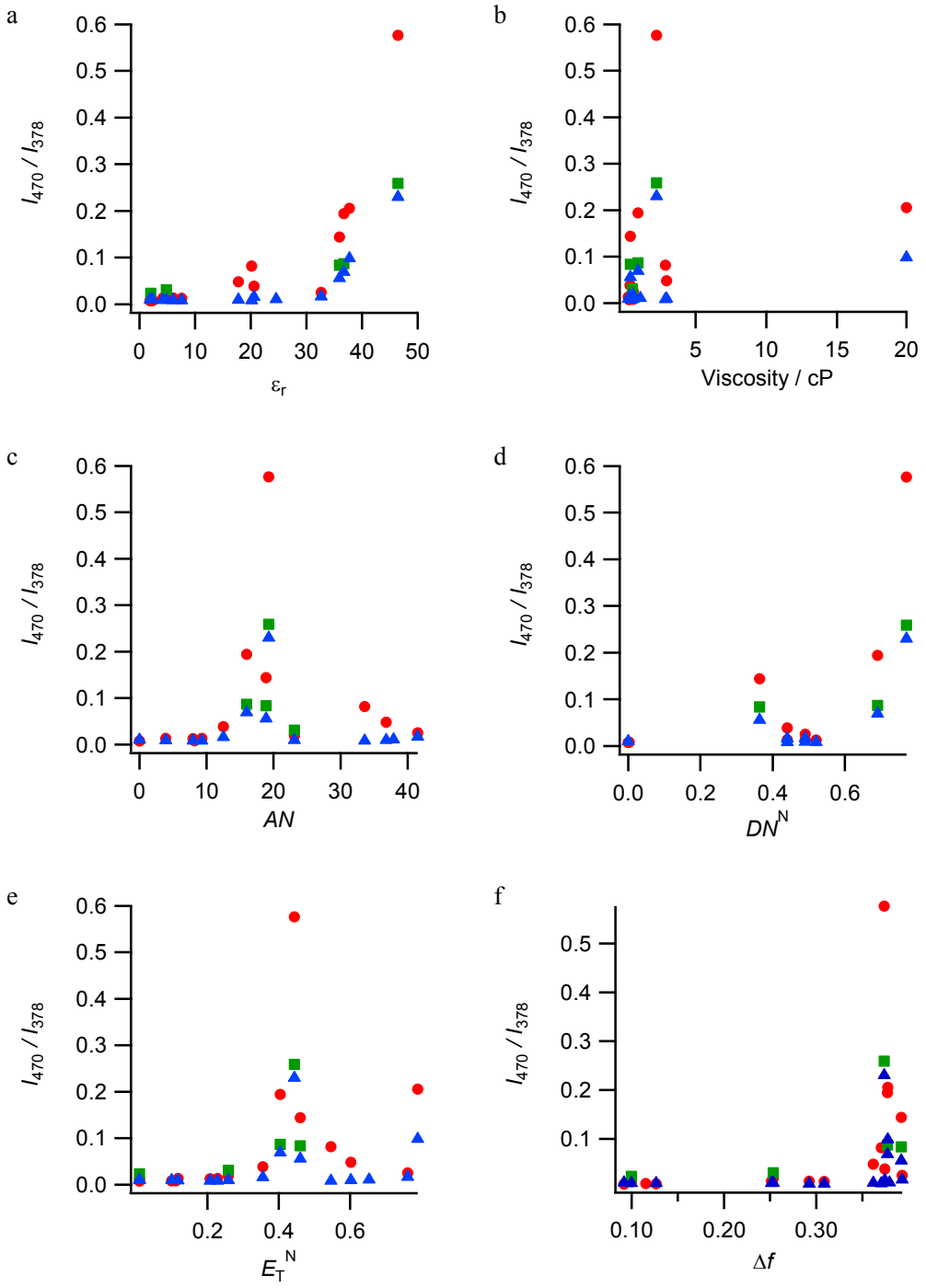

Fig. S19. Effect of solvent parameters, namely dielectric constant (ε_r), viscosity, acceptor number (*AN*), donor number (DN^N), normalized E_T value (E_T^N), and solvent orientation polarizability factor (Δf) on the ratios of excimer-like and monomer emissions (I_{470}/I_{378}) of **1a**–**c**. λ_{ex} = 348 nm. ●: **1a**, ■: **1b**, and ▲: **1c**.

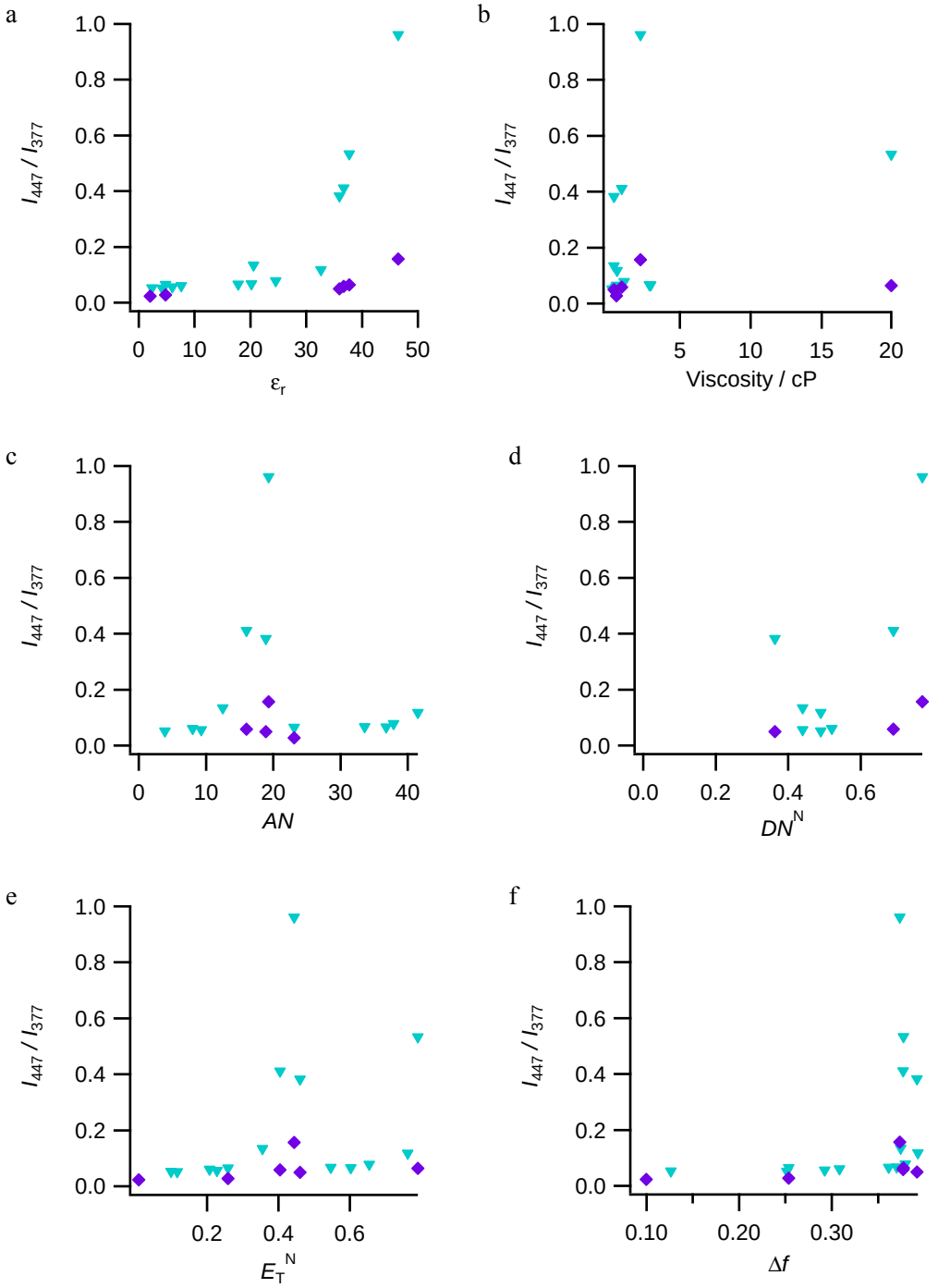

Fig. S20. Effect of the ratios of excimer-like and monomer emissions (I_{447}/I_{377}) of **3a** and **3b** on solvent parameters, namely dielectric constant (ε_r), viscosity, acceptor number (AN), donor number (DN^N), normalized E_T value (E_T^N), and solvent orientation polarizability factor (Δf) . λ_{ex} = 348 nm. ▼: **3a** and ◆: **3b**.

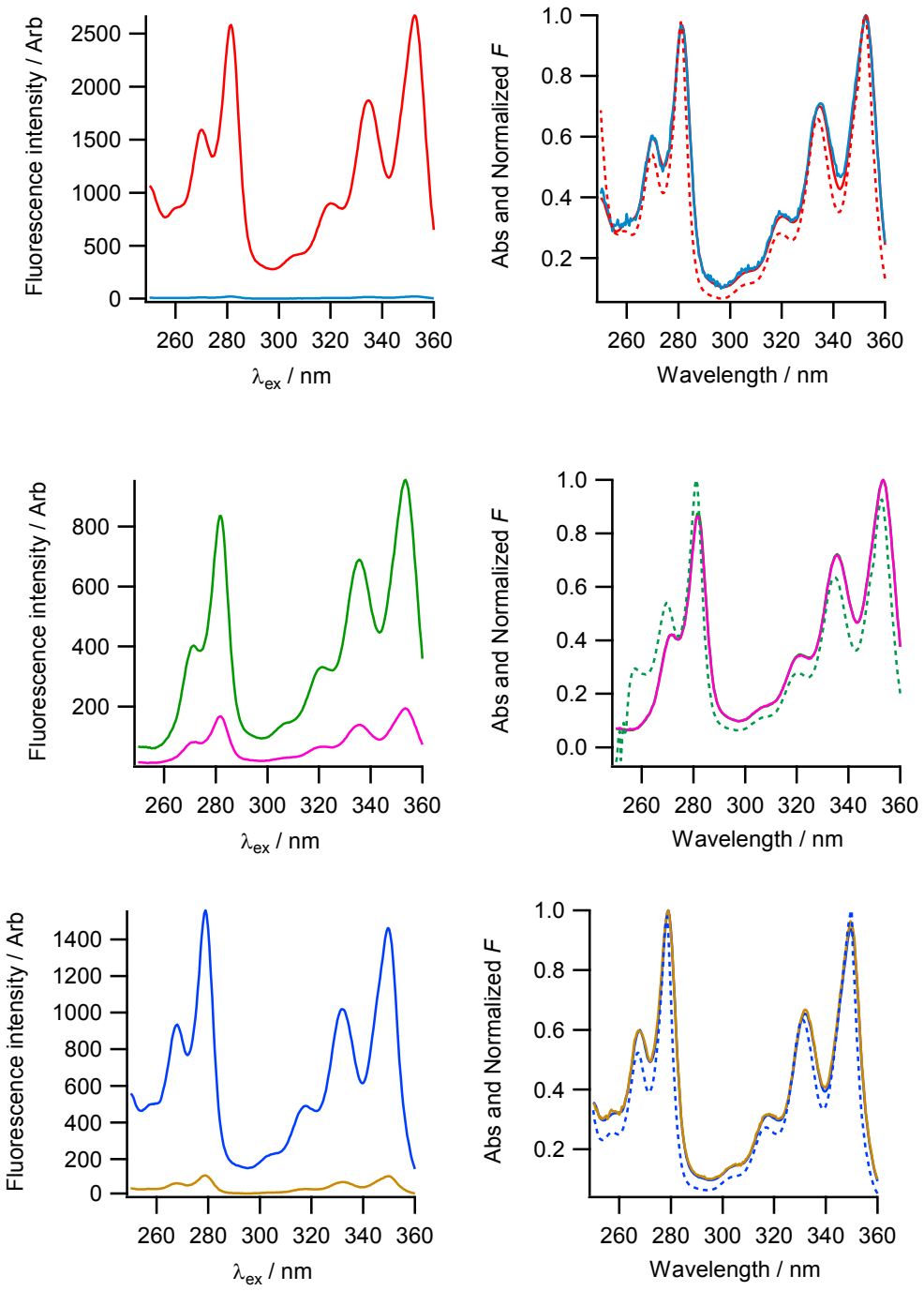

Fig. S21. (Left) Excitation spectra of **1c** in $CHCl_3$ at 379 (—) and 470 nm (—), in DMSO at 379 (—) and 470 nm (—), and in MeCN at 379 (—) and 470 nm (—), respectively. (Right) UV-vis spectra of **1c** in the corresponding solvents were overlapped as dotted lines to the normalized fluorescence spectra, respectively.

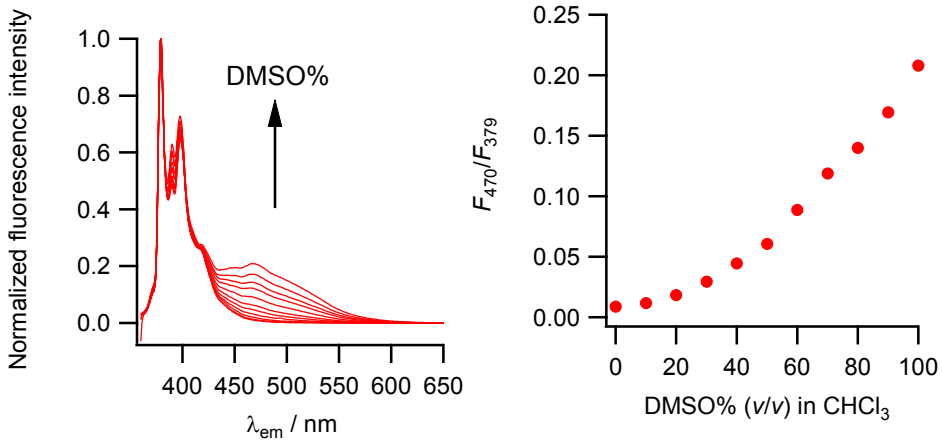

Fig. S22. Effect of DMSO–$CHCl_3$ on fluorescence spectra of **1c**. [**1c**] = 2.0×10^{-6} mol dm^{-3}, λ_{ex} = 348 nm at 298 K.

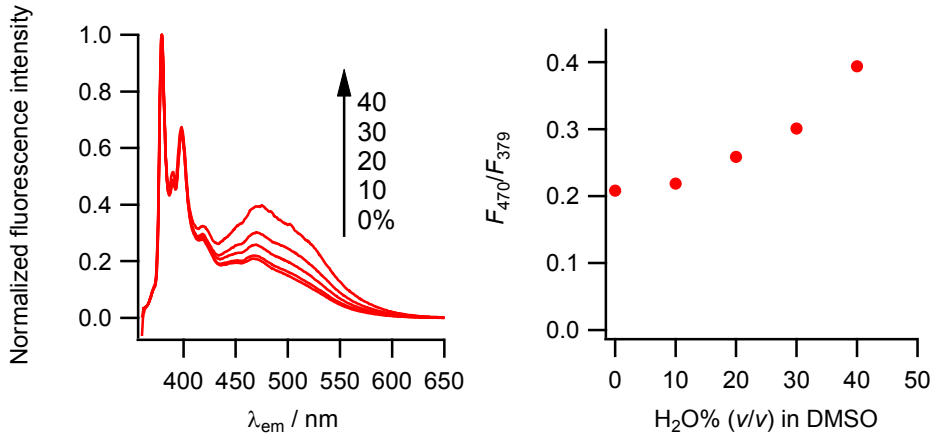

Fig. S23. Effect of water–DMSO on fluorescence spectra of **1c**. [**1c**] = 2.0×10^{-6} mol dm^{-3}, λ_{ex} = 348 nm at 298 K.

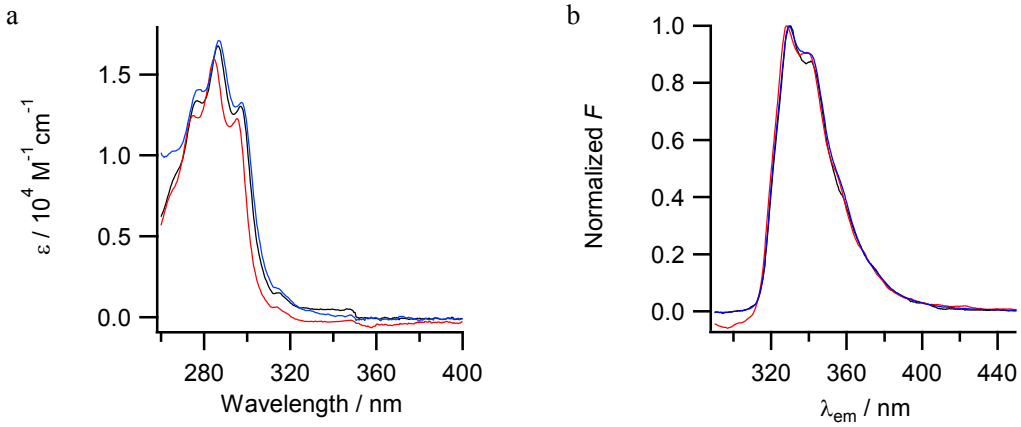

Fig. S24. UV-vis (a) and fluorescence (b) spectra of dimethyldi(1-naphthyl)silane in DMSO (—), MeCN (—), and $CHCl_3$ (—).

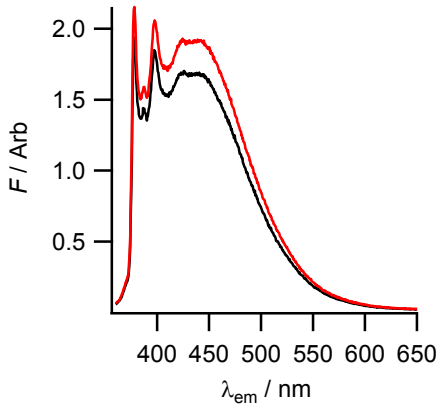

Fig. S25. Fluorescence spectra of **3a** under aerobic (—) and argon saturated (—) conditions in DMSO. [**3a**] = 2.0×10^{-6} mol dm^{-3}, λ_{ex} = 348 nm.

Table S1 The ratios of the fluorescence intensities of excimer and monomer emissions of **1a-c**, **3a**, and **3b** in various solvents

	I_{470}/I_{378}			I_{447}/I_{377}	
solvents	**1a**	**1b**	**1c**	**3a**	**3b**
hexane	0.0074		0.0102	ND[a]	
cyclohexane	0.0125	0.0234	0.0095	ND[a]	0.0233
benzene	0.0084				
toluene	0.0082		0.0094	0.0530	
ether	0.0132		0.0090	0.0522	
chloroform	0.0192	0.0309	0.0098	0.0653	0.0278
ethyl acetate	0.0136		0.0081	0.0565	
THF	0.0128		0.0083	0.0602	
1-butanol	0.0482		0.0096	0.0664	
2-propanol	0.0818		0.0084	0.0674	
acetone	0.0389		0.0160	0.1343	
ethanol			0.0106	0.0786	
methanol	0.0253		0.0165	0.1185	
acetonitrile	0.1439	0.0840	0.0558	0.3830	0.0491
DMF	0.1944	0.0872	0.0691	0.4117	0.0589
1,2-ethanediol	0.2056	ND[a]	0.0987	0.5336	0.0643
DMSO	0.5765	0.2592	0.2300	0.9618	0.1564

[a] Not determined due to the solubility of the compound in these solvents.

References

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