

Supporting Information

Pillar[5]arene-based supramolecular AIE hydrogel with white-light-emitting for ultrasensitive detection and effective separation of multianalytes

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The experimental details

1. Preparation of the hydrogel PDG

The mixture of **SPA** (8.7 mg, 9×10^{-3} mmol) and **DTB** (1.30 mg, 3×10^{-3} mmol) were added into a binary solution of DMSO and H₂O (v/v, 1:1, 200 μ L), the mixture solution was heated dissolve and completely dissolved, then cooled it to room temperature, obtaining stable bi-components hydrogel **PDG**.

2. Characterization of FT-IR spectra

Infrared spectra were recorded using a FT-IR spectrometer with KBr discs in the 4000-500 cm⁻¹ region. The solid powder of **SPA**, **DTB**, **PDG**, **PDG-Fe**, **PDG-Fe** + H₂PO₄⁻, **PDG-Cr**, **PDG-Cr** + CN⁻ and **PDG-Pb** were prepared by drying resulting gels on glass slices for a long time. All the samples were mixed well-distributedly with KBr to create compact pellets for the FT-IR detection.

3. Characterization of scanning electron microscopy (SEM)

The SEM images were performed on a JSM-6701F FE-SEM microscope. We prepared powder of **SPA**, xerogel **PDG** and xerogel **PDG-M** using pure organic solvents. A SEM sample was fabricated by spreading the solid powder on conductive plastic. Then gold powder was sprayed on the sample after the detection system was vacuumed. The SEM image of the solid powder was determined with an accelerating voltage of 8 kV.

4. The experimental details about the fluorescent titrations:

All the fluorescence spectroscopy was carried out in DMSO/H₂O (v/v, 1:1, 200 μ L) binary solution on a Shimadzu RF-5301 spectrometer. With different equivalents of cations were added into **PDG** (9.0×10^{-6} mol, 4.5×10^{-2} mol/L) while keeping the host concentration constant (4.54×10^{-2} mol/L) in all the experiments. The detection limits for ions were determined by fluorescent titrations and calculated on the basis of $3\sigma/s$ method.

For example, adding 0.01 equivalent Fe³⁺ aqueous solution to **PDG** (9.0×10^{-6} mol, 4.5×10^{-2} mol/L), and make Fe³⁺ diffused uniformly by heating dissolution. The stable metallogel was formed after cooled for one minute, and fluorescence intensity of **PDG-Fe** was measured on spectrometer. Increasing the amount of Fe³⁺ until the fluorescence of **PDG** was completely quenched, and reached the fluorescence titration endpoint. As a result, upon the addition of Cr³⁺ (13.5×10^{-6} mol, 67.5 mmol/L), Pb²⁺ (3.6×10^{-6} mol, 18.0 mmol/L) and Fe³⁺ (4.5×10^{-6} mol, 22.5 mmol/L) into **PDG** respectively, the fluorescence emission of **PDG** could be quenched. Meanwhile, the obtained coordinated metallogel was named as **PDG-Cr** and **PDG-Fe**. Next, we study the successive fluorescence response properties of **PDG-Cr** and

PDG-Fe to CN^- and H_2PO_4^- . CN^- and H_2PO_4^- were gradually added to **PDG-Cr** and **PDG-Fe**, respectively. As a result, CN^- (16.2×10^{-6} mol, 81.0 mmol/L) and H_2PO_4^- (15.3×10^{-6} mol, 76.5 mmol/L) could induce the **PDG-Cr** and **PDG-Fe** to emit white fluorescence again.

5. The film preparation experiments (such as PDG and PDG-Cr):

The **PDG**-based film was prepared by pouring heated DMSO- H_2O solution of **PDG** onto a clean glass surface and then drying in air. As shown in Fig. 7, the **PDG** film showed white fluorescence emission under UV (365 nm) light. When Cr^{3+} aqueous solution was added to **PDG**, the fluorescence of **PDG** disappeared instantly. In addition, when aqueous solution of CN^- was added, the fluorescence of **PDG-Cr** could be effectively restored.

6. Calculation formula of association constants (K_a)

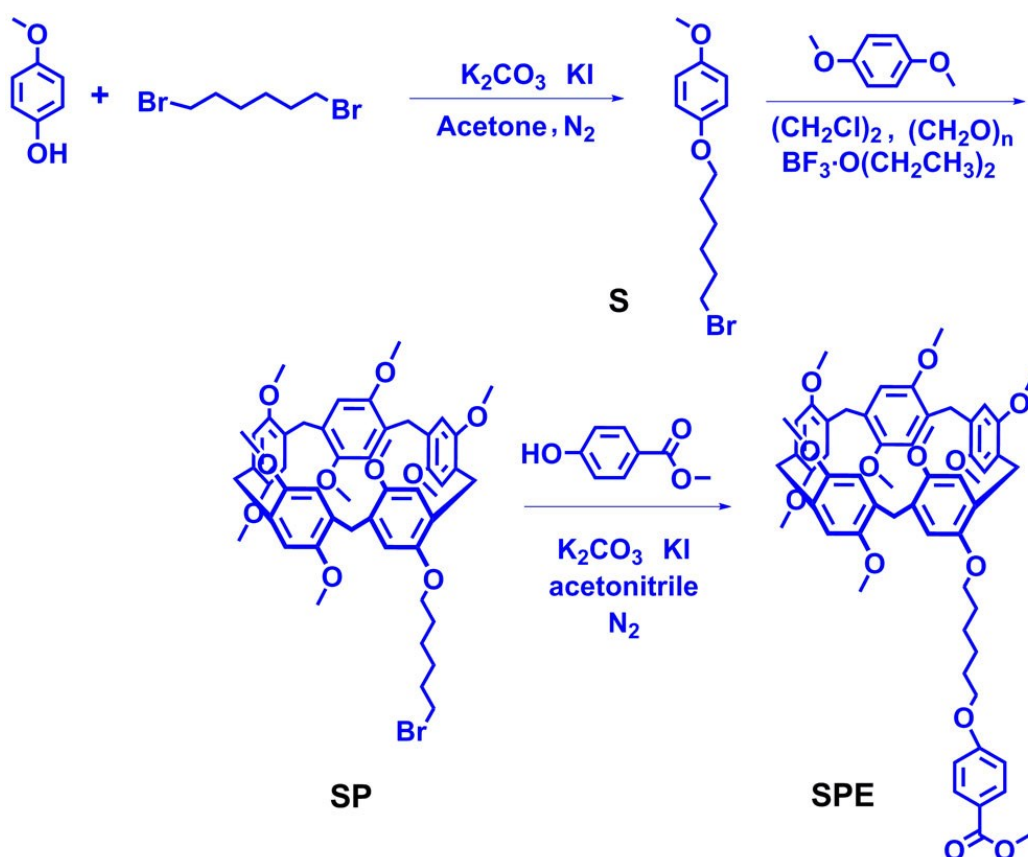
$$\log \frac{I - I_{\min}}{I_{\max} - I} = \log K_a + n \log [D]$$

$$K_a = 1.16 \times 10^4$$

I is the observed fluorescence intensity of **SPA** at the fixed concentrations of **DTB**. $I_{\max}$ and $I_{\min}$ are the corresponding maximum and minimum, respectively. $[D]$ is the corresponding concentration of **DTB**.

Materials and methods

Fresh doubly distilled water was used throughout the experiment. All chemicals were purchased from commercial suppliers and used without further purification unless otherwise noted. All anions and cations were purchased from Alfa Aesar and used as received. ^1H NMR (600 MHz or 400 MHz) and ^{13}C NMR spectra (151 MHz) were carried out with a Mercury-600 BB spectrometer. High-resolution mass spectra were performed on Bruker Esquire 3000 plus mass spectrometer equipped with ESI interface and ion trap analyzer. The infrared spectra were performed on a Digilab FTS-3000 Fourier transform-infrared spectrophotometer. Melting points were measured on an X-4 digital melting-point apparatus (uncorrected). Fluorescence spectra were recorded on a Shimadzu RF-5301PC spectrofluorophotometer. The morphologies and sizes of the xerogels were characterized using field emission scanning electron microscopy (FE-SEM, JSM-6701F) at an accelerating voltage of 8 kV.



Scheme S1 Synthesis of **S**, **SP** and **SPE**.

1. Synthesis of compound **S**

1-((6-bromohexyl)oxy)-4-methoxybenzene (**S**): 4-methoxyphenol (2.48 g, 20 mmol), K_2CO_3 (8.28 g, 60 mmol), KI (9.96 g, 60 mmol), 1, 6-dibromohexane (14.52 g, 60 mmol) and acetone (400 mL) were added into a 500 mL round-bottom flask. The mixture was heated reflux at 60 °C for 3 days. After the reaction completed, the solid was filtered off and the solvent was removed. Column chromatography (petroleum ether : ethyl acetate = 20 : 1) afforded a white solid **S** (4.83 g, 85%), m. p.: 85-87 °C. 1H NMR (400 MHz, $CDCl_3$) (Fig. S1) δ (ppm): 6.82 (s, 4H, ArH), 3.92-3.89 (t, J = 6.0 Hz, 2H, CH_2), 3.76 (s, 3H, CH_3), 3.43-3.40 (t, J = 6.0 Hz, 2H, CH_2), 1.90-1.86 (m, 2H, CH_2), 1.79-1.75 (m, 2H, CH_2), 1.52-1.47 (m, 4H, CH_2). ^{13}C NMR (151 MHz, $CDCl_3$) (Fig. S2) δ (ppm): 153.70, 115.41, 68.36, 55.72, 33.78, 32.67, 29.18, 27.92, 25.28.

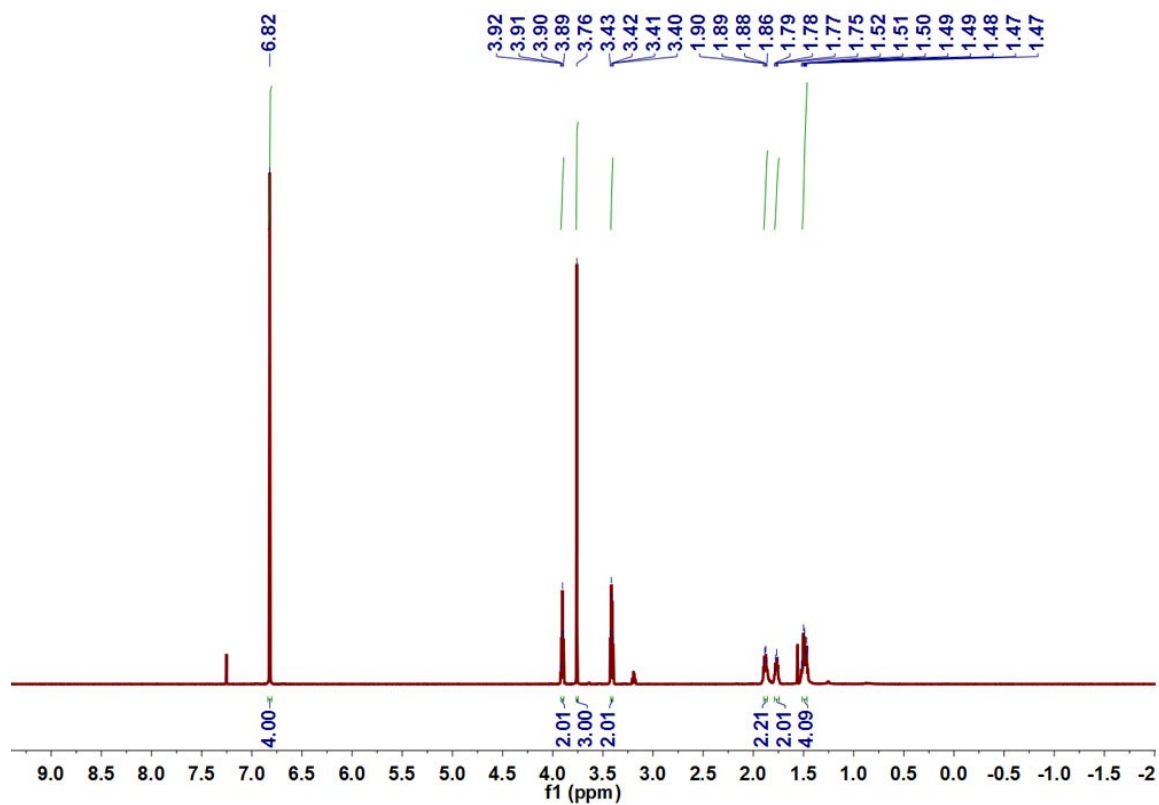


Fig. S1 ¹H NMR spectrum of S in CDCl₃.

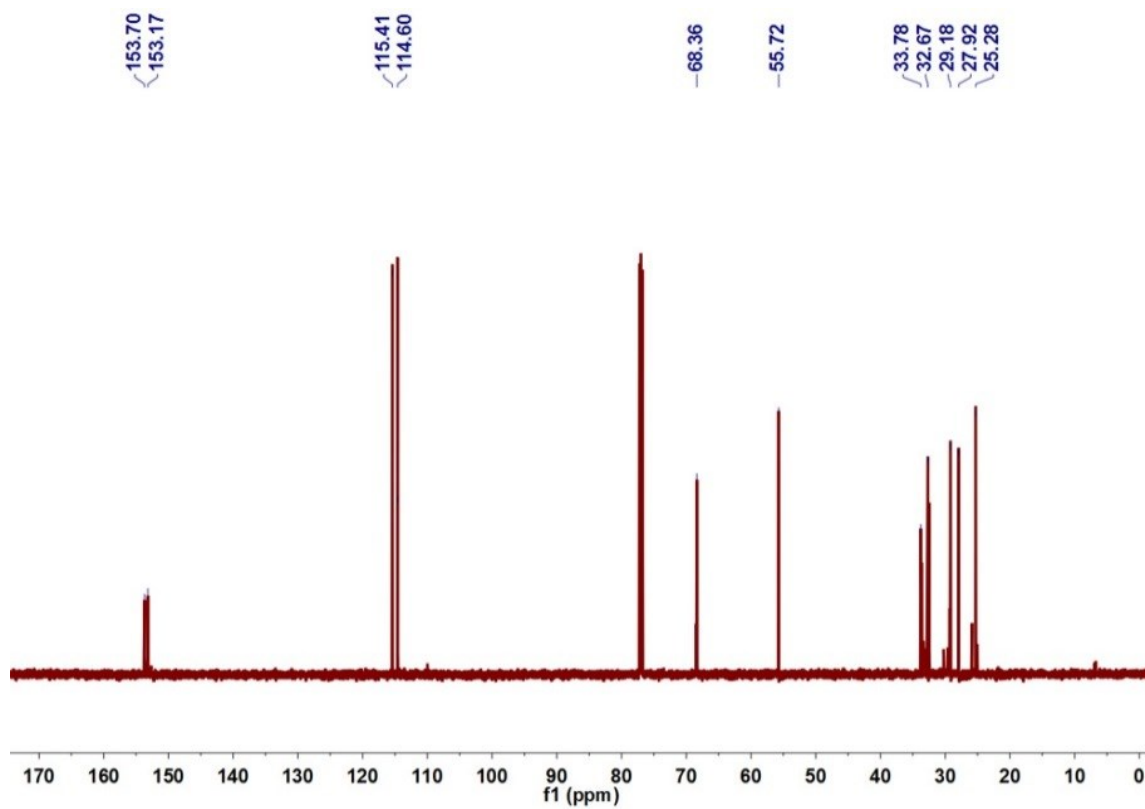


Fig. S2 ¹³C NMR spectrum of S in CDCl₃.

2. Synthesis of compound SP

Compound **S** (1.44 g, 5 mmol), 1, 4-dimethoxybenzene (3.25 g, 15 mmol) and paraformaldehyde (0.45 g, 15 mmol) were added to a solution of 1, 2-dichloroethane (250 mL). First, the mixture was stirred at room temperature for 30 min. Then, boron trifluoride diethyl etherate (5 mL, 47.6 mmol) was added to the solution, and stirred the mixture at 30 °C for 30 min. After the reaction completed, the reaction mixture **SP** was dissolved in CHCl₃ (300 mL) and washed thrice with H₂O (600 mL). The organic layer was dried over anhydrous Na₂SO₄ and evaporated to afford the crude product, which was isolated by column chromatography using petroleum ether/dichloromethane/ethyl acetate (v/v/v = 100 : 25 : 1) to give **SP** (2.83g, 63.12%) as a white solid, m. p.: 192-194 °C. ¹H NMR (Fig. S3) (400 MHz, CDCl₃) δ (ppm): 6.87-6.76 (m, 10H, ArH), 3.79-3.73 (m, 37H, 5ArCH₂, 9OCH₃), 3.72-3.63 (m, 4H, CH₂), 1.47-1.37 (m, 2H, CH₂), 1.36-1.35 (m, 2H, CH₂), 1.20-1.17 (m, 2H, CH₂), 0.81-0.79 (m, 2H, CH₂).

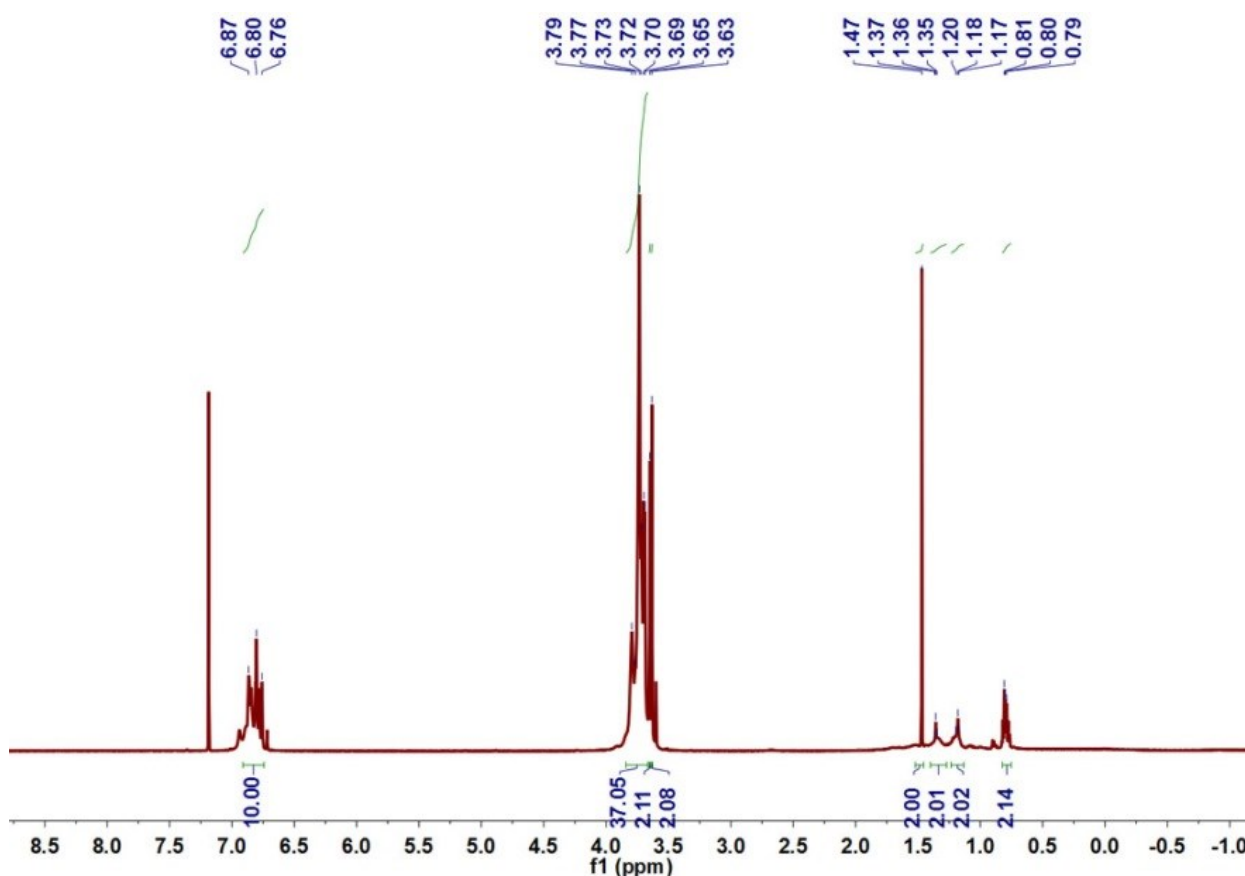


Fig. S3 ¹H NMR spectrum of **SP** in CDCl₃.

3. Synthesis of compound SPE

SP (0.449 g, 0.5 mmol), KI (0.83 g, 5 mmol) and acetonitrile (150 mL) were added into a 250 mL round-bottom flask. The mixture was stirred at 30 °C for 30 min. Then, K₂CO₃ (0.69 g, 5 mmol) and Methylparaben (0.152 g, 1 mmol) were added to the round-bottom flask. The mixture was heated at reflux under nitrogen protection at 85 °C for 12 hours. After the reaction completed, the solvent was dried and evaporated to afford the crude product, which was isolated by column chromatography using petroleum ether/1, 2-dichloroethane/ethyl acetate (v/v/v = 100 : 25 : 1) to give **SPE** (0.372 g, 83%) as a white solid, m. p.: 97-98 °C. ¹H NMR (Fig. S4) (600 MHz, CDCl₃) δ (ppm): 7.99-7.97 (d, *J* = 12.0 Hz, 2H, ArH), 6.86-6.85 (d, *J* = 12Hz, 2H, ArH), 6.76 (s, 10H, ArH), 3.98 (s, 4H, CH₂), 3.77 (s, 10H, ArH), 3.65 (s, 27H, 9OCH₃), 2.16 (s, 3H, CH₃), 1.54 (s, 2H, CH₂), 1.24 (s, 2H, CH₂). ¹³C NMR (Fig. S5) (151 MHz, CDCl₃) δ (ppm): 166.88, 162.87, 150.22, 149.53, 131.54, 128.43, 122.37, 114.03, 112.84, 68.12, 55.36, 51.74, 29.84, 29.04, 26.08. ESI-MS *m/z*: [**SPE**] calcd for C₅₈H₆₆O₁₃, 970.4503; found 970.4500 (Fig. S6).

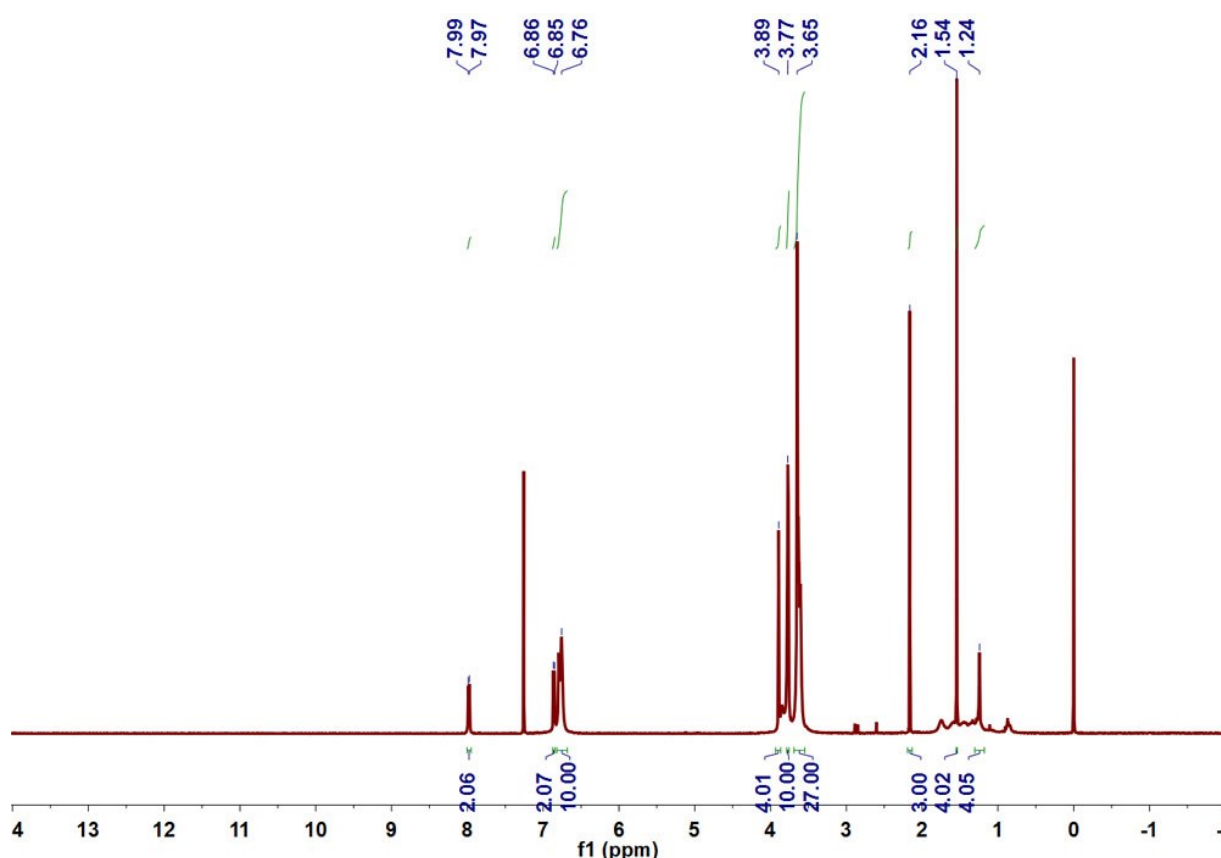


Fig. S4 ¹H NMR spectrum of **SPE** in CDCl₃.

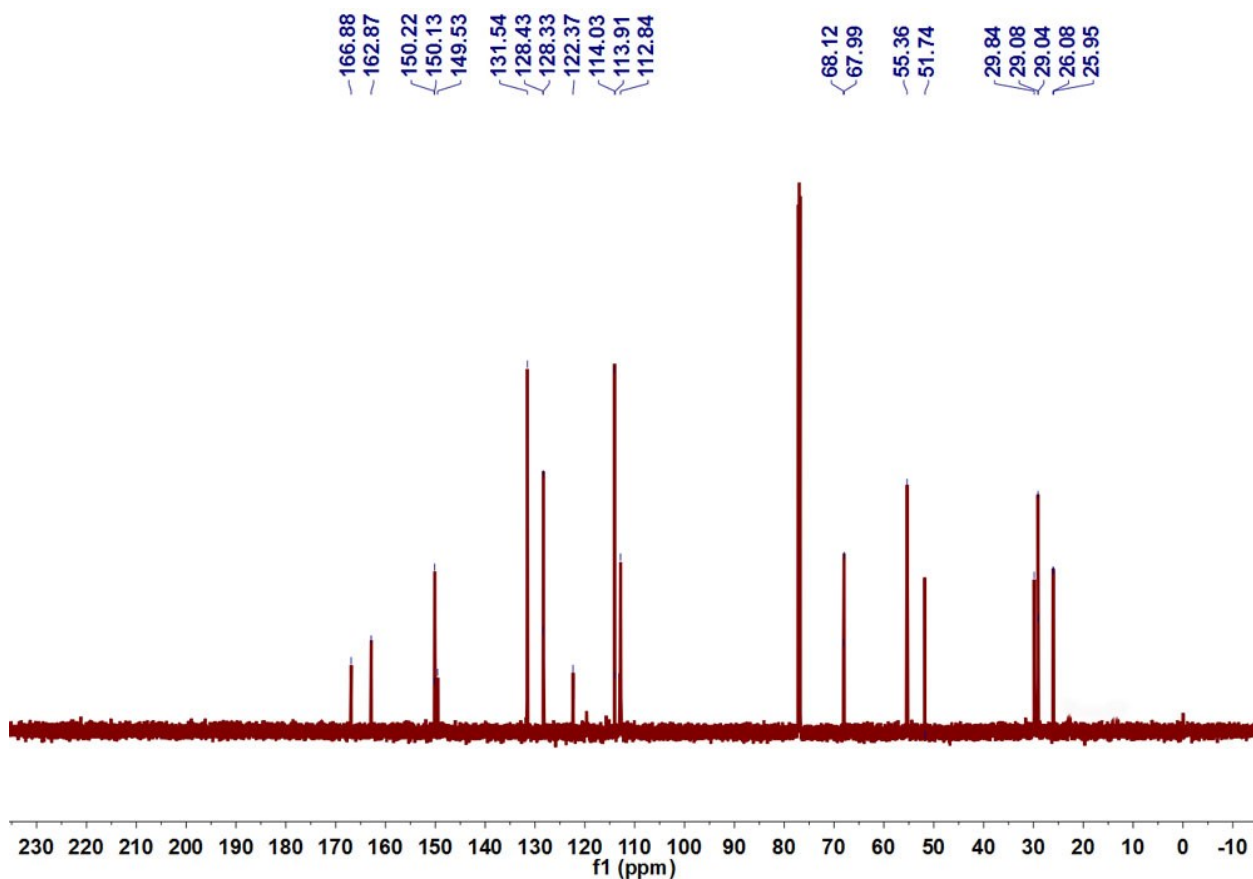


Fig. S5 ^{13}C NMR spectrum of SPE in CDCl_3 .

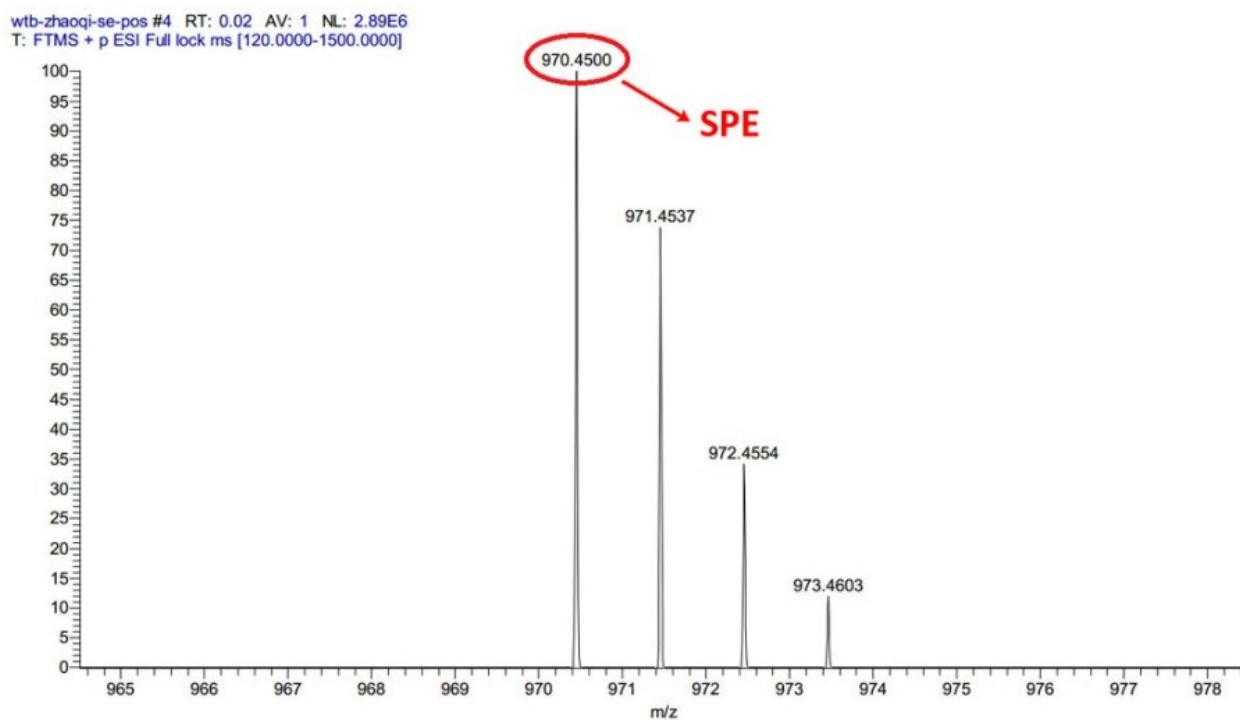
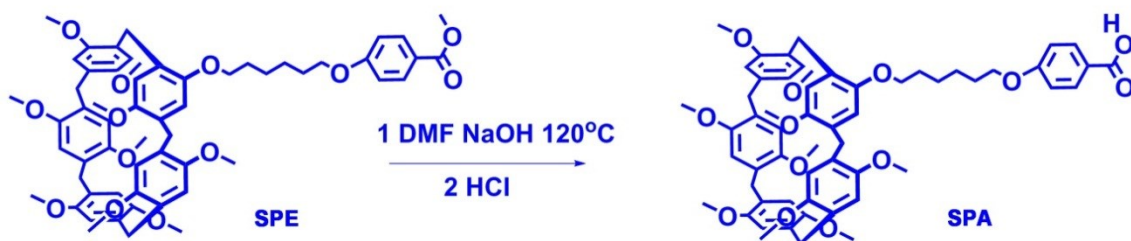


Fig. S6 HRMS spectrum of SPE.



Scheme S2 Synthesis of SPA.

4. Synthesis of compound SPA

SPE (0.097 g, 0.1 mmol) was added to a solution of NaOH (1 mol/L) aqueous solution in DMF (30 mL). The mixture was heated in a round-bottomed flask at 120 °C for 12 h. Then filtered under reduced pressure and the residue were recrystallized in ethanol and water to give a white solid **SPA** (0.0874, 90%), m. p.: 111-112 °C. ^1H NMR (Fig. S7) (600 MHz, $\text{DMSO}-d_6$) δ (ppm): 12.55 (s, 1H, OH), 7.86-7.85 (d, $J = 6.0$ Hz, 2H, ArH), 6.98-6.96 (d, $J = 12$ Hz, 2H, ArH), 6.74-6.78 (t, $J = 6.0$ Hz, 10H, ArH), 4.00-3.98 (t, $J = 6$ Hz, 2H, CH_2), 3.81-3.79 (t, $J = 6$ Hz, 2H, CH_2), 3.63-3.62 (m, 37H, 5Ar CH_2 , 9O CH_3), 1.73-1.68 (m, 4H, CH_2), 1.50-1.42 (m, 4H, CH_2). ^{13}C NMR (Fig. S8) (151 MHz, CDCl_3) δ (ppm): 162.69, 150.14, 132.16, 131.53, 128.42, 128.31, 119.66, 114.10, 112.92, 109.99, 68.15, 55.46, 55.25, 36.44, 31.43, 29.15, 26.04. ESI-MS m/z : [**SPA**] calcd for $\text{C}_{57}\text{H}_{64}\text{O}_{13}$, 956.4347; found 956.4325 (Fig. S9).

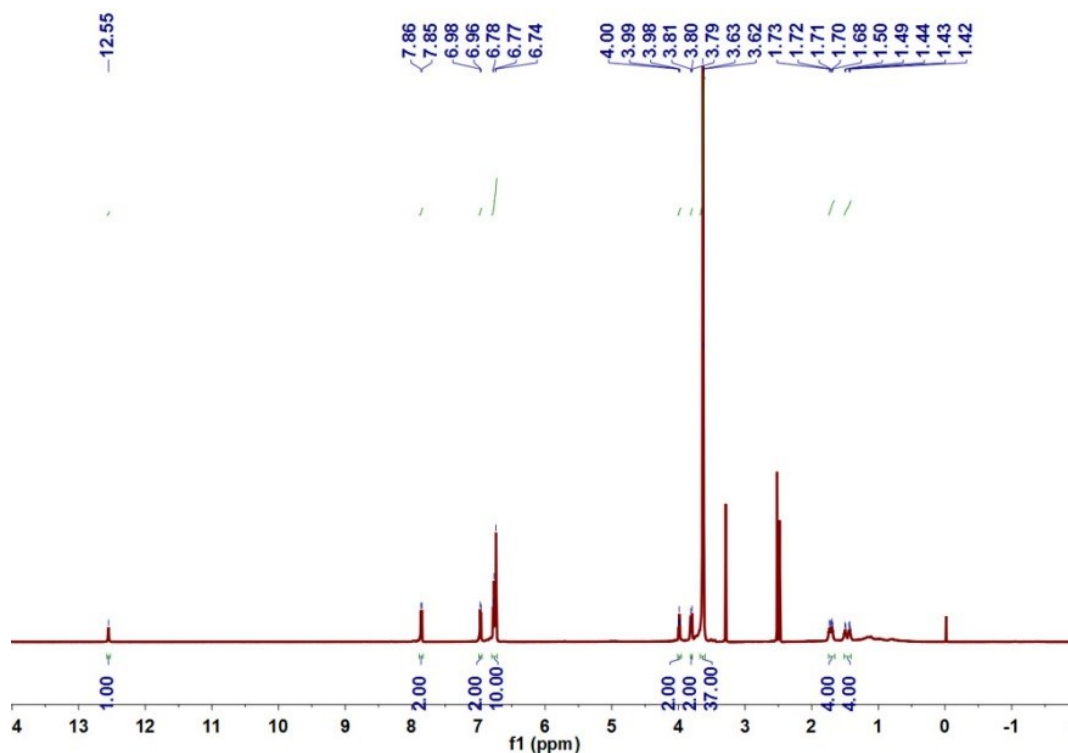


Fig. S7 ^1H NMR spectrum of SPA in $\text{DMSO}-d_6$.

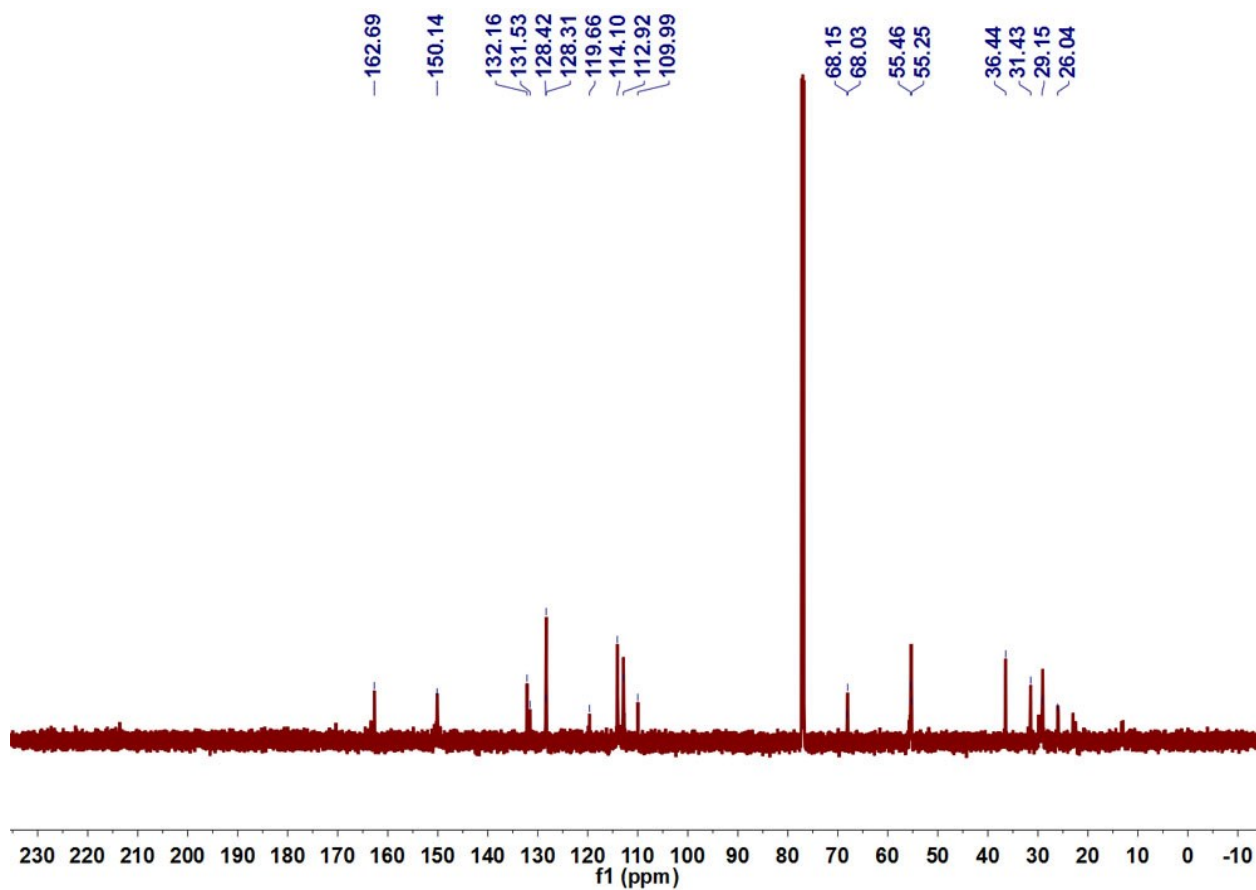


Fig. S8 ^{13}C NMR spectrum of SPA in CDCl_3 .

wtb-zhaoqi-sa-pos #112 RT: 0.50 AV: 1 NL: 4.03E5
T: FTMS + p ESI SIM ms [949.5000-964.5000]

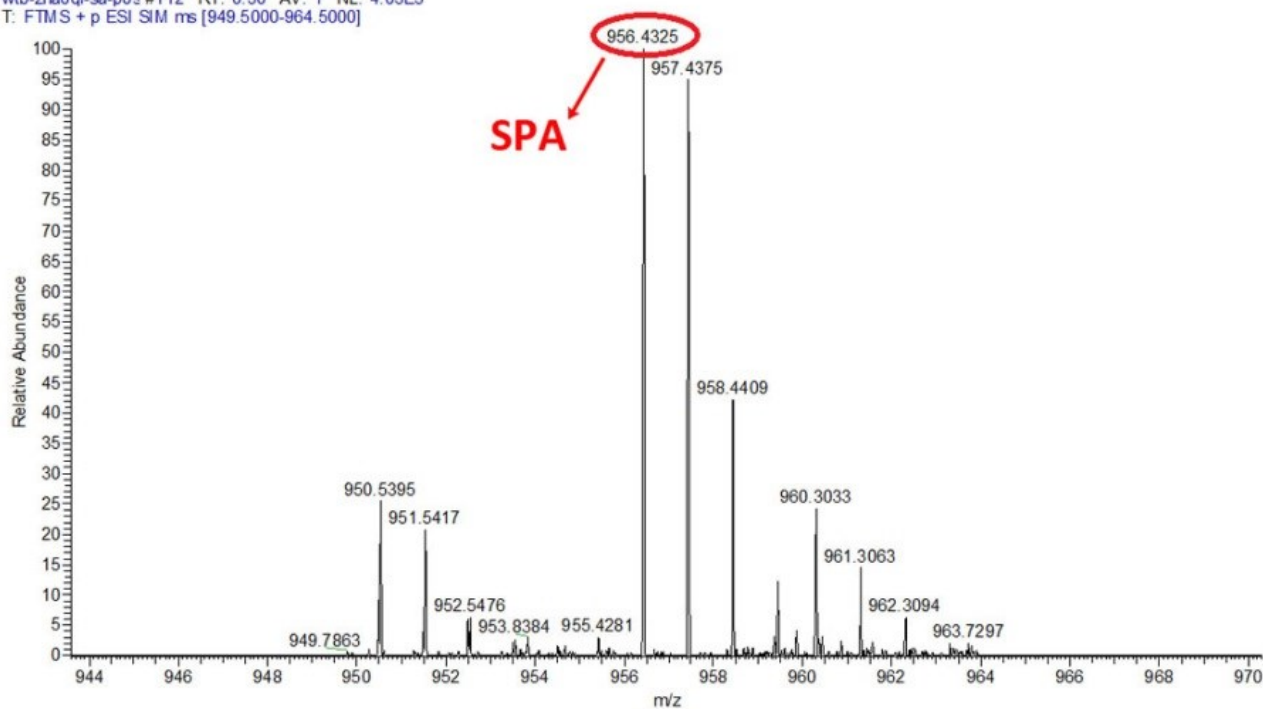
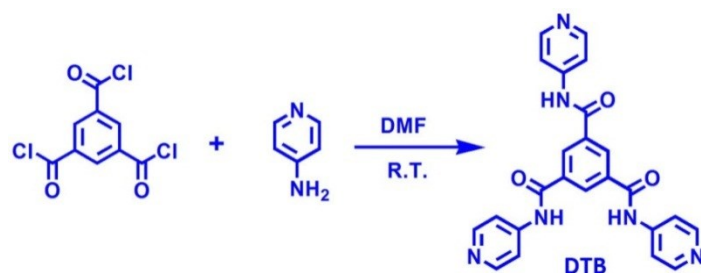


Fig. S9 HRMS spectrum of SPA.



Scheme S3 Synthesis of **DTB**.

5. Synthesis of compound **DTB**

To a solution of mixture of trimesoyl chloride (0.025 g, 0.1 mmol) and triethylamine (1-2 d) was slowly dropwise added into the solution of 4-aminopyridine (0.031 g, 0.33 mmol) in DMF (10 mL), the mixture was stirred at room temperature for 20 h, appearing a pale solid **DTB** (0.038 g, 87%). The solid was vacuumed suction filtration and washed with cold ethyl alcohol (10 mL), then drying product solid in a vacuum oven at 40 °C for 24 h, m. p.: 167-169 °C. ^1H NMR (Fig. S10) (400 MHz, $\text{DMSO}-d_6$) δ (ppm): 11.08-11.03 (t, $J = 18.0$ Hz, 3 H, NH), 8.79 (s, 1 H, ArH), 8.72 (s, 1 H, ArH), 8.63 (s, 1 H, ArH), 8.54-8.53 (d, 6 H, $J = 6.0$ Hz, ArH), 7.88-7.86 (m, 6 H, ArH). ^{13}C NMR (Fig. S11) (151 MHz, $\text{DMSO}-d_6$) δ (ppm): 166.05, 146.13, 135.38, 132.15, 114.60, 109.24. ESI-MS m/z : [**DTB**] calcd for $\text{C}_{24}\text{H}_{18}\text{N}_6\text{O}_3$, 439.1474, found 439.1512 (Fig. S12).

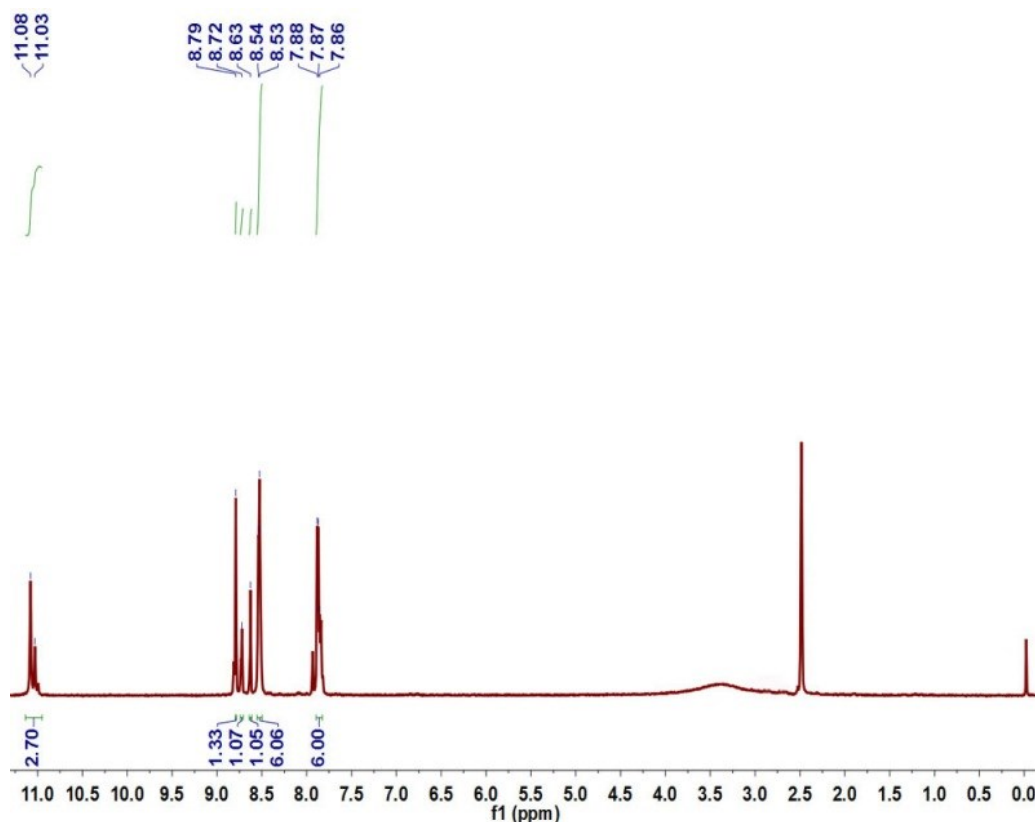


Fig. S10 ^1H NMR spectrum of **DTB** in $\text{DMSO}-d_6$.

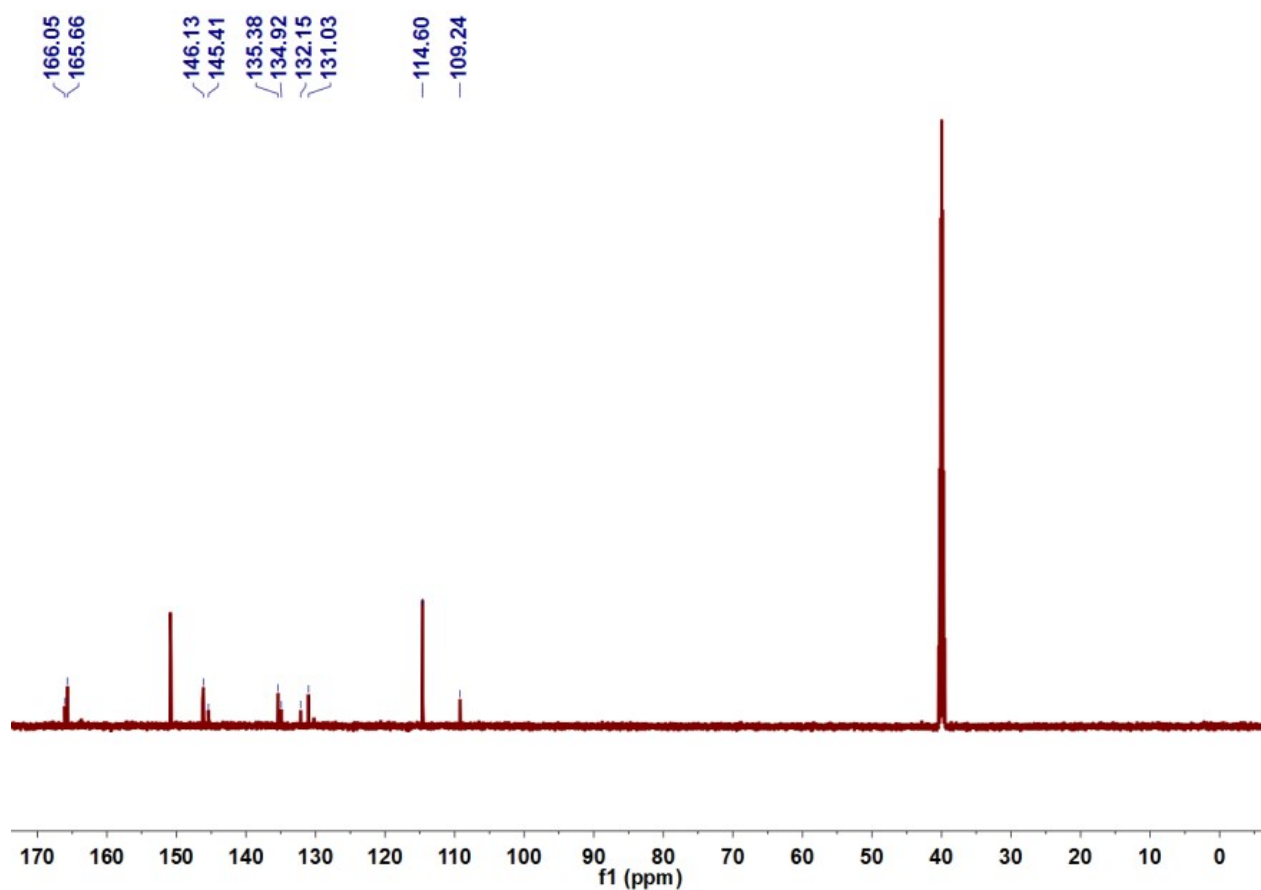


Fig. S11 ^{13}C NMR spectrum of **DTB** in $\text{DMSO-}d_6$.

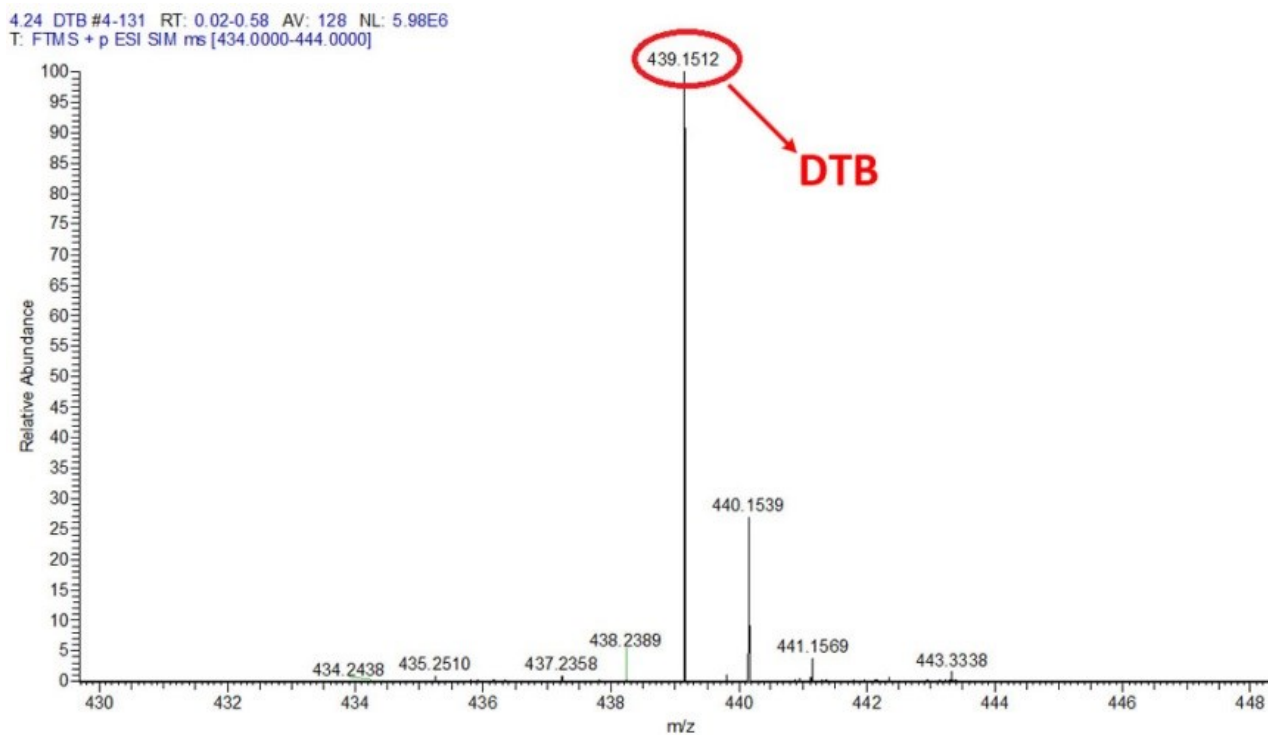


Fig. S12 HRMS spectrum of **DTB**.

Table S1 The sources of various cations.

Cations	Sources	Names
Hg ²⁺	Hg(ClO ₄) ₂ ·3H ₂ O	Mercury perchlorate trihydrate
Ca ²⁺	Ca(ClO ₄) ₂ ·6H ₂ O	Calcium perchlorate hexahydrate
Mg ²⁺	Mg(ClO ₄) ₂ ·6H ₂ O	Magnesium perchlorate hexahydrate
Ni ²⁺	Ni(ClO ₄) ₂ ·6H ₂ O	Nickel perchlorate hexahydrate
Cu ²⁺	Cu(ClO ₄) ₂ ·6H ₂ O	Copper perchlorate hexahydrate
Cr ³⁺	Cr(ClO ₄) ₃ ·6H ₂ O	Chromium perchlorate hexahydrate
Cd ²⁺	Cd(ClO ₄) ₂ ·6H ₂ O	Cadmium perchlorate hexahydrate
Pb ²⁺	Pb(ClO ₄) ₂ ·3H ₂ O	Lead perchlorate trihydrate
Ag ⁺	AgClO ₄	Silver perchlorate, anhydrous
Zn ²⁺	Zn(ClO ₄) ₂ ·6H ₂ O	Zinc perchlorate hexahydrate
Fe ³⁺	Fe(ClO ₄) ₃ ·6H ₂ O	Iron perchlorate hexahydrate
Ba ²⁺	Ba(ClO ₄) ₂	Barium perchlorate, anhydrous
Co ²⁺	Co(ClO ₄) ₂ ·6H ₂ O	Cobalt perchlorate hexahydrate
La ³⁺	LaCl ₃ ·7H ₂ O	Lanthanum chloride heptahydrate
Eu ³⁺	EuCl ₃ ·6H ₂ O	Europium chloride hexahydrate
Tb ³⁺	TbCl ₃ ·6H ₂ O	Terbium chloride hexahydrate

Table S2 The sources of various anions.

Anions	Sources	Names
AcO ⁻	(C ₄ H ₉) ₄ N(AcO)	Tetrabutylammonium acetate
HSO ₄ ⁻	(C ₄ H ₉) ₄ N(HSO ₄)	Tetrabutylammonium hydrogen sulfate
H ₂ PO ₄ ⁻	(C ₄ H ₉) ₄ N (H ₂ PO ₄)	Tetrabutylammonium dihydrogen phosphate
F ⁻	(C ₄ H ₉) ₄ NF	Tetrabutylammonium fluoride
Cl ⁻	(C ₄ H ₉) ₄ NCl	Tetrabutylammonium chloride
Br ⁻	(C ₄ H ₉) ₄ NBr	Tetrabutylammonium bromide
I ⁻	(C ₄ H ₉) ₄ NI	Tetrabutylammonium iodide
ClO ₄ ⁻	(C ₄ H ₉) ₄ N(ClO ₄)	Tetrabutylammonium perchlorate
SCN ⁻	NaSCN	Sodium sulfocyanate
CN ⁻	NaCN	Sodium cyanide
S ²⁻	Na ₂ S·9H ₂ O	Sodium sulfide nonahydrate
N ₃ ⁻	NaN ₃	Sodium azide

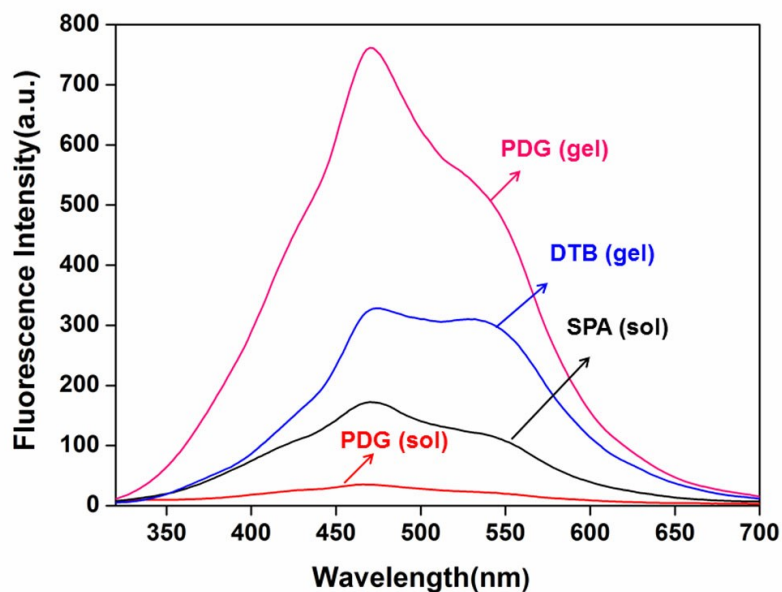


Fig. S13 Fluorescence spectra of the **SPA** (sol), **DTB** (gel), **PDG** (gel) and **PDG** (sol) in DMSO-H₂O binary solution ($\lambda_{\text{ex}} = 300$ nm).

Table S3 Gelation properties of supramolecular hydrogel **PDG**.

Entry	Solvent	State ^a	CGC ^b (%)	T _{gel} ^c (°C, wt%)
1	water	P	\	\
2	acetone	P	\	\
3	methanol	P	\	\
4	ethanol	P	\	\
5	isopropanol	P	\	\
7	acetonitrile	P	\	\
8	THF	P	\	\
9	DMF	S	\	\
10	DMSO	S	\	\
11	DMSO-H ₂ O	G	5	95(5)
11	CCl ₄	P	\	\
12	n-hexane	P	\	\
13	ethanediol	P	\	\
14	tert-butylalcohol	P	\	\
15	CH ₂ Cl ₂	S	\	\
16	CHCl ₃	P	\	\
17	CH ₂ ClCH ₂ Cl	P	\	\
18	petroleum ether	P	\	\
19	ethyl acetate	P	\	\
20	n-propanol	P	\	\
21	n-butyl alcohol	P	\	\
22	cyclohexanol	P	\	\

^aG, P and S denote gelation, precipitation and solution, respectively;

^bThe critical gelation concentration (wt%, 10 mg/mL = 1.0 %);

^cThe gelation temperature (°C).

Table S4 Optimum water contents of gelation conditions.

Entry	water solvent	State ^a	Tgel ^b (°C)
1	0%	S	\
2	10%	S	\
3	15%	S	\
4	20%	S	\
5	25%	S	\
6	30%	S	\
7	35%	S	\
8	40%	G	60
9	45%	G	77
11	50%	G	95
12	55%	G	82
13	60%	S	\

^aG, P and S denote gelation, precipitation and solution, respectively.

^bThe gelation temperature (°C).

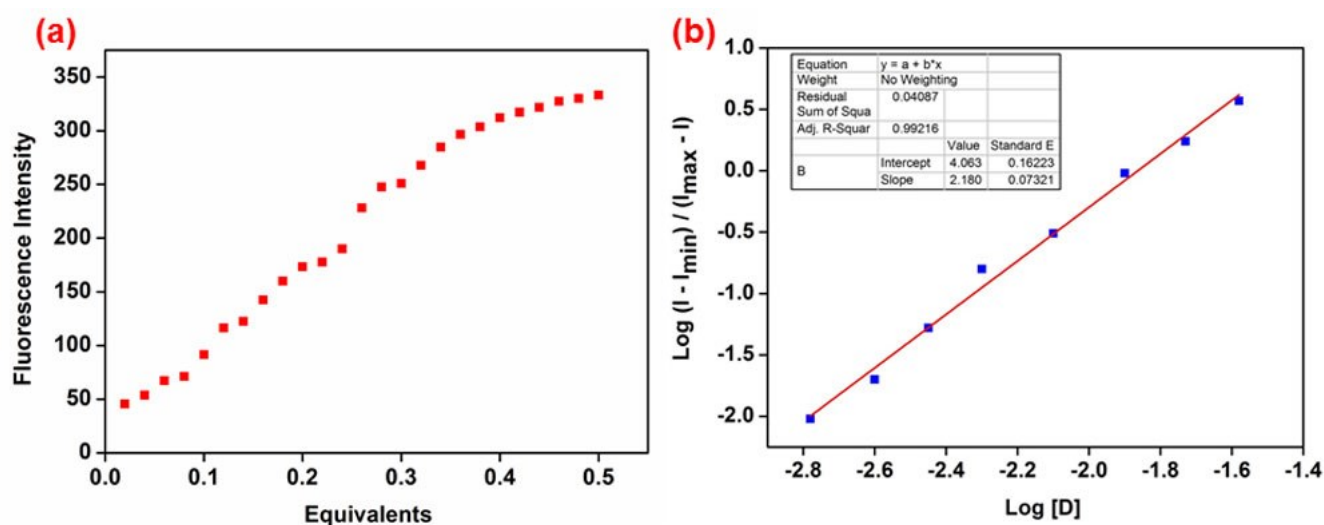


Fig. S14 (a) A plot of emission of SPA at 460 nm versus number of equivalents of DTB; (b) The binding constant and complex ratio of SPA and DTB with fluorescent titration.

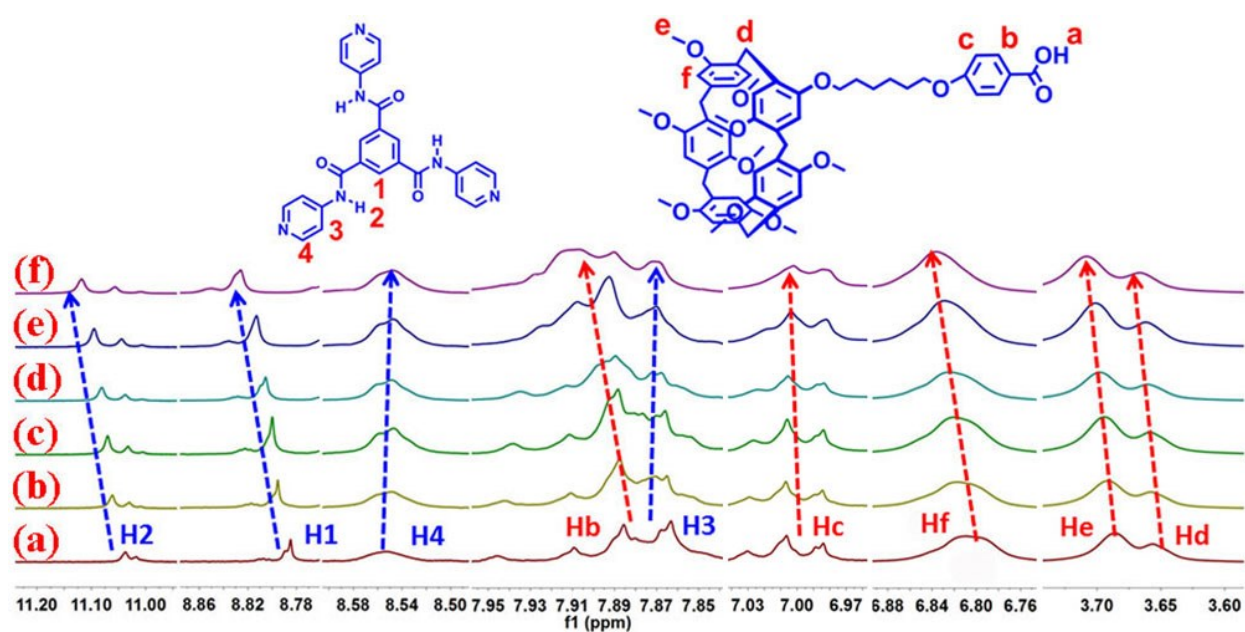


Fig. S15 The partial ^1H NMR spectra of **PDG** in $\text{DMSO}-d_6$ with different concentrations: (a) 5 mg mL^{-1} ; (b) 10 mg mL^{-1} ; (c) 15 mg mL^{-1} ; (d) 20 mg mL^{-1} ; (e) 30 mg mL^{-1} ; (f) 50 mg mL^{-1} .

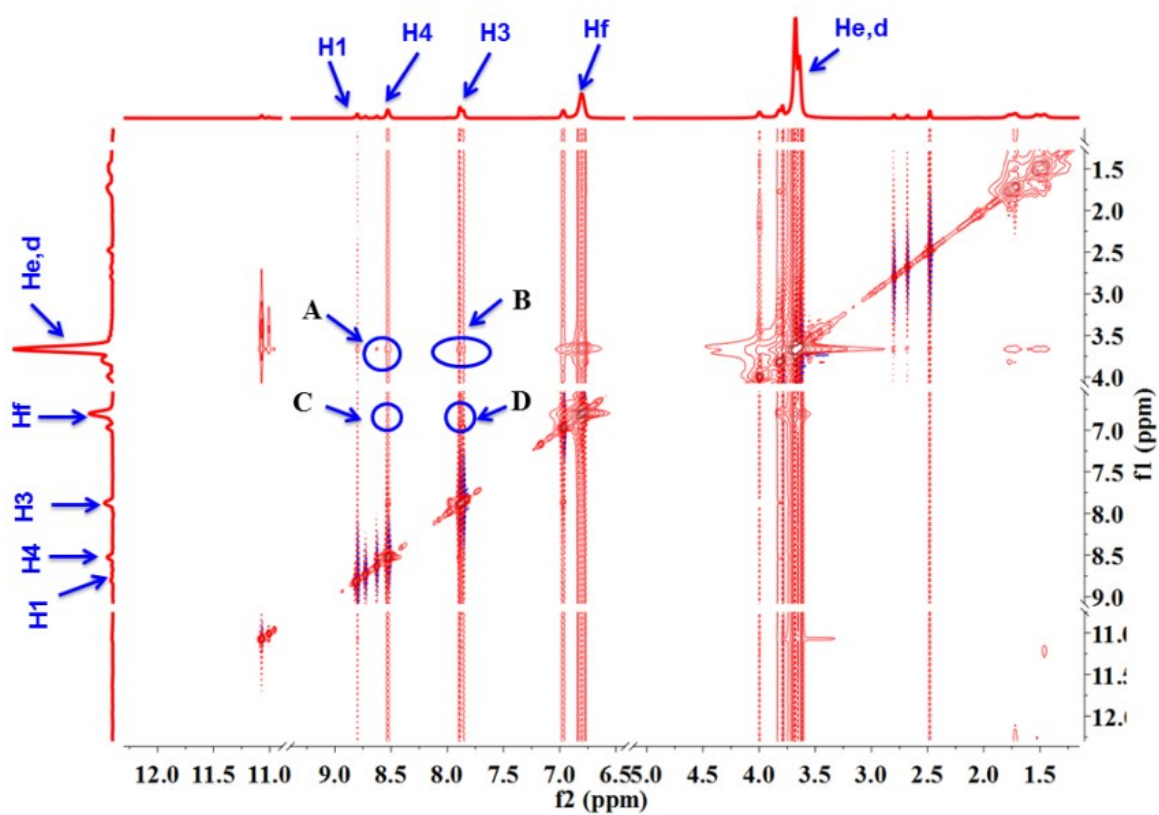


Fig. S16 2D NOESY NMR spectrum of **PDG** (70 mM) in $\text{DMSO}-d_6$ solution.

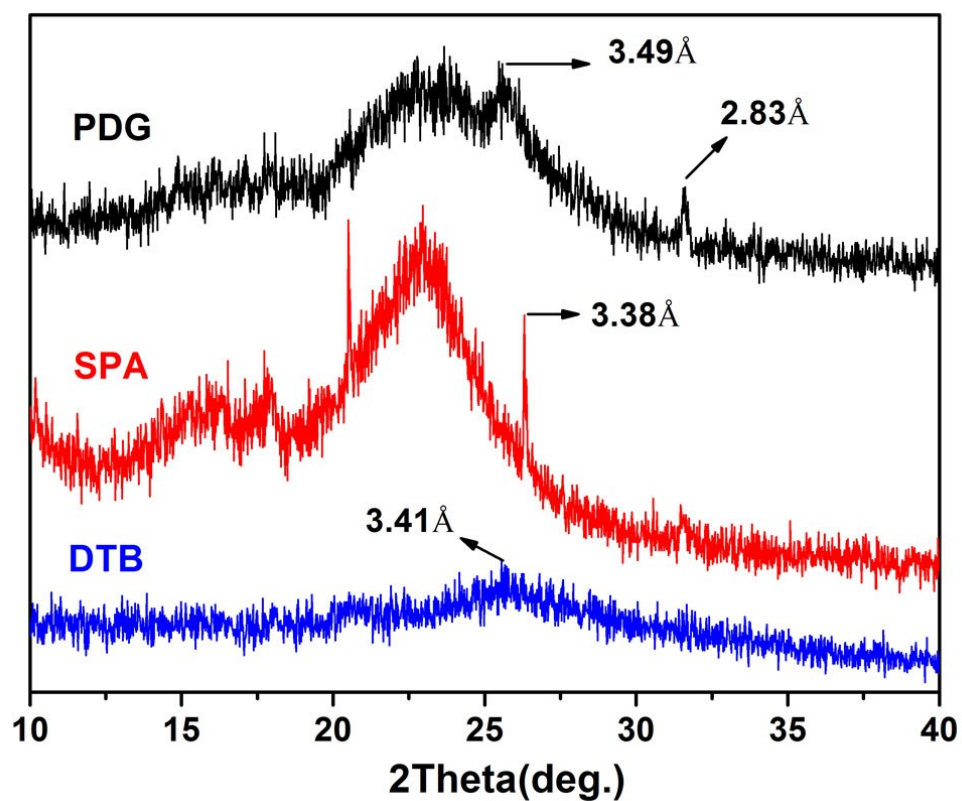


Fig. S17 XRD patterns of SPA, DTB and xerogel PDG.

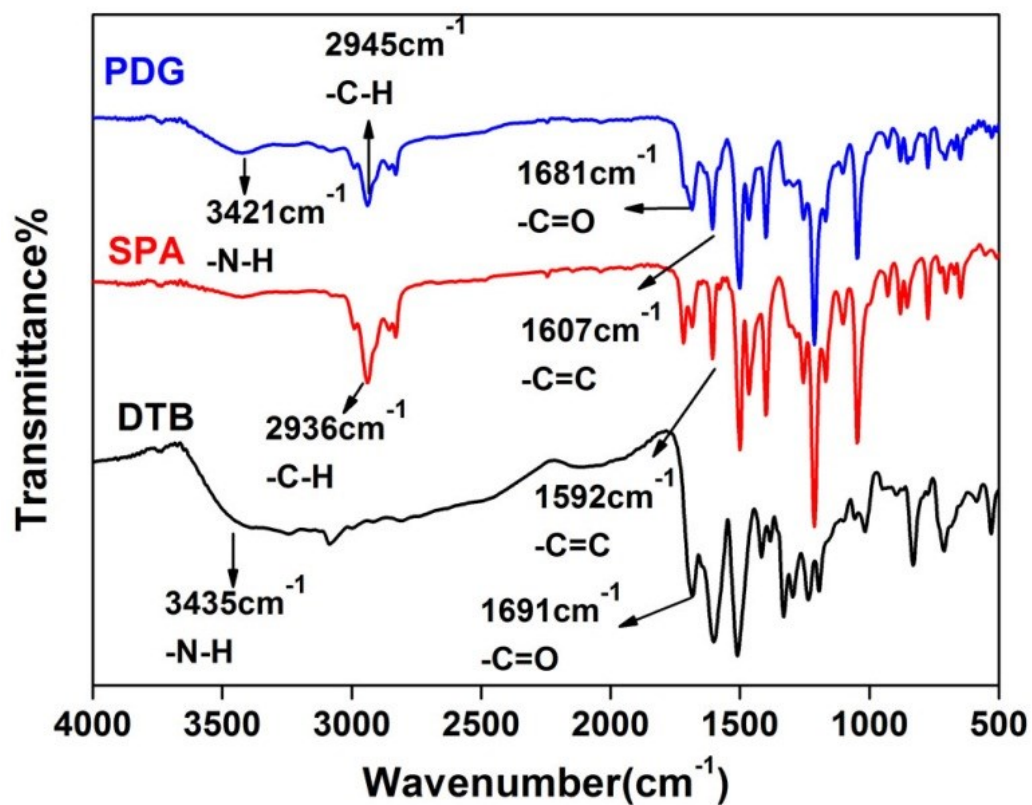


Fig. S18 FT-IR spectra of powder **SPA**, powder **DTB** and xerogel **PDG**.

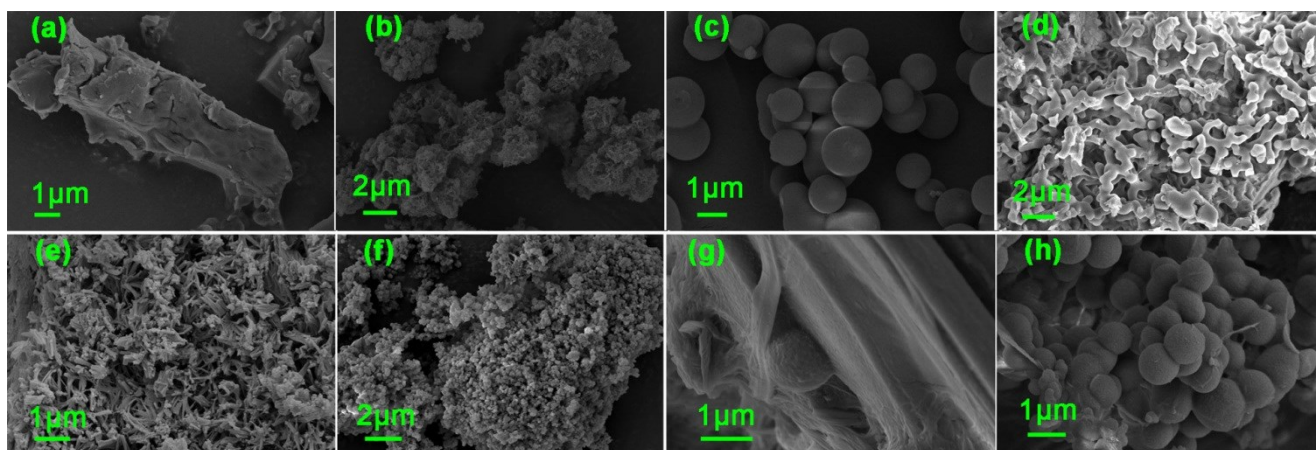


Fig. S19 SEM images of (a) **SPA**; (b) **DTB**; (c) **PDG**; (d) **PDG-Pb**; (e) **PDG-Cr**; (f) **PDG-Cr + CN⁻**; (g) **PDG-Fe**; (h) **PDG-Fe + H₂PO₄⁻**.

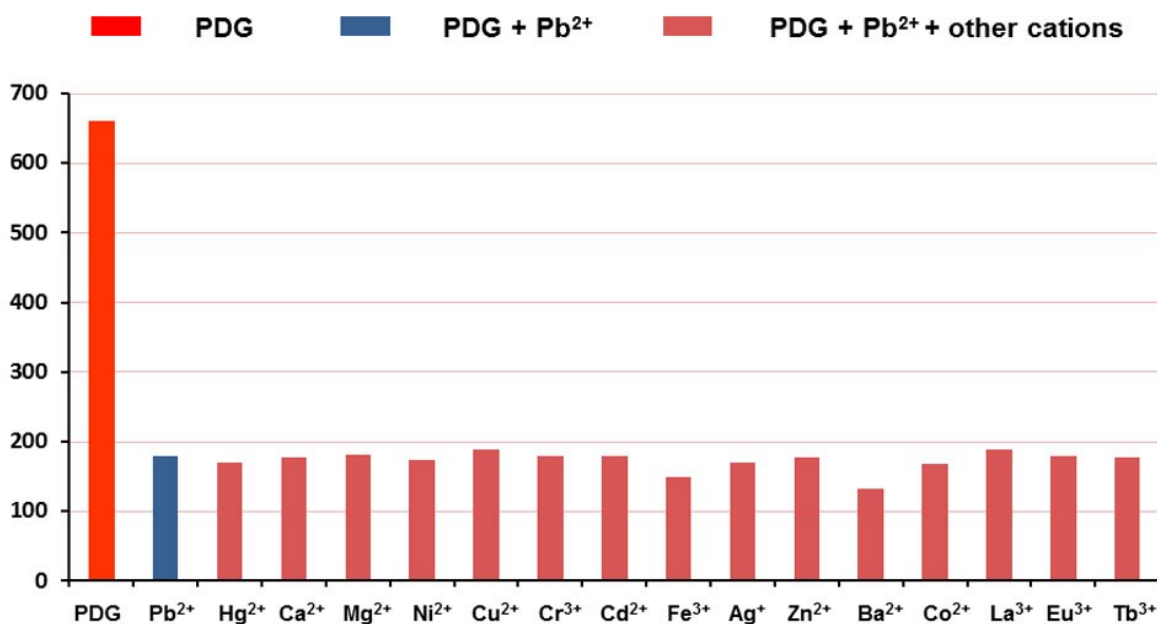


Fig. S20 Fluorescence responses of **PDG** to **Pb²⁺** in the presence of various cations aqueous solution (**Hg²⁺**, **Ca²⁺**, **Mg²⁺**, **Ni²⁺**, **Cu²⁺**, **Cr³⁺**, **Cd²⁺**, **Fe³⁺**, **Ag⁺**, **Zn²⁺**, **Ba²⁺**, **Co²⁺**, **La³⁺**, **Eu³⁺** and **Tb³⁺**) at 300 nm.

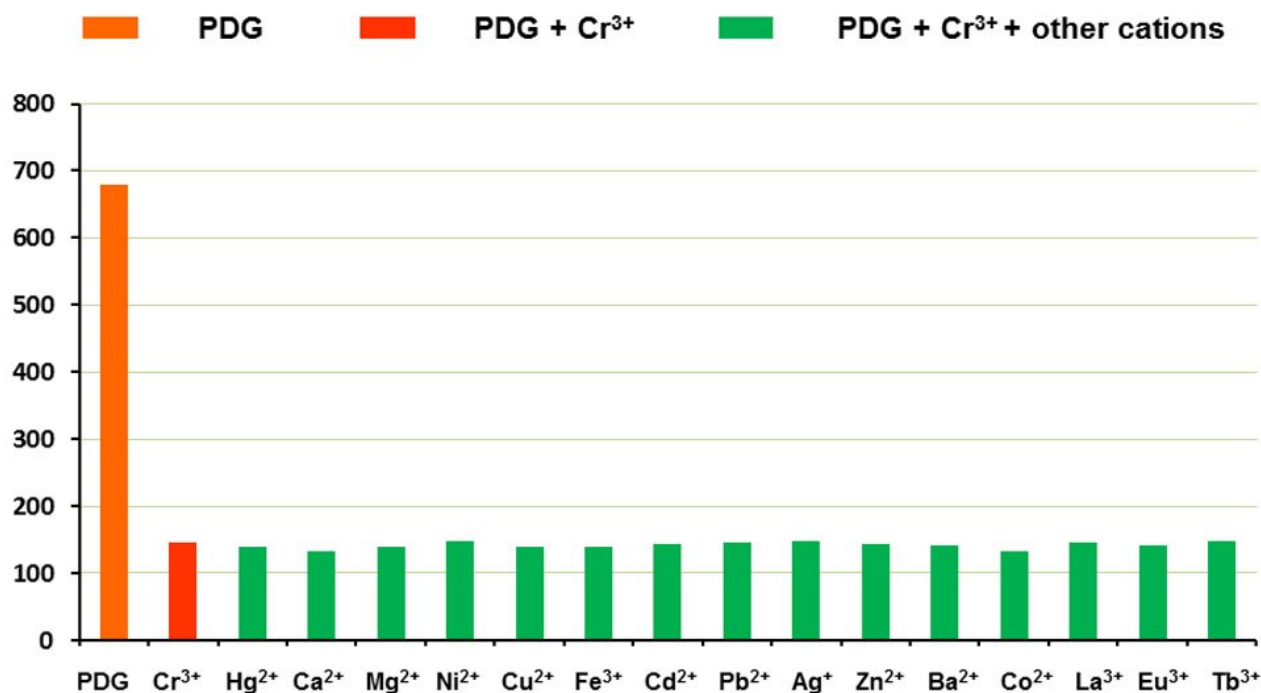


Fig. S21 Fluorescence responses of **PDG** to Cr³⁺ in the presence of various cations aqueous solution (Hg²⁺, Ca²⁺, Mg²⁺, Ni²⁺, Cu²⁺, Fe³⁺, Cd²⁺, Pb²⁺, Ag⁺, Zn²⁺, Ba²⁺, Co²⁺, La³⁺, Eu³⁺ and Tb³⁺) at 300 nm.

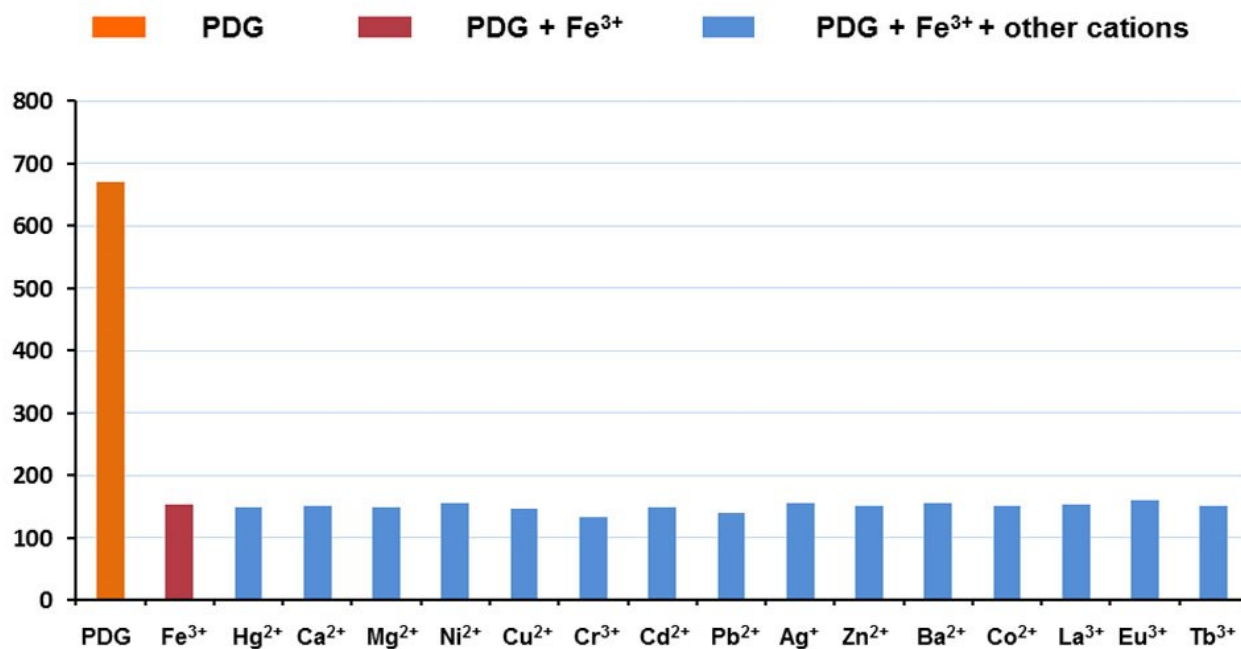


Fig. S22 Fluorescence responses of **PDG** to Fe³⁺ in the presence of various cations aqueous solution (Hg²⁺, Ca²⁺, Mg²⁺, Ni²⁺, Cu²⁺, Cr³⁺, Cd²⁺, Pb²⁺, Ag⁺, Zn²⁺, Ba²⁺, Co²⁺, La³⁺, Eu³⁺ and Tb³⁺) at 300 nm.

Table S5 A part of the literatures about the LODs (mol/L) of ions (Cr^{3+} , Pb^{2+} , Fe^{3+} and CN^-) are provided in the followed table, calculation formula and related date.

Journal	for Pb^{2+}	for Cr^{3+}	for Fe^{3+}	For CN^-
Chem. Res. Chinese Univ., 2010, 26, 194-197.	1.00×10^{-7}	-	-	-
J Hazard Mater, 2010, 177, 983-989.	1.00×10^{-6}	-	-	-
Optik, 2018, 158, 813-825.	-	2.37×10^{-8}	-	-
Cryst. Growth Des., 2017, 17, 4326-4335.	-	1.50×10^{-6}	-	-
Optik, 2018, 158, 813-825.	-	-	1.12×10^{-5}	-
Inorg. Chem., 2016, 55, 12660-12668.	-	-	1.00×10^{-7}	-
ACS Sens., 2016, 1, 1265-1271.	-	-	-	5.57×10^{-7}
Sens. Actuators B: Chem., 2017, 253, 942-948.	-	-	-	4.97×10^{-7}
This work	6.74×10^{-9}	7.18×10^{-9}	8.25×10^{-9}	3.86×10^{-8}

The result of the analysis as follows: For Cr^{3+}

Linear Equation: $Y = 320.23 \times X + 252.95$ $R^2 = 0.99136$

$S = 4.59 \times 10^8$

$$\delta = \sqrt{\frac{\sum_{i=1}^N (F_i - \bar{F})^2}{N - 1}} = 1.10 \quad (N = 20) \quad K = 3$$

$\text{LOD} = K \times \delta / S = 7.18 \times 10^{-9} \text{ M}$

Table S6 Comparison of the adsorption rates of the fluorescence sensing systems for Pb^{2+} , Cr^{3+} and Fe^{3+} .

Journal	for Pb^{2+}	for Cr^{3+}	for Fe^{3+}
Etc. Anal. Chem., 2013, 85, 7441-7446.	-	-	98.2%
Chemical Engineering Journal, 2018, 343, Pages 492-499.	-	-	90%
Ind. Eng. Chem. Res., 2013, 52, 6502-6512.	-	95.61%	-
RSC Adv., 2016, 6, 19780–19791.	93.85%	68.52%	-
Journal of Colloid and Interface Science, 2015, 442, 120-132.	70%	-	-
ACS Appl. Mater. Interfaces, 2019, 35, 32186-32191.	90%	-	-
Journal of the Taiwan Institute of Chemical Engineers, 2018, 93, 379-387.	96.58%	-	-
Journal of Colloid and Interface Science, 2015, 445, 294-302.	93.4%	-	-
This work	96.73%	98.35%	98.95%

Calculation method of adsorption percentage:
 Adsorption percentage (%) = $\left(1 - \frac{C_R \times V_R}{C_I \times V_I}\right) \times 100\%$
 (state: C_R is the residual concentration of Pb^{2+} , Cr^{3+} , and Fe^{3+} , C_I is the initial concentration of Pb^{2+} , Cr^{3+} , and Fe^{3+} , $V_R = V_I$)

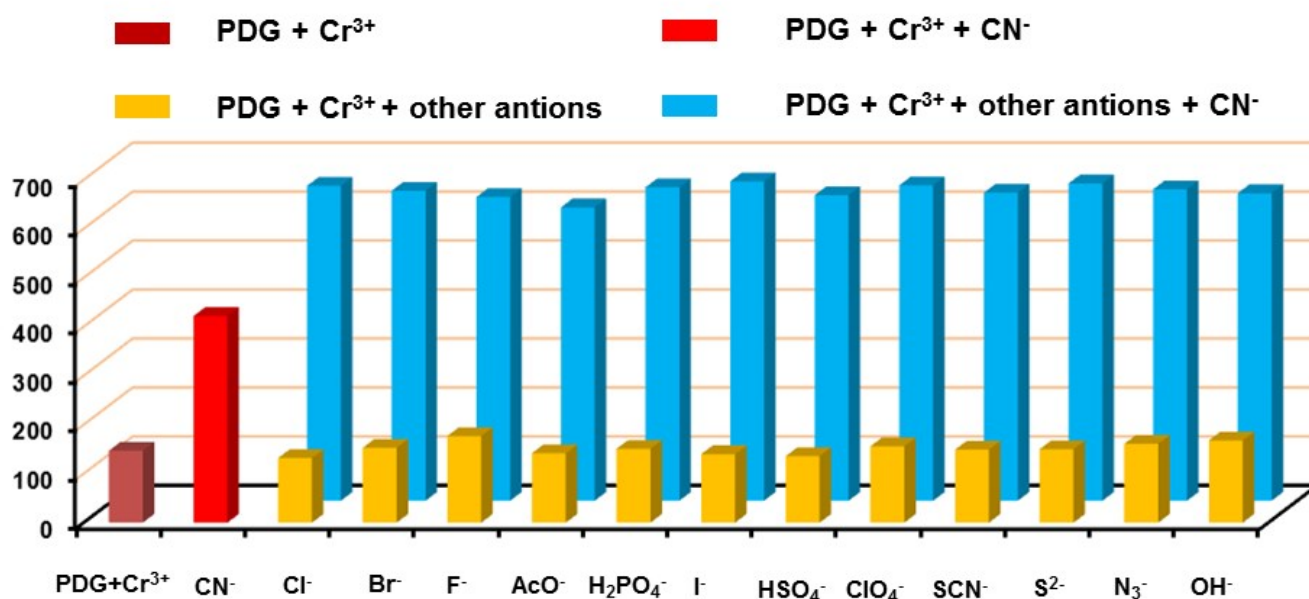


Fig. S23 Fluorescence responses of **PDG-Cr** to CN^- in the presence of various anions aqueous solution (CN^- , Cl^- , Br^- , F^- , AcO^- , H_2PO_4^- , I^- , HSO_4^- , ClO_4^- , SCN^- , S^{2-} , N_3^- , and OH^-) at 300 nm.

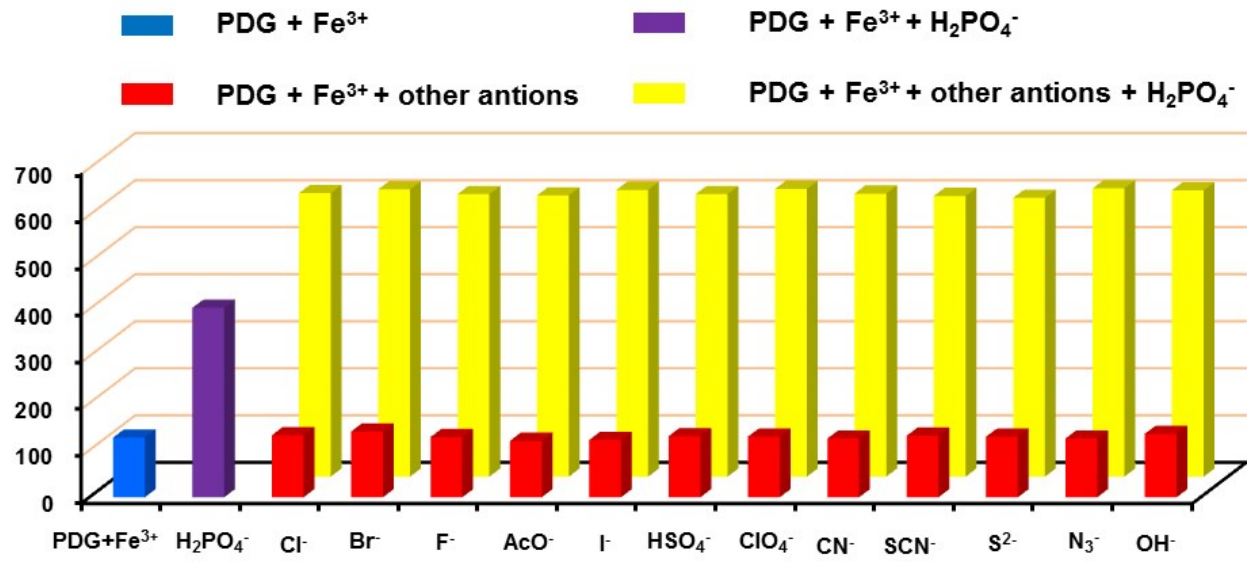


Fig. S24 Fluorescence responses of **PDG-Fe** to H_2PO_4^- in the presence of various anions aqueous solution (H_2PO_4^- , Cl^- , Br^- , F^- , AcO^- , I^- , HSO_4^- , ClO_4^- , CN^- , SCN^- , S^{2-} , N_3^- , and OH^-) at 300 nm.

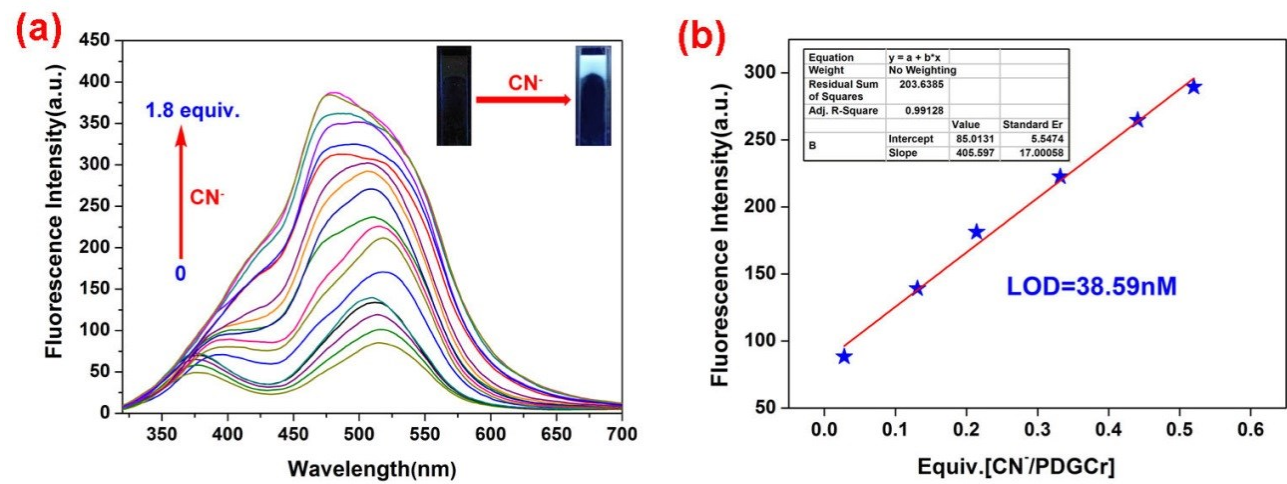


Fig. S25 (a) Fluorescent titration of **PDG-Cr** for CN^- ($\lambda_{\text{ex}} = 300 \text{ nm}$); (b) The photograph of the linear range for CN^- .

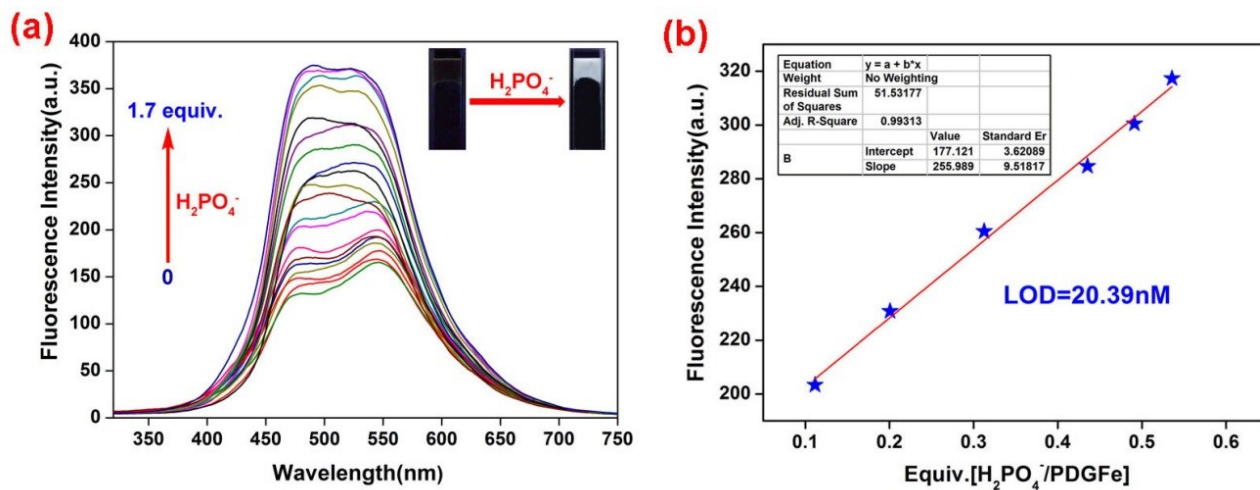


Fig. S26 (a) Fluorescent titration of **PDG-Fe** for H_2PO_4^- ($\lambda_{\text{ex}} = 300 \text{ nm}$); (b) The photograph of the linear range for H_2PO_4^- .

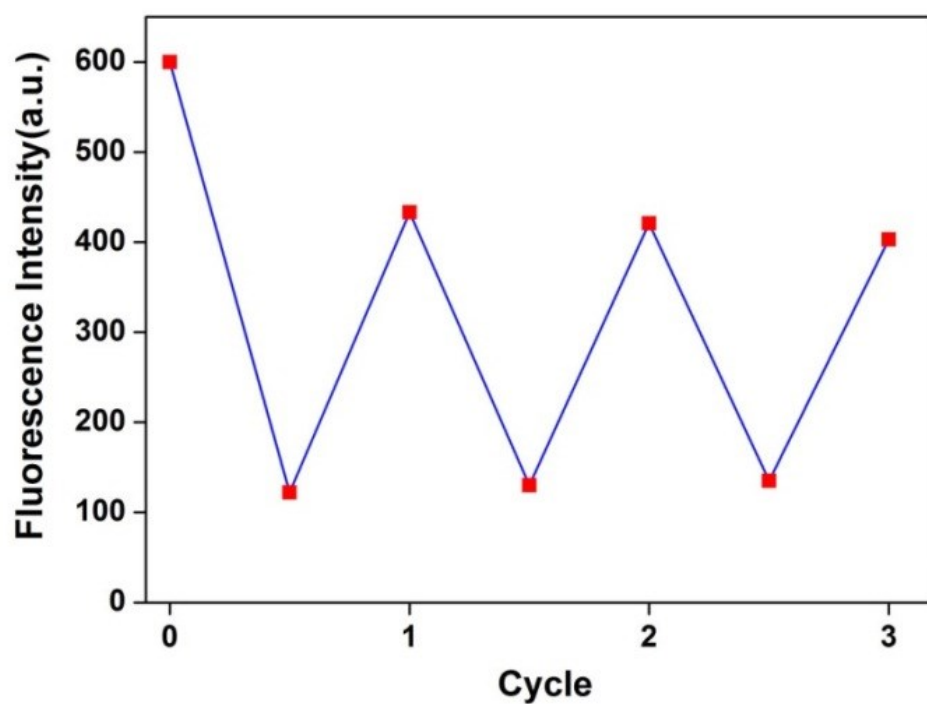


Fig. S27 Fluorescent "on-off-on" cycles of **PDG**, controlled by the alternative addition of Cr^{3+} and CN^- ($\lambda_{\text{ex}} = 300 \text{ nm}$).

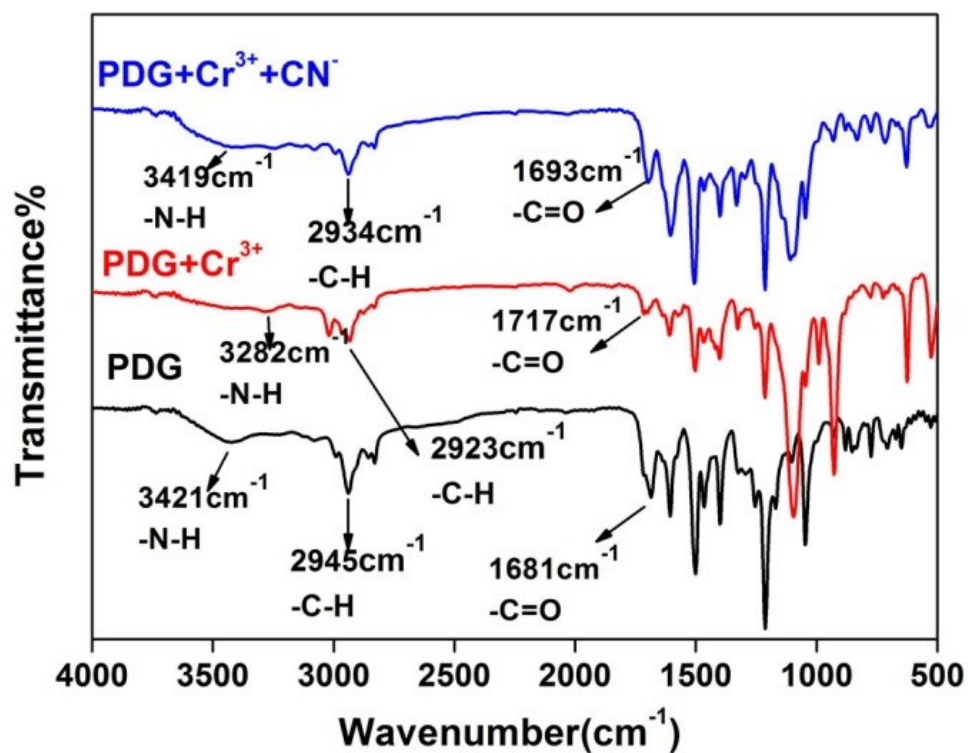


Fig. S28 FT-IR spectra of PDG, PDG-Cr and PDG-Cr + CN⁻.

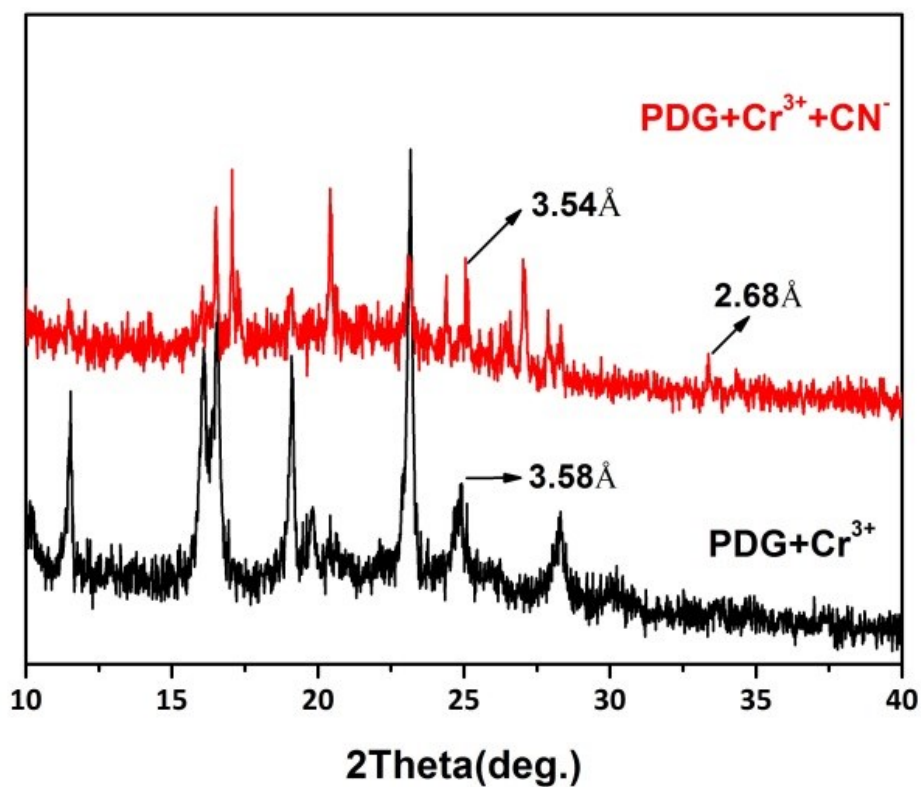


Fig. S29 XRD patterns of xerogel PDG-Cr and xerogel PDG-Cr + CN⁻.

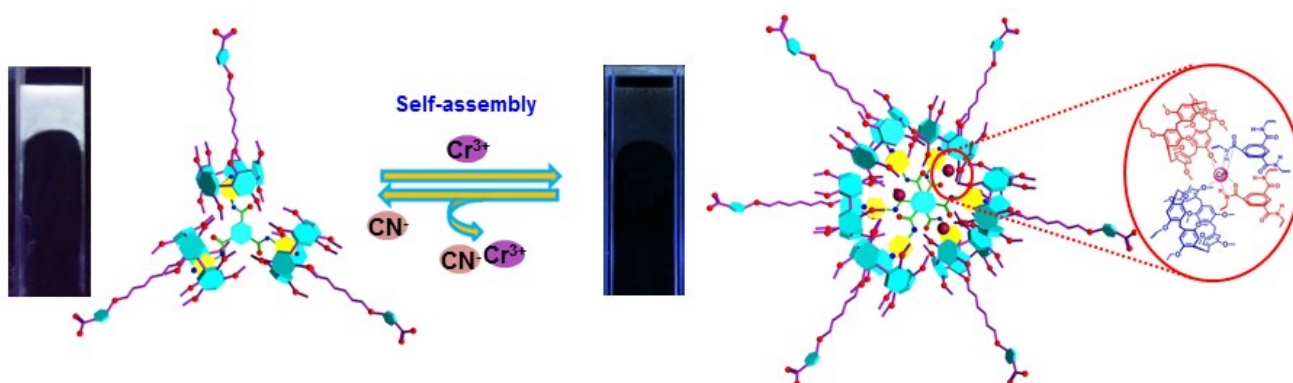


Fig. S30 A possible sensing mechanism of the **PDG** to Cr^{3+} and the **PDG-Cr** to CN^- .

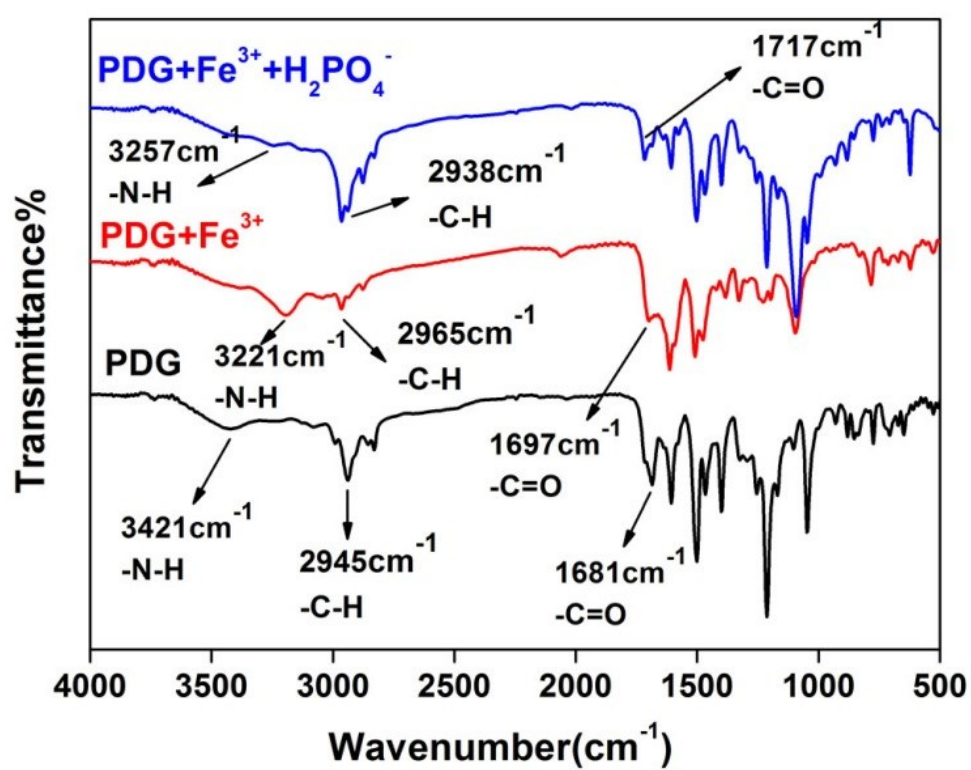


Fig. S31 FT-IR spectra of **PDG**, **PDG-Fe** and **PDG-Fe + H_2PO_4^-** .

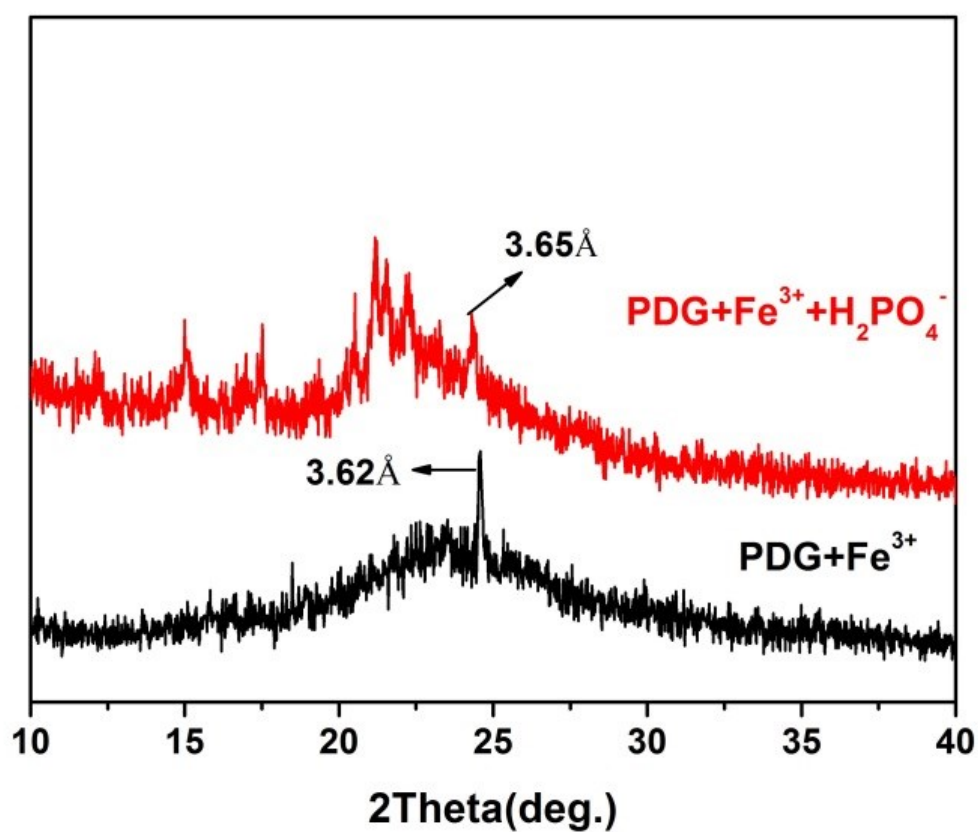


Fig. S32 XRD patterns of xerogel **PDG-Fe** and xerogel **PDG-Fe** + H_2PO_4^- .

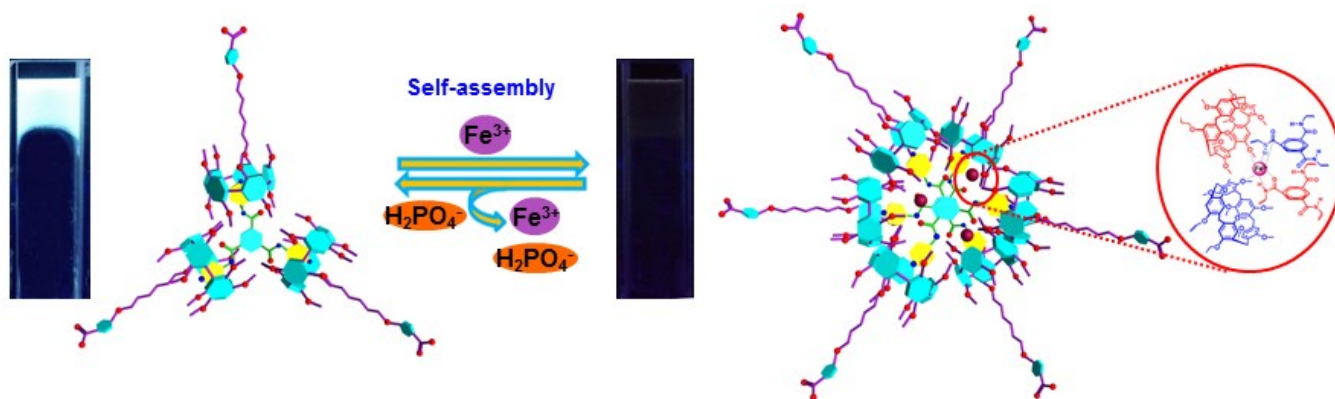


Fig. S33 A possible sensing mechanism of the **PDG** to Fe^{3+} and the **PDG-Fe** to H_2PO_4^- .

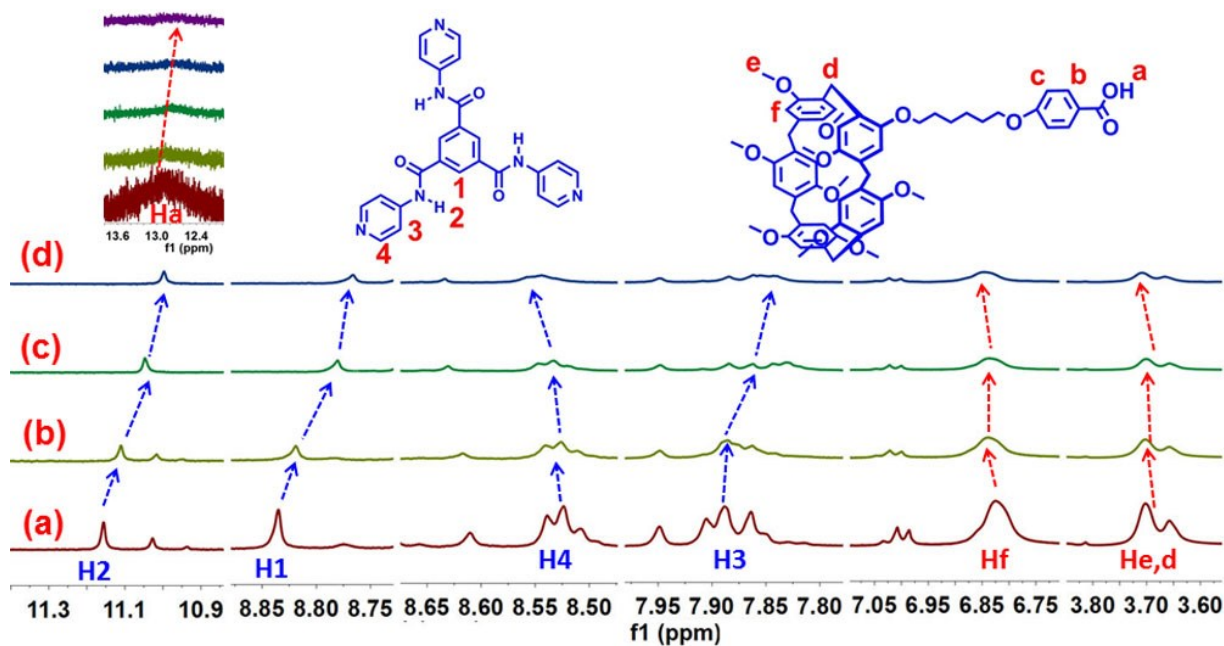


Fig. S34 Partial ^1H NMR spectra of **PDG** in $\text{DMSO-}d_6$ with different equivalent Pb^{2+} : (a) Free **PDG** (0.01 M) ; (b) 0.1 equiv.; (c) 0.5 equiv.; (d) 1.0 equiv..

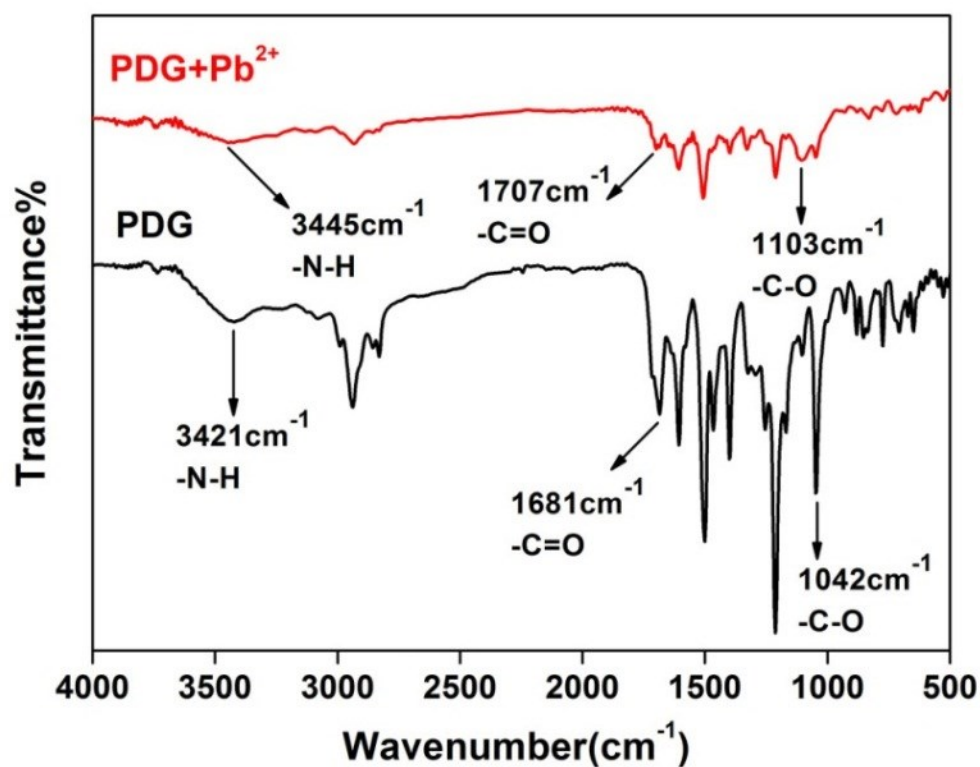


Fig. S35 FT-IR spectra of **PDG** and **PDG-Pb**.

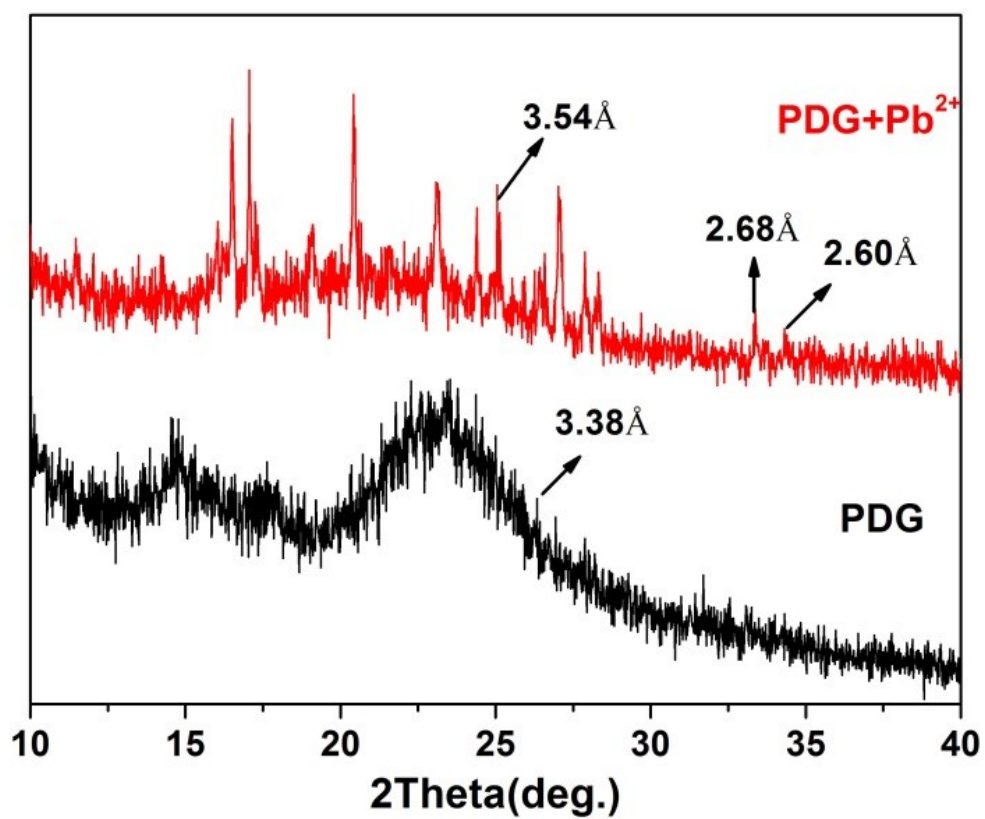


Fig. S36 XRD patterns of xerogel **PDG** and xerogel **PDG-Pb**.

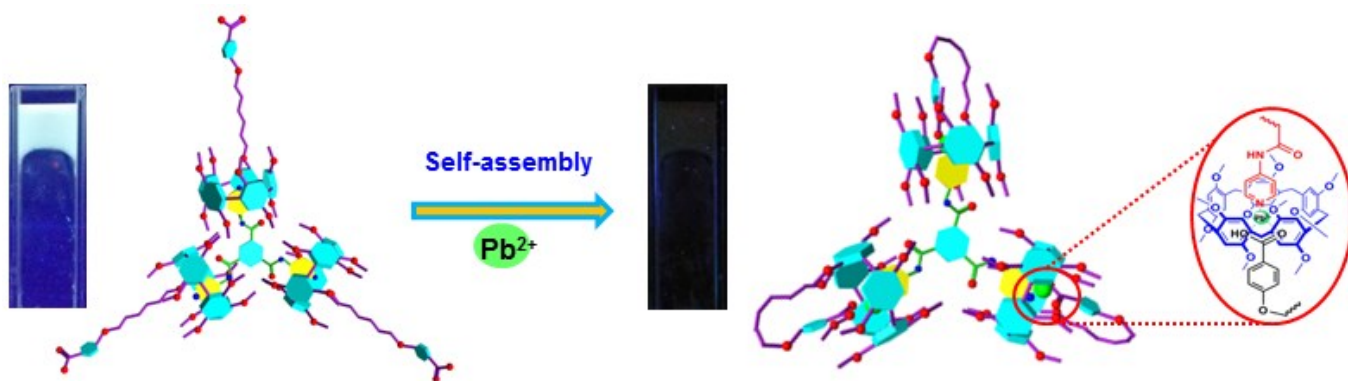


Fig. S37 A possible sensing mechanism of the **PDG** to **Pb²⁺**.