

The locations of triphenylamine and tetraphenylethene on a cyclohexyl ring define the luminogen as an AIEgen or a DSEgen

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1. Experimental

2.1 Reagents and instruments

All reagents and solvents are of analytical grade purchased from the commercial suppliers and used without further purification unless otherwise stated. Fourier transform infrared (FT-IR) spectra were obtained on the Nicolet Summit spectrometer (Thermo Fisher Scientific, Waltham, America) in the range of 4000-400 cm^{-1} , (KBr pellet). The ^1H NMR and ^{13}C NMR (100 MHz) spectra were obtained on a Bruker Avance 400 MHz spectrometer (Bruker, Massachusetts, America) with CDCl_3 and $\text{DMSO-}d_6$ as solvents, and tetramethylsilane (TMS) was used as an internal standard. High resolution mass spectroscopy (HRMS) was performed on a triple quadrupole mass spectrometer (ThermoFisher, Waltham, America) equipped with atmospheric pressure chemical ionization (APCI). UV-vis absorption spectra were recorded using a UV-3600 spectrometer (Shimadzu, Kyoto, Japan) with samples in solution and a quartz cuvette (path length 1 cm). Fluorescence spectra were obtained on a Hitachi FL-7000 (Hitachi High-Technologies Corporation, Tokyo, Japan). The absolute fluorescence quantum yields and lifetimes were measured on HORIBA Fluoro Max-4p spectrofluorometer using an integrating sphere (HORIBA Scientific, F-3092 integrating sphere (HORIBA JOBIN YVON, Paris, French)). Photo images were taken using a digital camera (Nikon D7000).

2.2 General procedures to prepare sample solutions

In this work, the test concentration of the three luminogens was 10 μM , which was obtained by diluting the aliquoted stock sample solutions (10 mM) with different solvents into calculated volume. The sample cell for UV-vis absorption spectrometry and fluorescence emission spectrometry was 1.0×1.0 cm double-sided transparent quartz cuvette and 1.0×1.0 cm four-sided transparent quartz cuvette.

2.3 Theoretical calculations

The ground state geometries and frontier molecular orbitals of the molecules were calculated using the hybrid B3LYP functional and the 6-31G* basis set, and density functional theory (DFT) was employed to optimize the geometries of the ground state (S_0) in the gas phase without conformation restrictions. Time-dependent DFT (TD-DFT) calculations was carried out to predict the vertical electronic excitation energies with the same functional and basis set. The electronic

geometries and frontier molecular orbital structures were plotted using the Gaussview 5.0 program package.

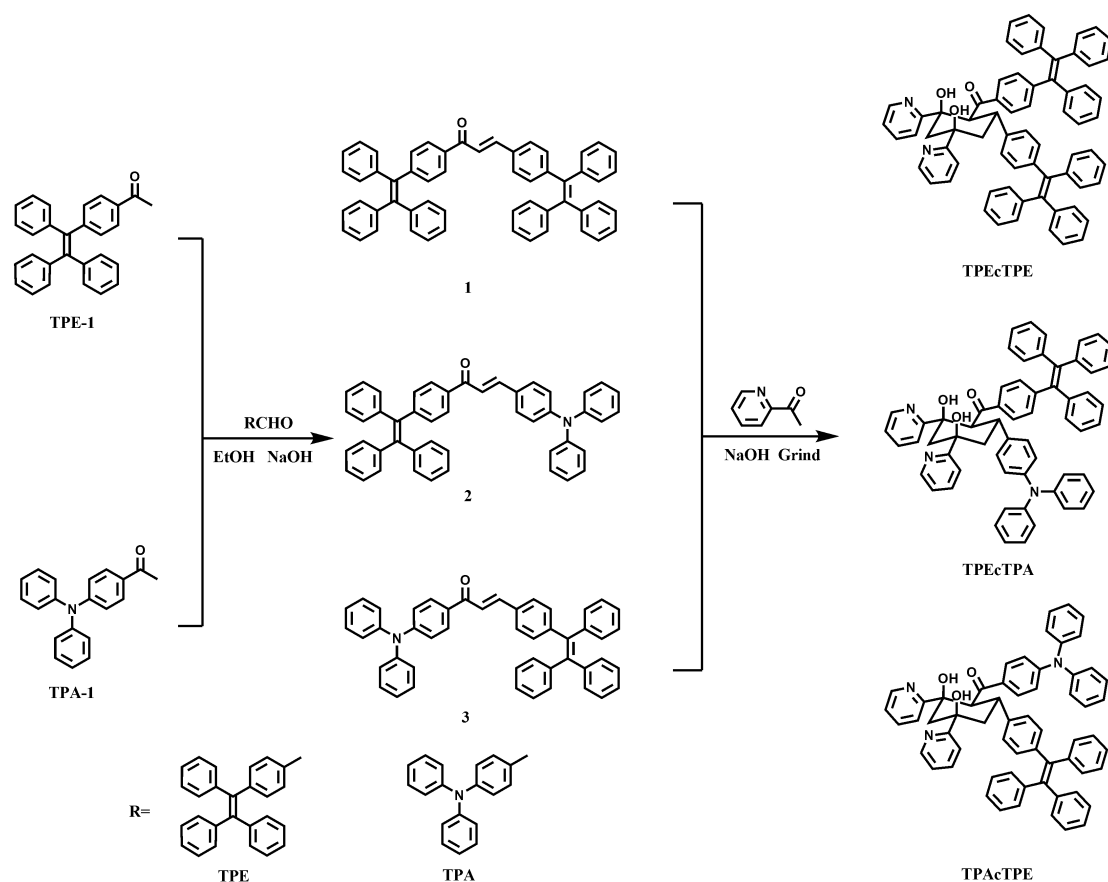
2.3 Details of PA detection

Stock solutions of the three organic luminogens (10 mM) were prepared by dissolving 9.58 mg of **TPEcTPE**, 8.71 mg of **TPEcTPA** and 8.71 mg of **TPAcTPE** in 10 mL of N,N-dimethylformamide, respectively. The working solutions were prepared by diluting three luminescent stock solutions to 10 μ M using pure THF and THF/H₂O (1:99) mixture, respectively. The selectivity of **TPEcTPE**, **TPEcTPA** and **TPAcTPE** were determined by monitoring the fluorescence intensities in the presence of 2,4,6-trinitrophenol (PA), 4-nitrobenzene (NB), p-dihydroxybenzene (p-DHM), o-nitrophenol (O-NP), m-dihydroxybenzene (m-DHB), p-methylphenol (p-MP) and P-nitrotoluene (P-NT). Besides, the strips of Whatman filter papers were prepared by cut into fixed shape (3.5 cm \times 3.5 cm rectangular) into dichloromethane solutions of TPAcTPE for 2 min, then dried under vacuum and stored for use, which enable the detection of PA in a feasible method.

2.4 Details of trace water detection

Stock solutions of TPAcTPE (10 mM) were prepared by dissolving 8.71 mg of **TPAcTPE** in 10 mL of N,N-dimethylformamide. The solution was then diluted to 10 μ M with the THF solvent and mixed evenly. The sample solutions in the pure organic solvents were freshly prepared prior to each measurement according to the above procedure. Then the sample solutions were added with different equivalent amounts of water into the sample solution. Then, the photoluminescence spectra were measured and recorded.

2.5 Synthesis



Scheme S1. Synthetic route for TPEcTPE, TPEcTPA, and TPAcTPE.

2.3.1 Synthesis of compounds 1-3

Compounds **TPE-1** and **TPA-1** were synthesized according to literature methods [S1,S2].

Compound 1: To a stirred solution of **TPA-1** (200 mg, 0.70 mmol) and **TPE-CHO** (251 mg, 0.70 mmol) in ethanol (25 mL) was added NaOH (0.10 g, 2.50 mmol) and 15 mL H₂O and the reaction mixture was stirred at room temperature for overnight. The progress of the reaction was monitored by thin layer chromatography (TLC). After completion, the reaction mixture was cooled to room temperature and the precipitate was filtered, washed with water, and further purified by flash chromatography using petroleum ether (PE)/ethyl acetate (EA) (50/1, v/v) as eluant. Pure product (263 mg, 0.41 mmol) as yellow solid was obtained, yield 60%. ¹H NMR (CDCl₃, 400 MHz) δ : 7.00-7.16 (m, 25H), 7.29-7.37 (m, 6H), 7.42 (d, 1H, $J = 15.60$ Hz), 7.69 (d, 1H, $J = 15.60$ Hz), 7.86 (d, 2H, $J = 8.64$ Hz). ¹³C NMR (CDCl₃, 100 MHz) δ : 188.27, 152.14,

146.60, 146.21, 143.53, 143.41, 142.06, 140.32, 133.28, 131.98, 131.46, 131.41, 131.40, 130.80, 130.19, 129.70, 127.94, 127.89, 127.86, 127.76, 126.86, 126.74, 126.07, 124.73, 121.53, 119.95.

Compound 2: To a stirred solution of **TPE-1** (200 mg, 0.53 mmol) and **TPE-CHO** (193 mg, 0.53 mmol) in ethanol (25 mL) was added NaOH (0.10 g, 2.50 mmol) and 15 mL H₂O and the reaction mixture was stirred at room temperature for overnight. The progress of the reaction was monitored by TLC. After completion, the reaction mixture was cooled to room temperature and the precipitate was filtered, washed with water, and further purified by flash chromatography using PE/EA (15/1, v/v) as eluant. Pure product (165 mg, 0.23 mmol) as yellow solid was obtained, yield 43%. ¹H NMR (CDCl₃, 400 MHz) δ : 7.01-7.14 (m, 34H), 7.33-7.39 (m, 3H), 7.66 (d, 1H, J = 15.68 Hz), 7.73 (d, 2H, J = 8.32 Hz). ¹³C NMR (CDCl₃, 100 MHz) δ : 189.84, 148.82, 146.48, 144.27, 143.50, 143.36, 143.35, 143.28, 143.18, 142.66, 142.17, 140.26, 140.06, 136.13, 133.03, 132.00, 131.65, 131.45, 131.41, 131.40, 128.02, 127.98, 127.95, 127.90, 127.80, 127.77, 127.01, 126.88, 126.85, 126.77, 121.59.

Compound 3: To a stirred solution of **TPE-1** (200 mg, 0.53 mmol) and **TPA-CHO** (146 mg, 0.53 mmol) in ethanol (25 mL) was added NaOH (0.10 g, 2.50 mmol) and 15 mL H₂O and the reaction mixture was stirred at room temperature for overnight. The progress of the reaction was monitored by TLC. After completion, the reaction mixture was cooled to room temperature and the precipitate was filtered, washed with water, and further purified by flash chromatography using PE/EA (10/1, v/v) as eluant. Pure product (227 mg, 0.36 mmol) as yellow solid was obtained, yield 52%. ¹H NMR (CDCl₃, 400 MHz) δ : 6.99-7.13 (m, 25H), 7.27-7.33 (m, 5H), 7.45 (d, 2H, J = 8.64 Hz), 7.70-7.75 (m, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ : 189.90, 150.21, 148.58, 146.95, 144.37, 143.39, 143.31, 143.24, 142.56, 140.12, 136.48, 131.61, 131.42, 131.37, 129.78, 129.60, 128.03, 127.97, 127.93, 127.79, 126.97, 126.82, 125.56, 124.19, 121.68, 119.40.

2.3.2 Synthesis of compound **TPEcTPE**

Compound 1 (500 mg, 0.69 mmol), 2-acetylpyridine (338 mg, 2.79 mmol) and NaOH (139 mg, 3.49 mmol) was thoroughly mixed and ground in a pestle and mortar for half an hour. The resulting mixture was dissolved in dichloromethane, extracted and dried, and further purified by flash chromatography using PE/EA (5/1, v/v) as eluant. Pure product **TPEcTPE**(407 mg, 0.43 mmol) as white solid. Yield: 61%. FT-IR (cm⁻¹, KBr) ν : 3057, 3025, 2922, 2858, 1649, 1592, 1494, 1447, 1407, 1216, 754, 701, 626. ¹H NMR (DMSO-d₆, 400 MHz) δ : 8.50 (d, 1H, J = 4.80 Hz), 8.26 (d, 1H, J

= 4.72 Hz), 7.79 (d, 1H, $J = 8.00$ Hz), 7.67 (t, 1H, $J = 7.56$ Hz), 7.50-7.58 (m, 2H), 7.25 (s, 1H), 7.23 (s, 1H), 7.13-6.82 (m, 36H), 6.71 (d, 2H, $J = 8.20$ Hz), 6.03 (d, 2H, $J = 8.12$ Hz), 4.77 (d, 1H, $J = 11.84$ Hz), 3.82 (t, 1H, $J = 12.40$ Hz), 2.88 (d, 1H, $J = 14.52$ Hz), 2.66 (d, 1H, $J = 13.36$ Hz), 2.03-1.92 (m, 2H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ : 208.62, 165.53, 163.97, 148.37, 148.29, 143.99, 143.80, 143.61, 143.49, 143.30, 143.17, 142.83, 141.93, 140.72, 140.55, 139.82, 139.76, 136.78, 136.54, 131.50, 131.43, 131.36, 131.29, 131.22, 130.96, 128.05, 127.94, 127.86, 127.78, 127.75, 127.71, 127.65, 127.06, 126.86, 126.53, 126.45, 122.08, 121.85, 119.92, 119.53, 80.03, 75.90, 54.51, 46.62, 45.63, 40.88. HR-MS (APCI-MS): m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{69}\text{H}_{55}\text{N}_2\text{O}_3^+$: 959.4213, found: 959.4275.

2.3.3 Synthesis of compound TPEcTPA

Compound **TPEcTPA** was synthesized according to the similar procedure described for the synthesis of compound **TPEcTPE** using **TPA-CHO** instead of **TPE-CHO**. The difference is that the crude product was further purified by flash chromatography using PE/EA (3/1, v/v) as eluant to afford compound **TPEcTPA** (325 mg, 0.37 mmol). Yield: 47%. FT-IR (cm^{-1} , KBr) ν : 3032, 2918, 1740, 1639, 1588, 1508, 1491, 1446, 1403, 1320, 1280, 1219, 1182, 1080, 996, 843, 773, 753, 701, 627, 545, 507. ^1H NMR (DMSO- d_6 , 400 MHz) δ : 8.51 (d, 1H, $J = 5.72$ Hz), 8.28 (d, 1H, $J = 5.72$ Hz), 7.81 (d, 1H, $J = 8.00$ Hz), 7.69-7.52 (m, 3H), 7.29 (d, 2H, $J = 8.52$ Hz), 7.19-7.15 (m, 3H), 7.13-6.97 (m, 14H), 6.96-6.84 (m, 14H), 6.78 (d, 2H, $J = 8.92$ Hz), 6.17 (d, 1H, $J = 2.08$ Hz), 6.10 (s, 1H), 4.80 (d, 1H, $J = 11.88$ Hz), 3.85 (t, 1H, $J = 12.64$ Hz), 2.87 (d, 1H, $J = 14.58$ Hz), 2.75 (t, 1H, $J = 13.40$ Hz), 2.09 (d, 1H, $J = 13.40$ Hz), 1.98 (d, 1H, $J = 14.58$ Hz). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ : 208.64, 170.92, 167.37, 166.69, 165.56, 163.97, 148.35, 148.28, 148.26, 143.99, 143.79, 143.60, 143.29, 143.16, 142.82, 141.92, 140.71, 140.55, 139.81, 139.76, 136.76, 131.49, 131.43, 131.35, 131.29, 131.22, 130.95, 128.04, 127.93, 127.85, 127.78, 127.74, 127.71, 127.64, 127.05, 126.85, 126.52, 126.44, 122.06, 121.84, 119.91, 119.52, 80.04, 75.90, 54.50, 46.63, 45.63, 40.89. HR-MS (APCI-MS): m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{61}\text{H}_{50}\text{N}_3\text{O}_3^+$: 872.3852, found: 872.3921.

2.3.4 Synthesis of compound TPActPE

TPActPE was synthesized according to the similar procedure described for the synthesis of compound **TPEcTPE** using **TPA-1** instead of **TPE-1**. The difference is that the crude product was further purified by flash chromatography using PE/EA (1/1, v/v) as eluant to afford compound

TPAcTPE (351 mg, 0.40 mmol). Yield: 56%. FT-IR (cm^{-1} , KBr) ν : 3053, 3025, 2913, 1634, 1508, 1489, 1223, 994, 896, 843, 751, 698, 619, 577, 537. ^1H NMR ($\text{DMSO-}d_6$, 400 MHz) δ : 8.53 (s, 1H), 8.33 (s, 1H), 7.82 (s, 1H), 7.69-7.25 (m, 7H), 7.12-6.88 (m, 25H), 6.76-6.71 (m, 6H), 6.42 (s, 1H), 6.16 (s, 1H), 4.78 (d, 1H, $J = 11.88$ Hz), 3.82 (t, 1H, $J = 12.84$ Hz), 2.93-2.72 (m, 2H), 2.06-1.93 (m, 2H). ^{13}C NMR ($\text{DMSO-}d_6$, 100 MHz) δ : 206.54, 165.83, 164.42, 152.00, 148.33, 146.36, 144.05, 143.80, 143.72, 141.94, 140.61, 140.06, 136.76, 136.70, 131.89, 131.43, 131.36, 131.29, 130.70, 130.55, 129.71, 127.81, 127.70, 127.66, 127.57, 126.50, 126.38, 126.12, 124.89, 118.56, 80.08, 75.94, 53.62, 46.55, 45.15, 40.86. HR-MS (APCI-MS): m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{61}\text{H}_{50}\text{N}_3\text{O}_3^+$: 872.3852, found: 872.3908.

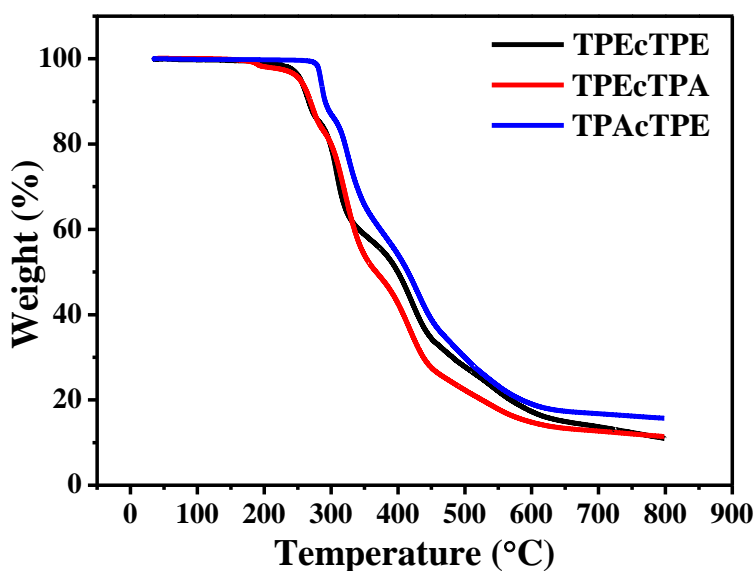


Fig. S1 UV-vis spectra of (a) TPEcTPE, (b) TPEcTPA, and (c) TPAcTPE in solvents with varying polarity, $c = 10 \mu\text{M}$, 25°C .

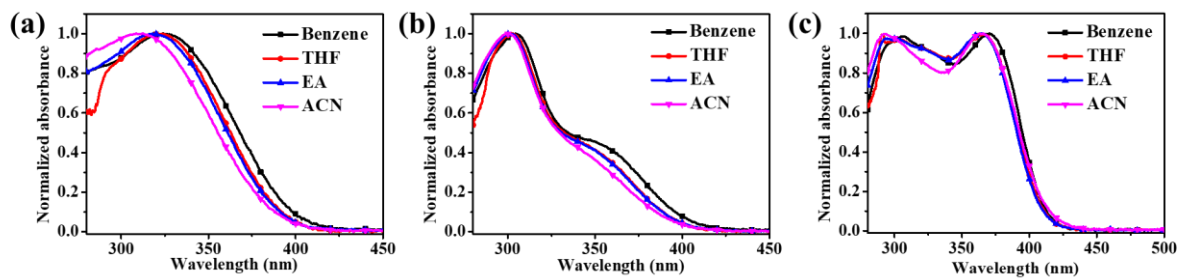


Fig. S2 UV-vis spectra of (a) TPEcTPE, (b) TPEcTPA, and (c) TPAcTPE in solvents with varying polarity, $c = 10 \mu\text{M}$, 25°C .

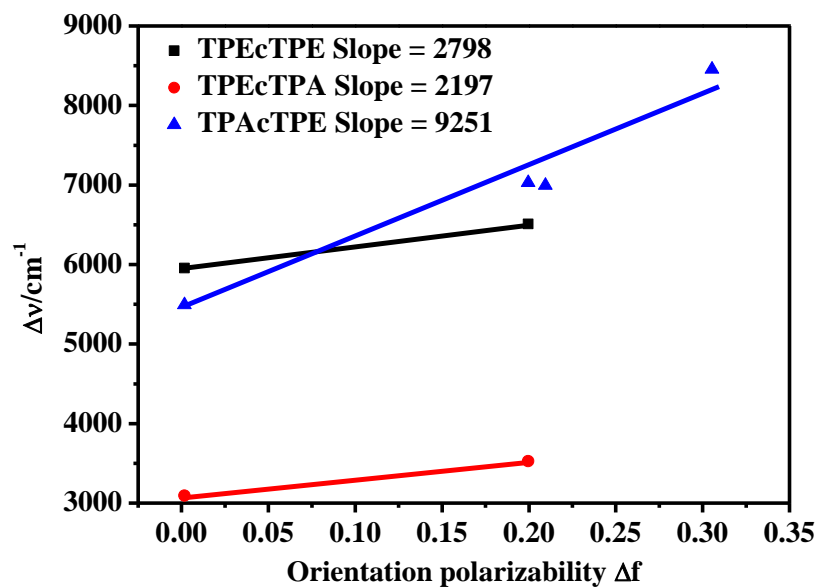


Fig. S3 Plot of Stokes shift ($\Delta\nu$) versus solvent orientation polarizability (Δf) showing the solvatochromism effect of TPEcTPE, TPEcTPA, and TPAcTPE.

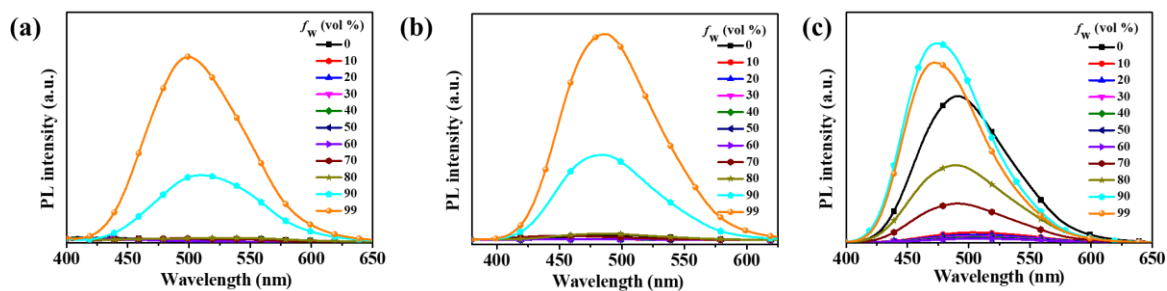


Fig. S4 PL spectra of (a) TPEcTPE, (b) TPEcTPA, and (c) TPAcTPE in THF/water mixtures with different water fraction (v/v %), $c = 10 \mu\text{M}$, 25°C .

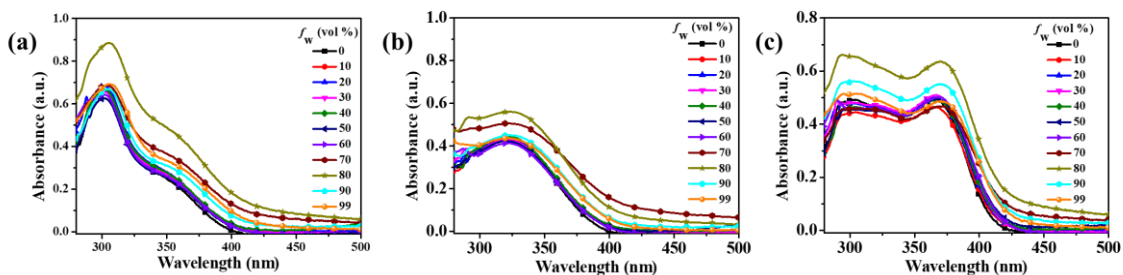


Fig. S5 UV-vis spectra of (a) TPEcTPE, (b) TPEcTPA, and (c) TPAcTPE in THF/water mixtures with different water fraction (v/v %), $c = 10 \mu\text{M}$, 25°C .

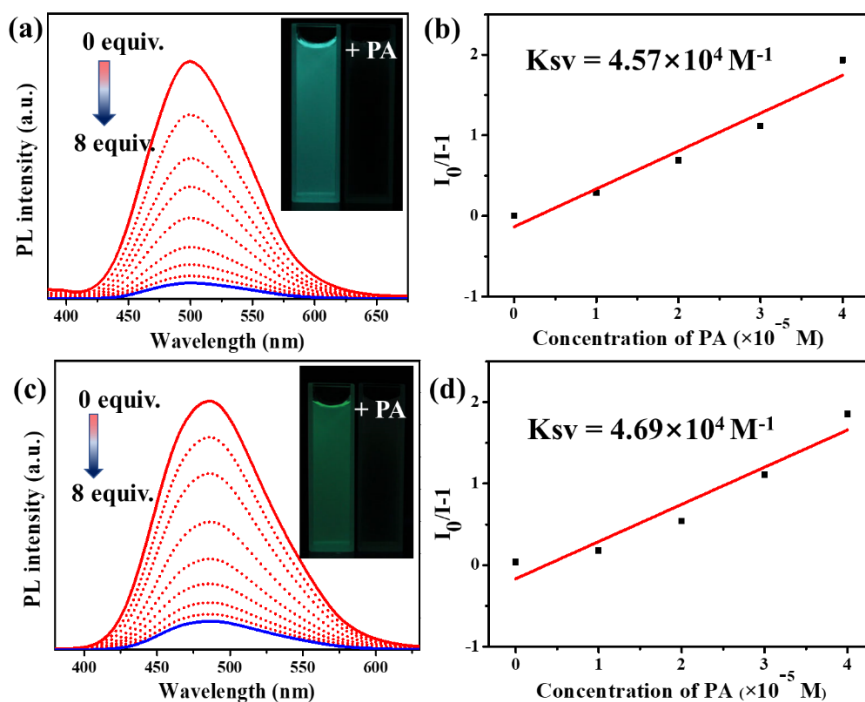


Fig. S6 PL spectra and the corresponding Stern-Volmer plot of (a, b) TPEcTPE and (c, d) TPEcTPA in THF/water mixtures (v/v 99%) with different PA concentration. Inset images show the photographs of samples before and after PA addition (8 equiv.) under 365 nm UV light illumination. $c = 10 \mu\text{M}$, 25°C .

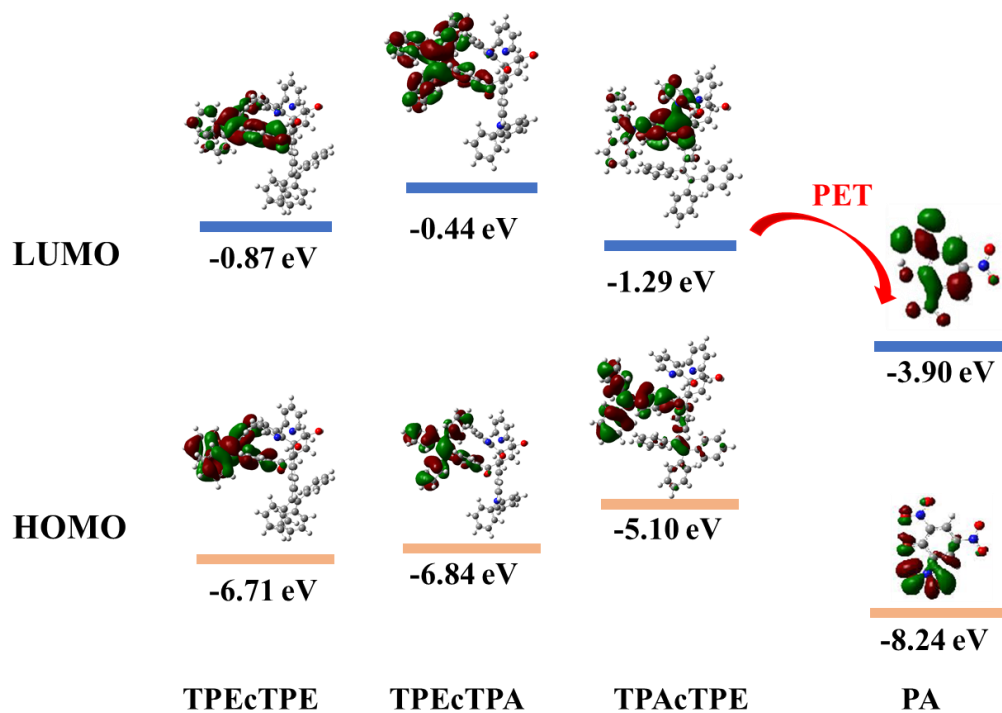


Fig. S7 Illustration of the PET process from the LUMOs of TPATPE, TPEcTPE, and TPEcTPA to the LUMO of PA.

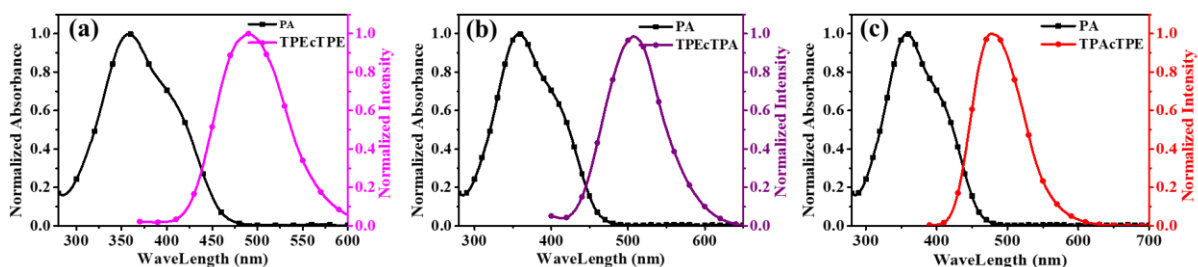


Fig. S8 Normalized UV-vis spectrum of PA overlap with normalized PL spectra of (a) TPEcTPE, (b) TPEcTPA, and (c) TPAcTPE in THF/Water mixture (v/v 99%). $c = 10 \mu\text{M}$, 25°C .

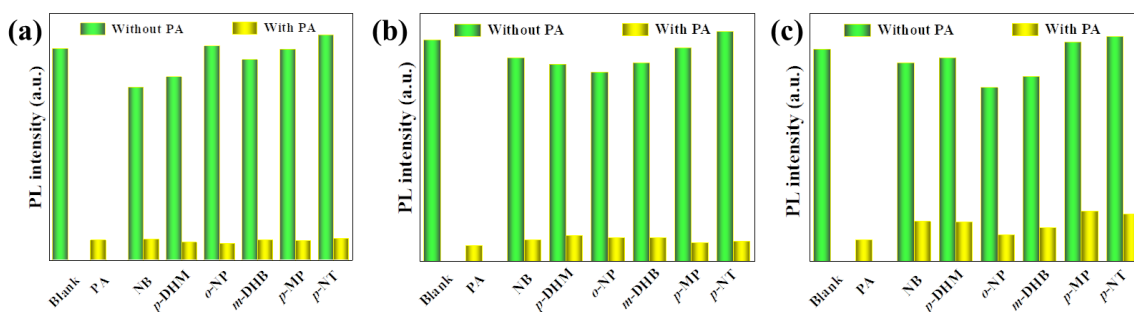


Fig. S9 Quenching effect of (a) TPEcTPE, (b) TPEcTPA, and (c) TPAcTPE in THF/Water mixture (v/v 99%) with various representative NACs before and after PA addition. 4-nitrobenzene (NB), p-dihydroxybenzene (p-DHM), o-nitrophenol (o-NP), m-dihydroxybenzene (m-DHB), p-methylphenol (p-MP) and p-nitrotoluene (p-NT). $c = 10 \mu\text{M}$, $25 \text{ }^\circ\text{C}$.

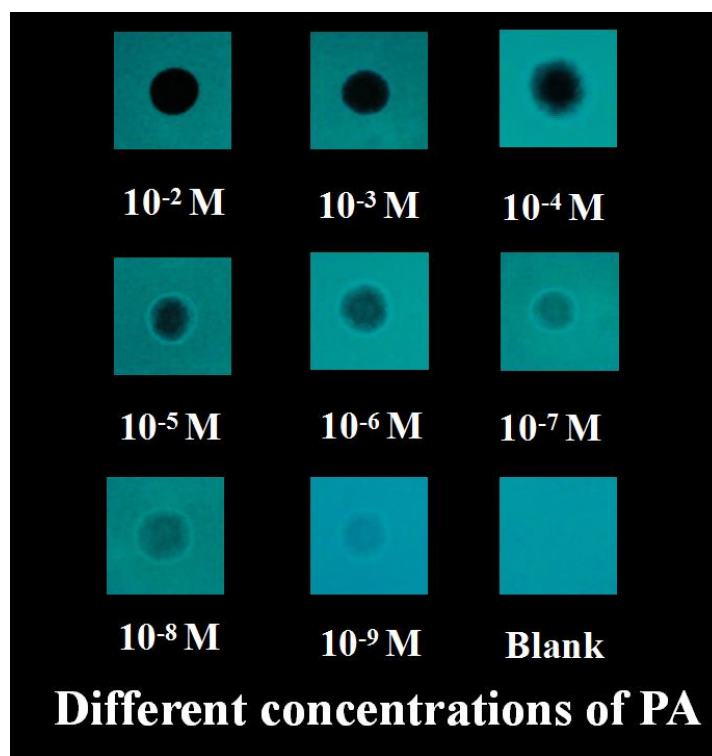


Fig. S10 PL quenching of TPAcTPE-coated filter paper by PA with various concentrations.

Table S1 A comparison of water detection of our work with the results collected from the reported literature

Materials	Response mode	Detection method	LOD (% , v/v)	Ref.
Triazaborolopyridinium derivatives (Compound 8)	Turn-off	Fluorescence	0.028	[S3]
EDA and catechol	Ratiometric	Fluorescence	0.178	[S4]
Triaminobenzene dihydrochloride and urea	Turn-on	Fluorescence	0.072	[S5]
Resorcylic acid and ethylenediamine (N-GCDs)	Turn-off	Fluorescence	0.36	[S6]
Carbon quantum dots (CDs)	Ratiometric	Fluorescence	0.103	[S7]
EDA and isophthalic acid	Turn-off	Phosphorescence	0.011	[S8]
Resorcinol-derive CDs	Turn-off	Fluorescence	0.006	[S9]
Lanthanide MOF	Ratiometric	Fluorescence	0.01	[S10]
Rhodamine 6G @Eu-MOF	Ratiometric	Fluorescence	0.032	[S11]
CDs/Cu NCs	Turn-off	Fluorescence	0.02	[S12]
triarylmethane derivatives (PyMBD)	Turn-off	Fluorescence	0.15	[S13]
Tetraphenylethylene based benzothiadiazole derivatives(<i>p</i> -NO ₂)	Turn-off	Fluorescence	0.014	[S14]
High-crystallinity COF	Ratiometric	Fluorescence	0.026	[S15]
BODIPY derivative	Ratiometric	Fluorescence	0.003	[S16]
organic DSEgens(TPAcTPE)	Turn-off	Fluorescence	0.00305	This work

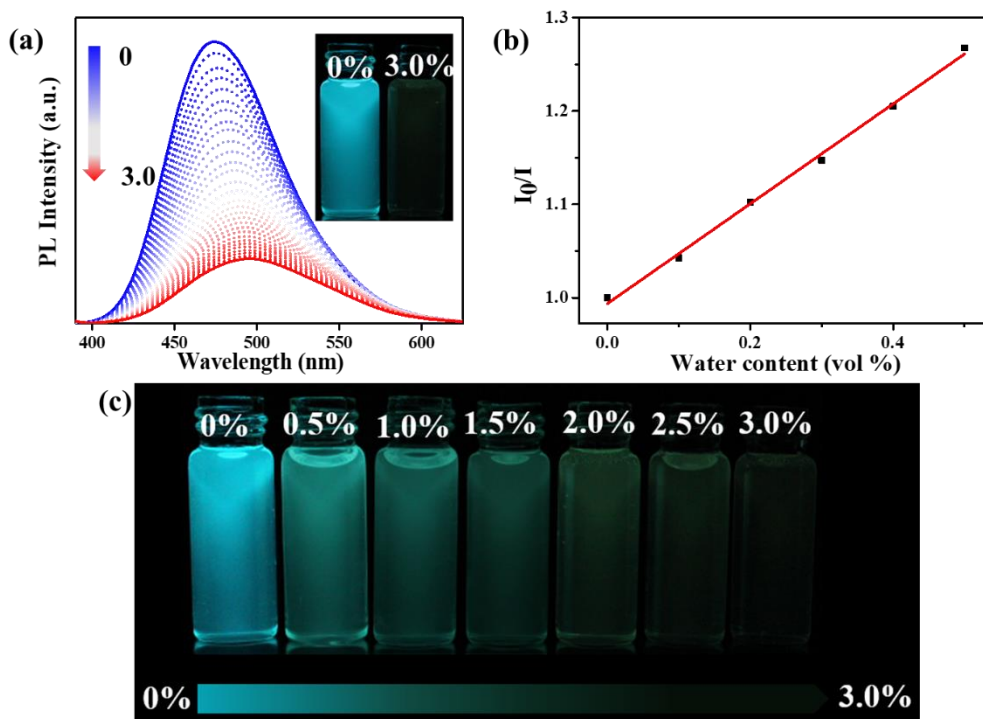


Fig. S11 (a) PL spectra of TPACTPE in dioxane upon addition of water. (b) Linear plotting of I_0/I against water content from 0 – 0.5%, I_0 is the original PL intensity, I is the PL intensity after addition of PA. (c) Photographs of THF solutions of TPACTPE with different water content (labeled on the top of the bottle) under 365 nm UV illumination. $c = 10 \mu\text{M}$, $25 \text{ }^\circ\text{C}$.

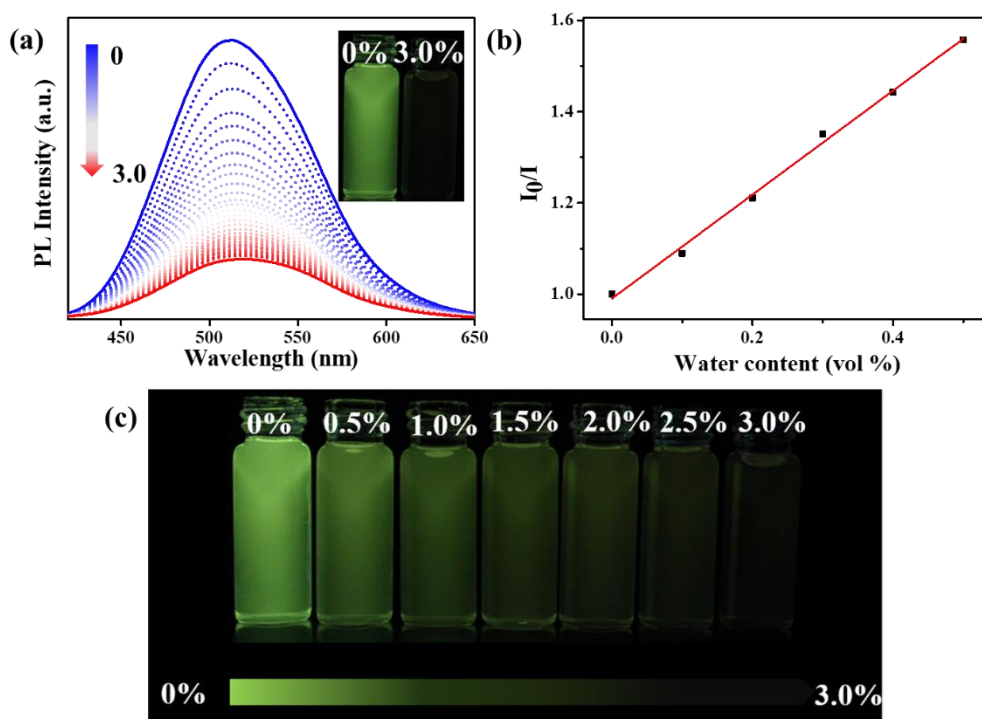


Fig. S12 (a) PL spectra of TPAcTPE in acetone upon addition of water. (b) Linear plotting of I_0/I against water content from 0 – 0.5%, I_0 is the original PL intensity, I is the PL intensity after addition of PA. (c) Photographs of THF solutions of TPAcTPE with different water content (labeled on the top of the bottle) under 365 nm UV illumination. $c = 10 \mu\text{M}$, $25 \text{ }^\circ\text{C}$.

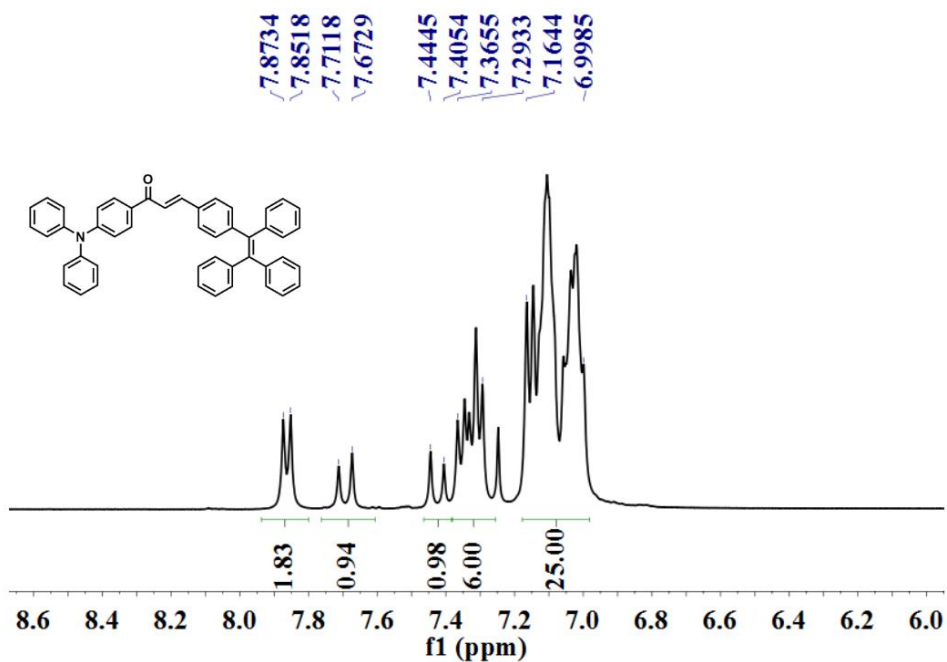


Fig. S13 ¹H NMR spectrum of compound 1 in CDCl₃.

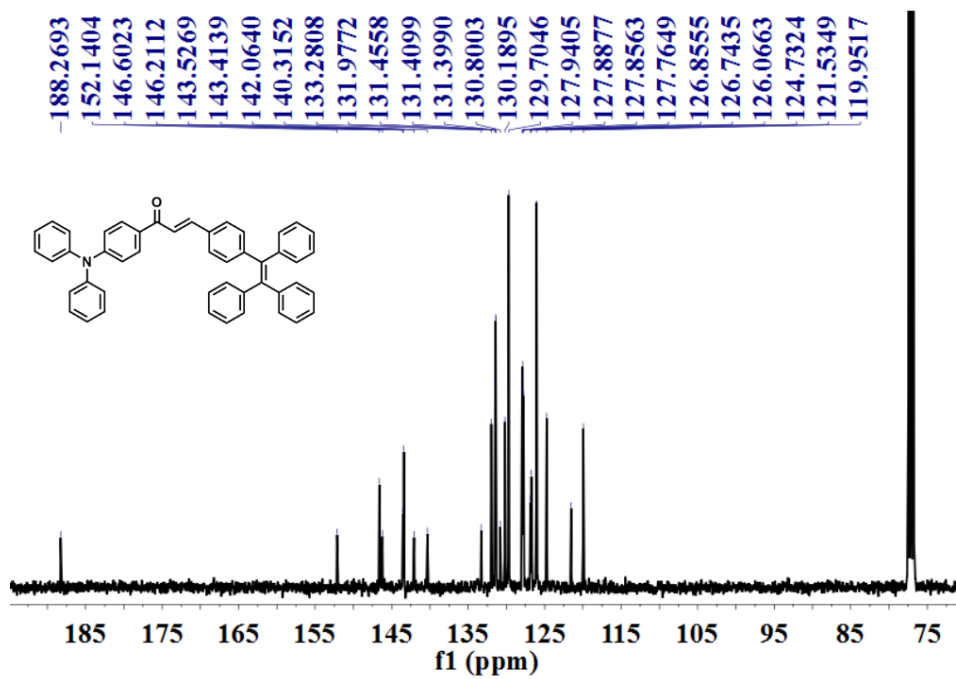


Fig. S14 ¹³C NMR spectrum of compound 1 in CDCl₃.

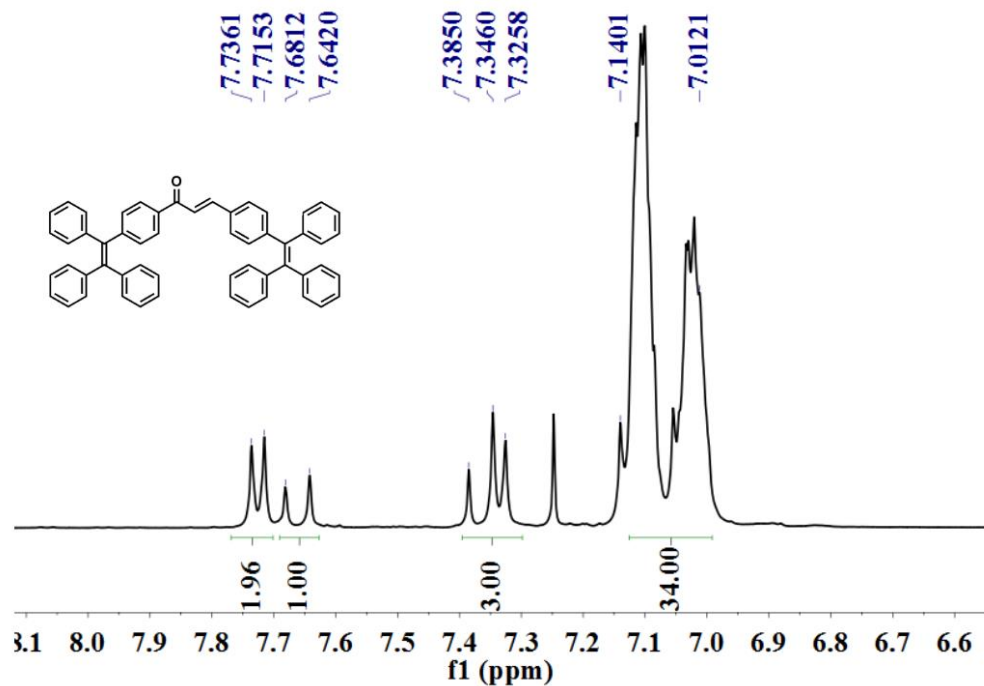


Fig. S15 ¹H NMR spectrum of compound 2 in CDCl₃.

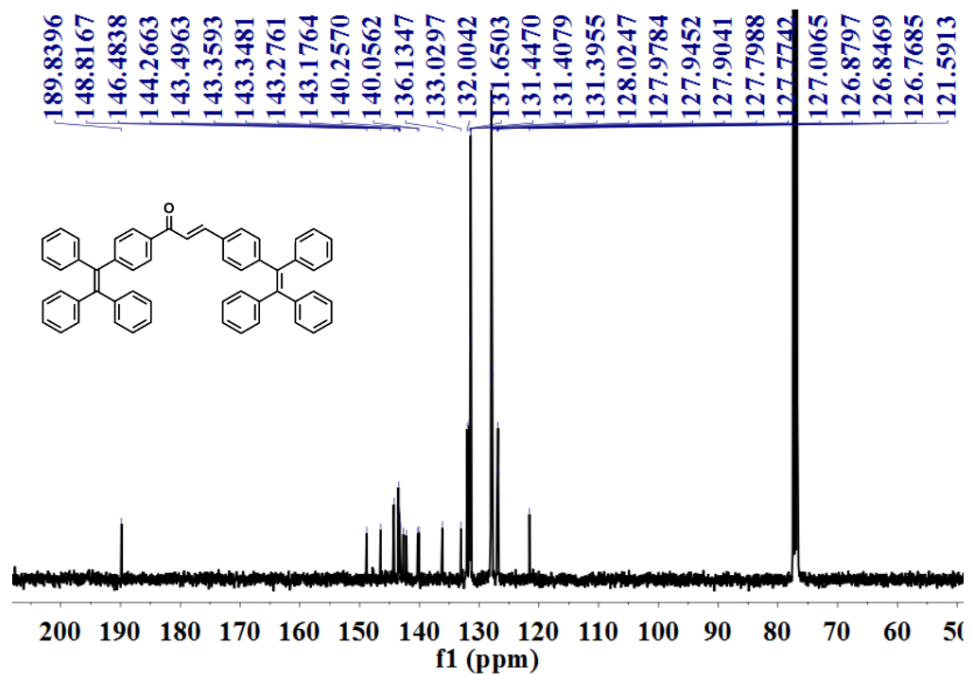


Fig. S16 ¹³C NMR spectrum of compound 2 in CDCl₃.

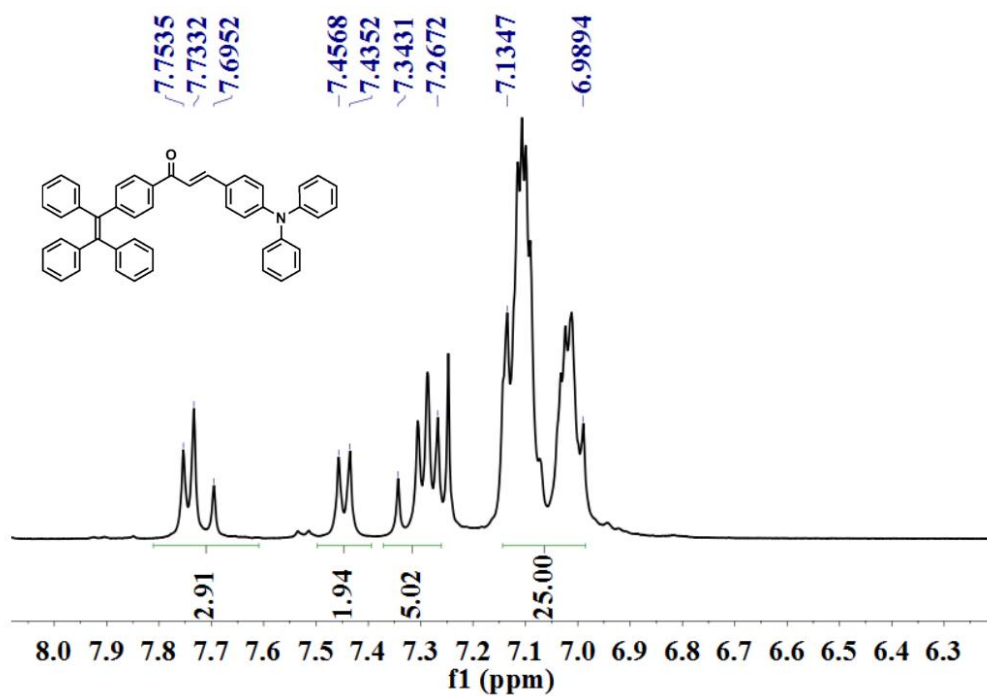


Fig. S17 ¹H NMR spectrum of compound 3 in CDCl₃.

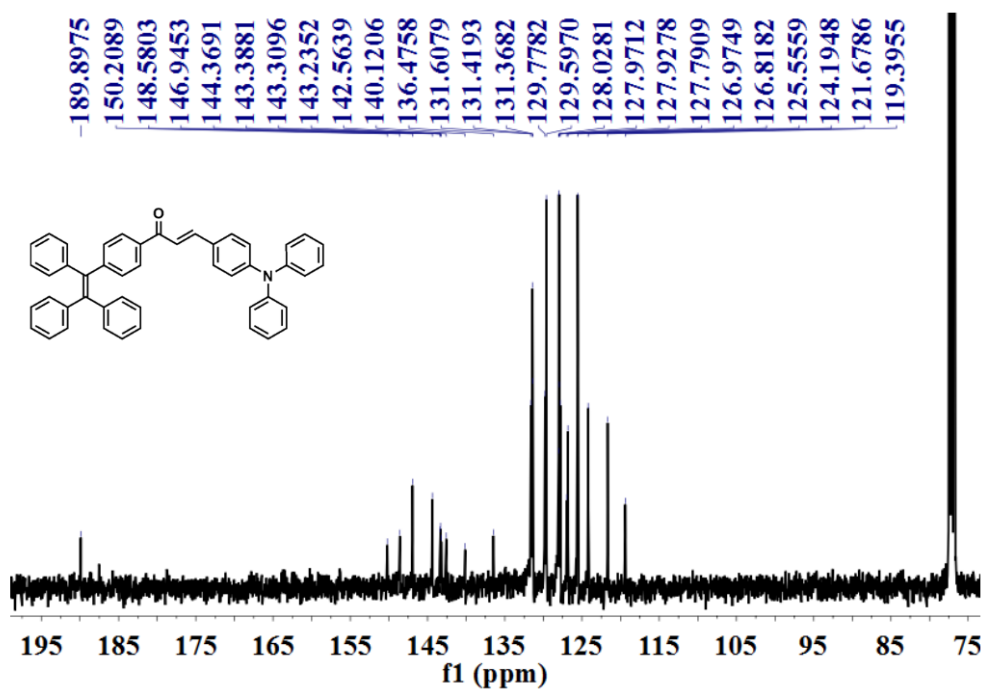


Fig. S18 ¹³C NMR spectrum of compound 3 in CDCl₃.

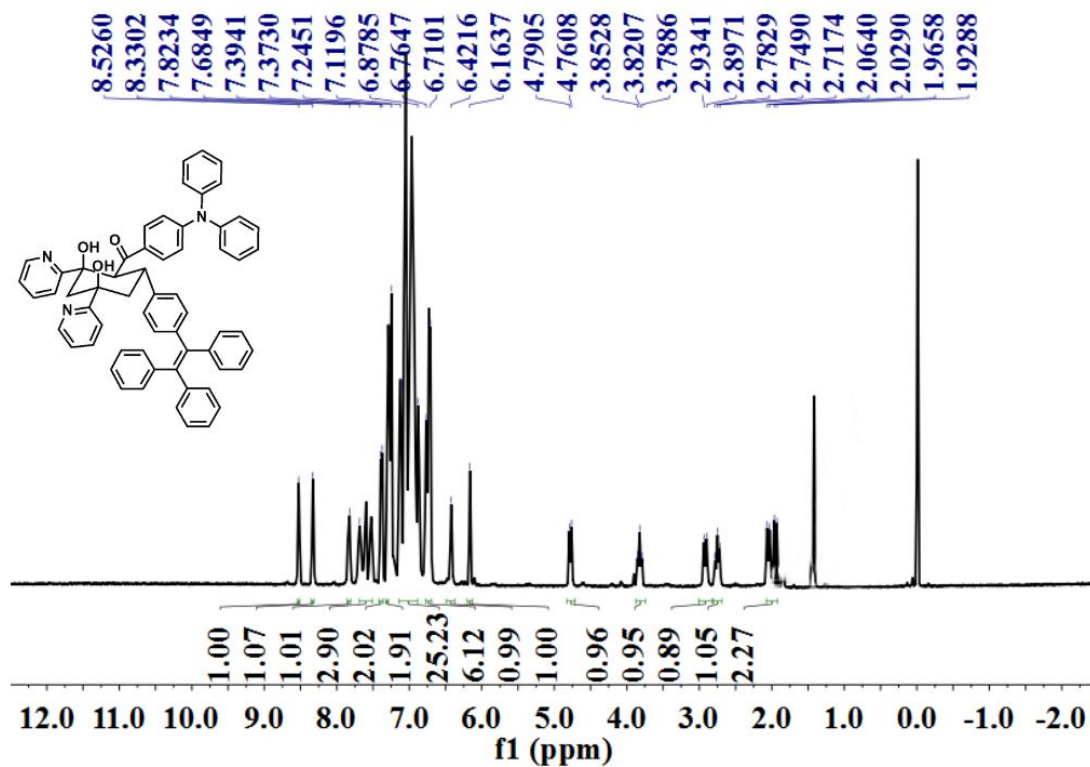


Fig. S19 ^1H NMR spectrum of compound TPAcTPE in DMSO-d_6 .

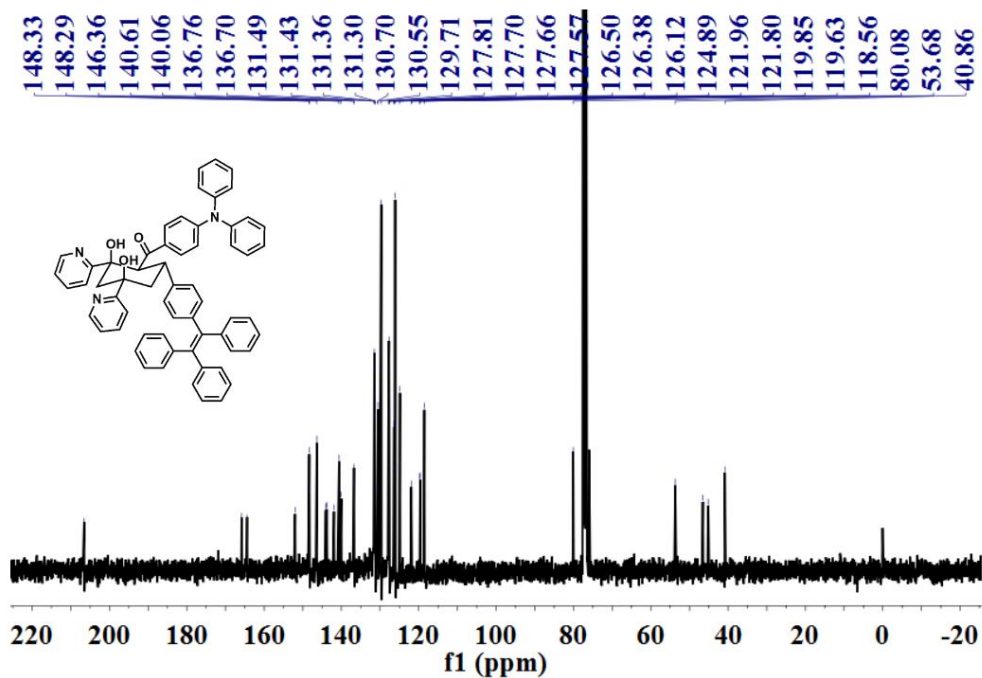


Fig. S20 ^{13}C NMR spectrum of compound TPAcTPE in DMSO-d_6 .

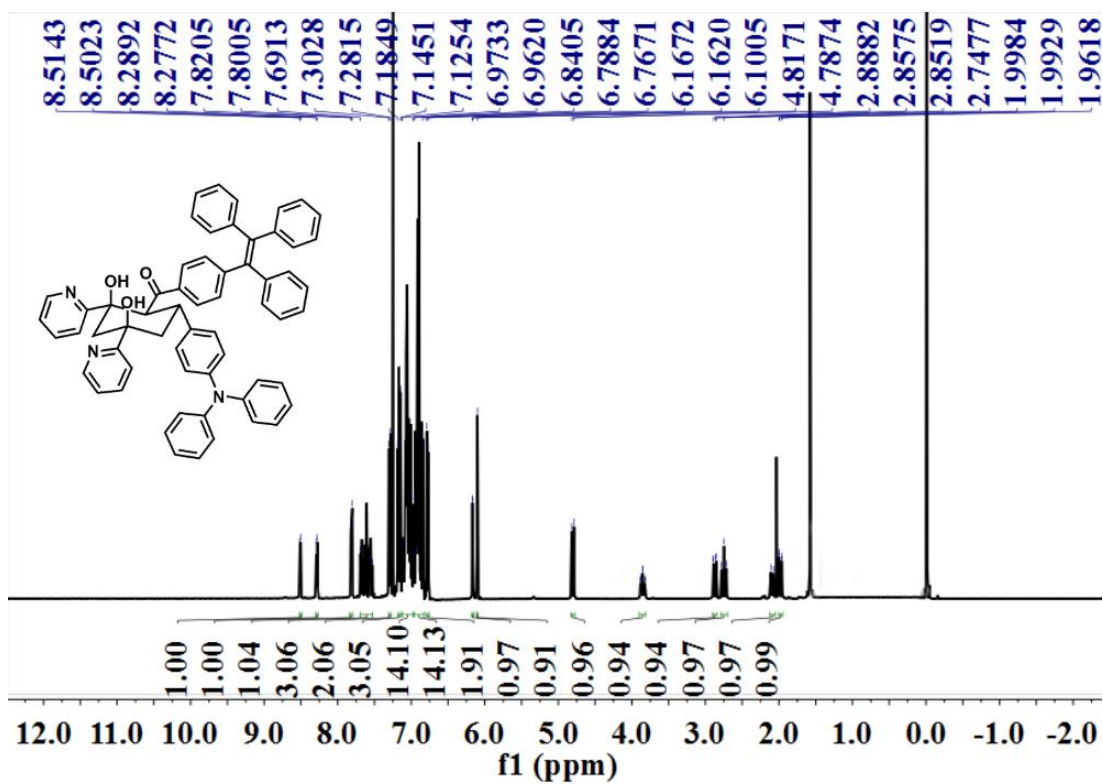


Fig. S23 ^1H NMR spectrum of compound TPEcTPA in DMSO- d_6 .

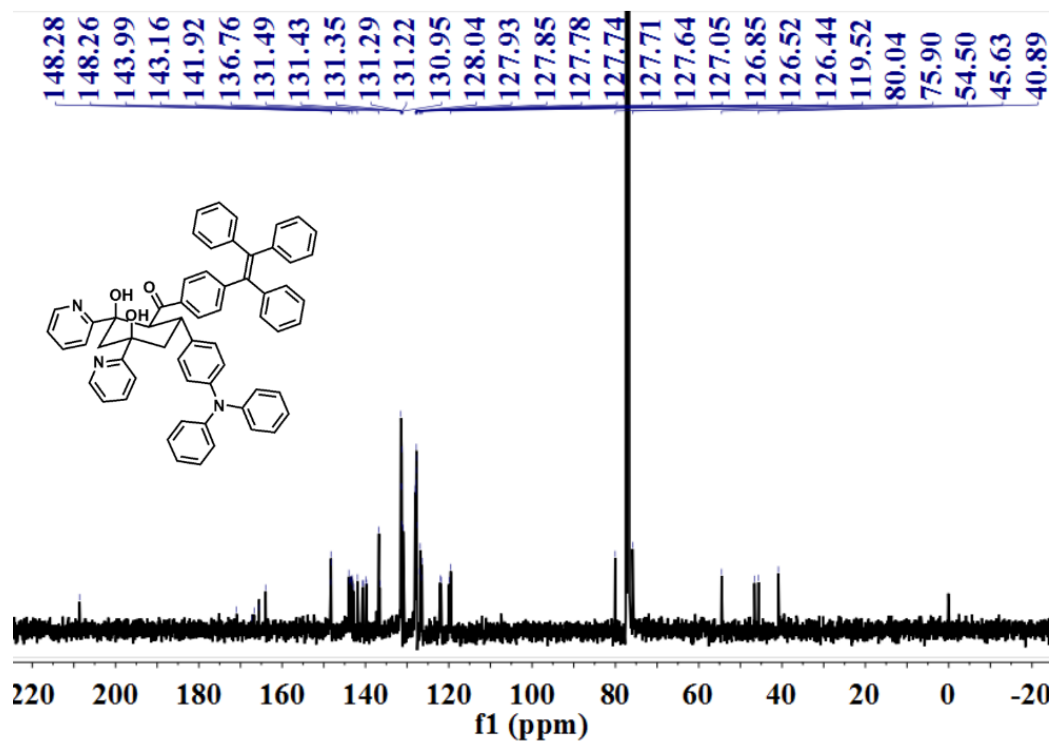


Fig. S24 ^{13}C NMR spectrum of compound TPEcTPA in DMSO- d_6 .

References:

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