Supporting Information for

Neutral branched platinum-acetylide complex possessing a tetraphenylethylene core: preparation of luminescent organometallic gelator and its unexpected spectroscopic behaviour during sol-to-gel transition

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1. General information
All reagents were analytical reagents and used without further treatment. THF used were dried according to standard procedures and degassed under N\textsubscript{2} for 30 minutes. Column chromatography was conducted by using silica-gel column.

\textsuperscript{1}H NMR, \textsuperscript{31}P NMR and \textsuperscript{13}C NMR spectra were recorded on Bruker 400 MHz Spectrometer (\textsuperscript{1}H: 400 MHz; \textsuperscript{31}P: 161.9 MHz; \textsuperscript{13}C: 100 MZH) at 298 K. The \textsuperscript{1}H and \textsuperscript{13}C NMR chemical shifts were reported relative to residual solvent signals, and \textsuperscript{31}P NMR resonances were referenced to an internal standard sample of 85\% H\textsubscript{3}PO\textsubscript{4} (δ 0.0). Coupling constants (\textit{J}) were denoted in Hz and chemical shifts (\textit{δ}) in ppm. Multiplicities were denoted as follows: s = singlet, d = doublet, m = multiplet, br = broad. Fluorescence spectra were recorded on Varian Cary Eclipse.

2. The synthesis of complex TPA
**Scheme S1** The synthesis of complex TPA.

Compound 2: A solution of trans-PtI$_2$(PMe$_3$)$_2$ (639 mg, 1.02 mmol) and CuI (4 mg, 10 mol%) in a mixture of THF/Et$_2$NH (40 mL THF and 45 mL Et$_2$NH) was stirred at room temperature. Then compound 1 (91 mg, 0.21 mmol) dissolved in THF was added dropwise to the reaction mixture under an atmosphere of nitrogen. The reaction was stirred at room temperature for 3 hours. The solvent was then removed in vacuo, the resulting residue was separated by column chromatography on silica gel and the yellow solid 2 was obtained (417 mg, 84%). $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 7.02-7.04 (m, 9H), 6.85-6.83 (m, 9H), 1.80-1.78 ppm (m, 72H). $^{31}$P NMR (CDCl$_3$, 161.9 MHz): $\delta = -22.49$ (s, $J_{Pt-P} = 1128.44$ Hz).


Compound TPA: A solution of compound 2 (150 mg, 0.07 mmol) and CuI (2 mg, 10 mol%) in a mixture of THF/Et$_2$NH (10 mL THF and 10
mL Et₂NH) was stirred at room temperature. Then compound 3 (200 mg, 0.07 mmol) dissolved in THF was added dropwise to the reaction mixture under an atmosphere of nitrogen. The reaction was stirred at room temperature for 2 hours. The solvent was then removed in vacuo, the resulting residue was separated by column chromatography on silica gel and the yellow solid TPA was obtained (266 mg, 92%). ¹H NMR (CDCl₃, 400 MHz): δ 7.63 (s, 4H), 7.48 (d, J = 8.4 Hz 8H), 7.20 (d, J = 7.6 Hz 8H), 7.02 (br, 17H), 6.84-6.82 (m, 8H), 4.03-4.01 (m, 24H), 1.80-1.78 (m, 96H), 1.47 (br, 24H), 1.26 (m, 216H), 0.90-0.86 (m, 36H). ³¹P NMR (CDCl₃, 161.9 MHz): δ = -20.21 (s, J_{Pt-P} = 1148 Hz). ¹³C NMR (CDCl₃, 100 MHz): δ 165.38, 153.12, 141.46, 135.50, 131.65, 131.21, 130.48, 129.95, 119.72, 105.80, 73.51, 69.43, 31.88, 30.28, 29.61, 29.34, 26.04, 22.64, 15.60, 15.40, 15.21, 14.00. MALDI-MS: calcd for [M + H]⁺: 4905.84, found: 4905.80.

3. The AIEE behavior of complex 2 in CH₂Cl₂/n-hexane
Fig. S1 Fluorescence spectra (a) and fluorescence intensity at 522 nm (b) of complex 2 in the CH₂Cl₂/n-hexane mixtures with different n-hexane fractions.

4. Fluorescence spectra of complexes 2 and TPA in CH₂Cl₂ and n-
hexane

**Fig. S2** Fluorescence spectra of complexes 2 and TPA in CH$_2$Cl$_2$ (a) and $n$-hexane (b).

5. **SEM images of xerogel of TPA**
6. Concentration-dependent $^1$H NMR spectra of TPA

![Fig. S4](image_url)

Fig. S4 The $^1$H NMR spectra of TPA in CDCl$_3$ with different concentrations.

7. Temperature-depended emission spectra of TPA
Fig. S5 The emission spectra of TPA in ethyl acetate with different temperatures.

8. The emission spectra of TPA in different solvents

Fig. S6 The emission spectra of TPA in different solvents such as hexane, toluene, dichloromethane, tetrahydrofuran, ethyl acetate, acetonitrile, methanol, and dimethyl sulfoxide.

9. The normalized absorption spectra of TPA in CH$_2$Cl$_2$/n-hexane
Fig. S7 The normalized absorption spectra of TPA in the CH₂Cl₂/\textit{n}-hexane mixtures with different \textit{n}-hexane fractions.

10. The $^1$H, $^{31}$P, and $^{13}$C NMR spectra of TPA
Fig. S8 The $^1$H NMR (a), $^{31}$P NMR (b), and $^{13}$C NMR (c) spectra of TPA in CDCl$_3$.

11. The partial $^{31}$P NMR spectra of 2 and TPA

Fig. S9 The partial $^{31}$P NMR spectra of 2 and TPA in CDCl$_3$. 
12. MALDI-TOF-MS of TPA

![Fig. S10 MALDI-TOF-MS of TPA.](image)

13. IR spectra of the xerogel TPA

![Fig. S11 IR spectra of the xerogel TPA.](image)
14. X-ray diffraction diagrams of the xerogel TPA

Fig. S12 X-ray diffraction diagrams of the xerogel TPA. The inset for the diffraction in wide-angle region.