

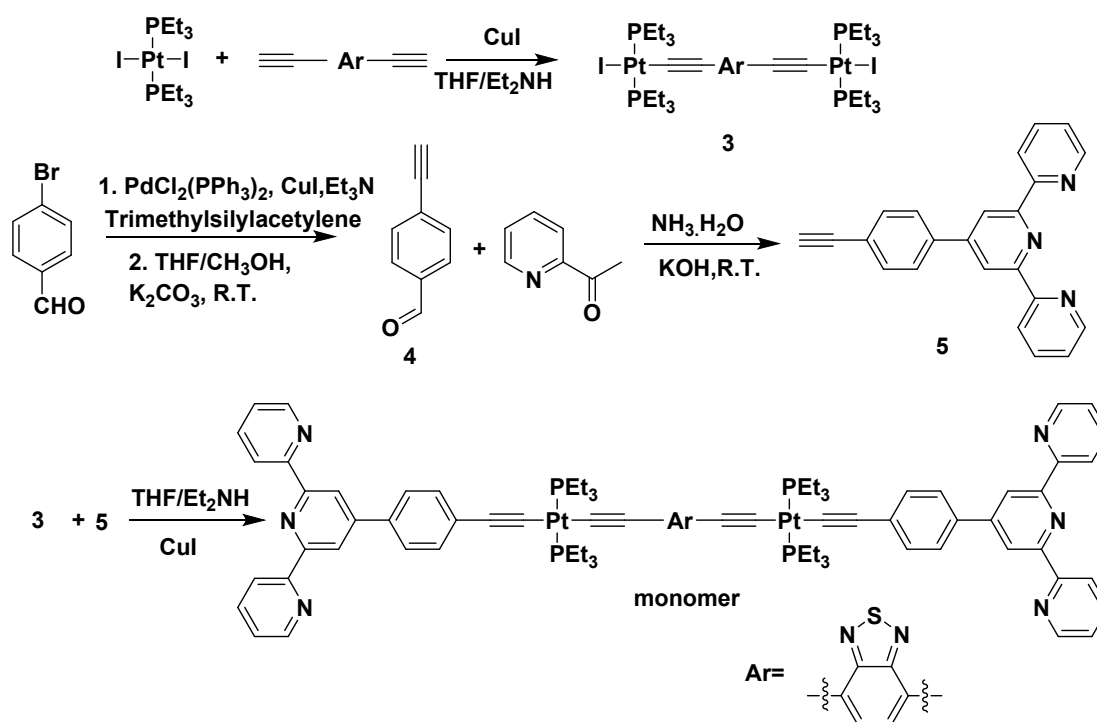
Supporting Information

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1. Materials and Methods

4-Bromobenzaldehyde, 2-Acetylpyridine, Triethylphosphine, copper(I) iodide, *Trans*-dichloro bis(triphenyl-phosphine)palladium(II) were reagent grade and used as received. *Trans*-[Pt(PEt₃)₂I₂], compound **3** and compound **4** were synthesized according to the previously reported method^[S1-S3]. Other reagents and solvents were employed as purchased. ¹H NMR spectra were collected on a Varian Unity INOVA-300 or INOVA-400 spectrometer with TMS as the internal standard. ¹³C NMR spectra were recorded on a Varian Unity INOVA-300 spectrometer at 100 MHz. The two-dimensional DOSY experiment was performed on a Varian Unity INOVA-400 MHz spectrometer. Electrospray ionization mass spectra (ESI-MS) were obtained on a Bruker Esquire 3000 plus mass spectrometer (Bruker-Franzen Analytik GmbH Bremen, Germany) equipped with an ESI interface and ion trap analyzer. The UV/Vis spectra were recorded on a Beijing Persee TU-1901 UV-Vis spectrometer. Viscosity measurements were carried out with a Ubbelohde semi-micro dilution viscometer (Shanghai Liangjing Glass Instrument Factory, 0.36 mm inner diameter) at 25 °C in CHCl₃/CH₃OH (2:1, v:v). ITC experiments were carried out using a Microcal VP-ITC apparatus.

2. Synthetic routes to the targeted monomer 1



Scheme S1. Synthetic route to the monomer.

2.1 Synthesis of compound 5

Compound **4** (0.56 g, 4.3 mmol), 2-Acetylpyridine (1.04 g, 8.6 mmol), KOH (0.5 g, 8.9 mmol) were dissolved in 20 mL $\text{NH}_3\cdot\text{H}_2\text{O}$ for 15 h at room temperature. The reaction mixture was filtrated and the solid was recrystallized by employing $\text{C}_2\text{H}_5\text{OH}$ to provide compound **5** a grayish yellow solid (430 mg, 30%). ^1H NMR spectrum of compound **5** is shown in Figure S1, ^1H NMR (300 MHz, CDCl_3 , room temperature) $\delta(\text{ppm})$: δ 8.73 (s, 4H), 8.68 (d, $J = 6$ Hz, 2H), 7.85-7.91 (m, 4H), 7.65 (d, $J = 9$ Hz, 2H), 7.34-7.38 (m, 2H), 3.19 (s, 1H).^[S4]

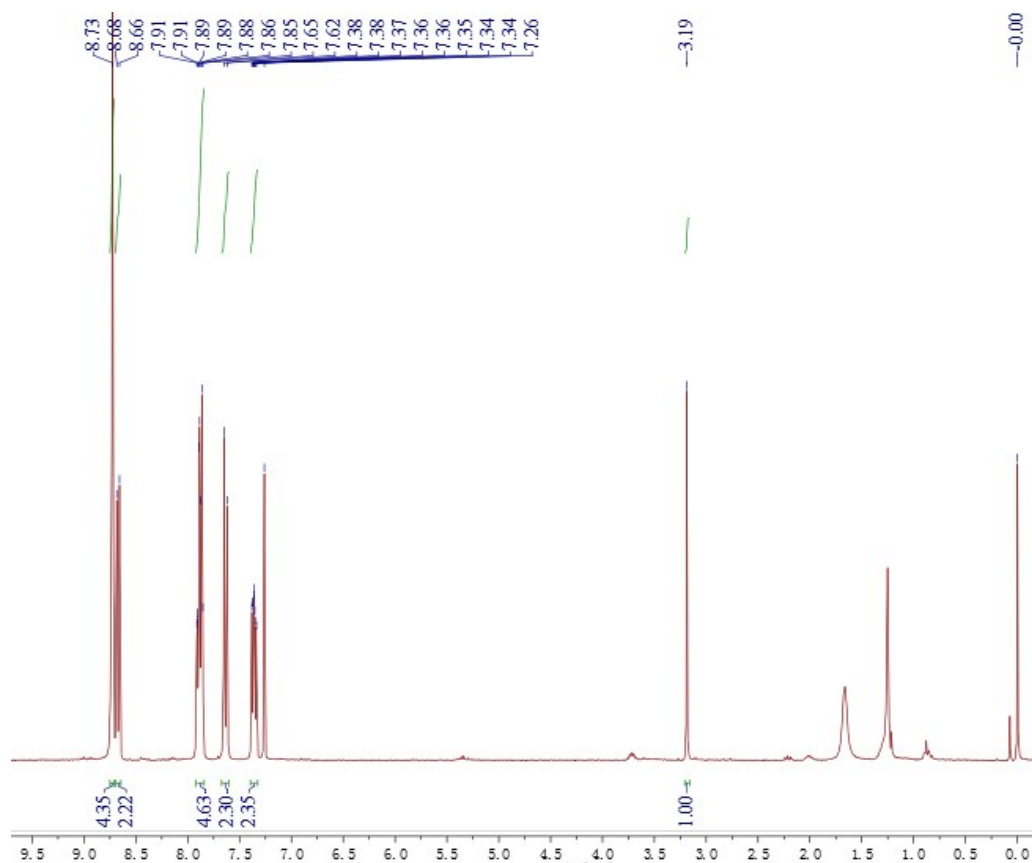


Figure S1. ^1H NMR spectrum (300 MHz, CDCl_3 , room temperature) of compound **5**

2.2 Synthesis of monomer

Compound **5** (186 mg, 0.56 mmol), CuI (15 mg, 0.08 mmol) were stirred in THF/ Et_2NH (30 mL, 1:1, v/v) at room temperature under N_2 atmosphere. Then compound **3** was added slowly to the reaction and continue stirred for 24 h. The reaction mixture was concentrated and the residue was extracted with $\text{H}_2\text{O}/\text{CH}_2\text{Cl}_2$. The organic extracts were combined and concentrated, which was further purified by flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{CH}_3\text{OH}=60:1$, v/v as the eluent) to provide monomer as a orange solid (187 mg, 49.1%). ^1H NMR spectrum of monomer is shown in Figure S2, ^1H NMR (300 MHz, CDCl_3 , room temperature) $\delta(\text{ppm})$: 8.73-8.74 (m, 8H), 8.68 (d, $J = 9.0$ Hz, 4H), 7.85-7.91 (m, 4H), 7.82 (d, $J = 6.0$ Hz, 4H), 7.43(d, $J = 9$ Hz, 4H), 7.34-7.37 (m, 6H), 2.26-2.34 (m, 24H), 1.25-1.32 (m, 36H).

The ^{13}C NMR spectrum of monomer is shown in Figure S3. ^{13}C NMR (100 MHz, CDCl_3 , room temperature) δ (ppm): 155.35, 155.24, 154.80, 148.98, 148.08, 135.81, 133.65, 130.36, 129.53, 128.82, 125.85, 122.73, 120.32, 118.03, 117.37, 116.62, 109.83, 108.97, 105.78, 15.38, 7.43. ESI-MS m/z : $[\text{M} + \text{H}]^+$, calculated 1709.78; found 1710.44. ^{31}P NMR (162 MHz, CDCl_3 , room temperature) δ (ppm): 11.35.

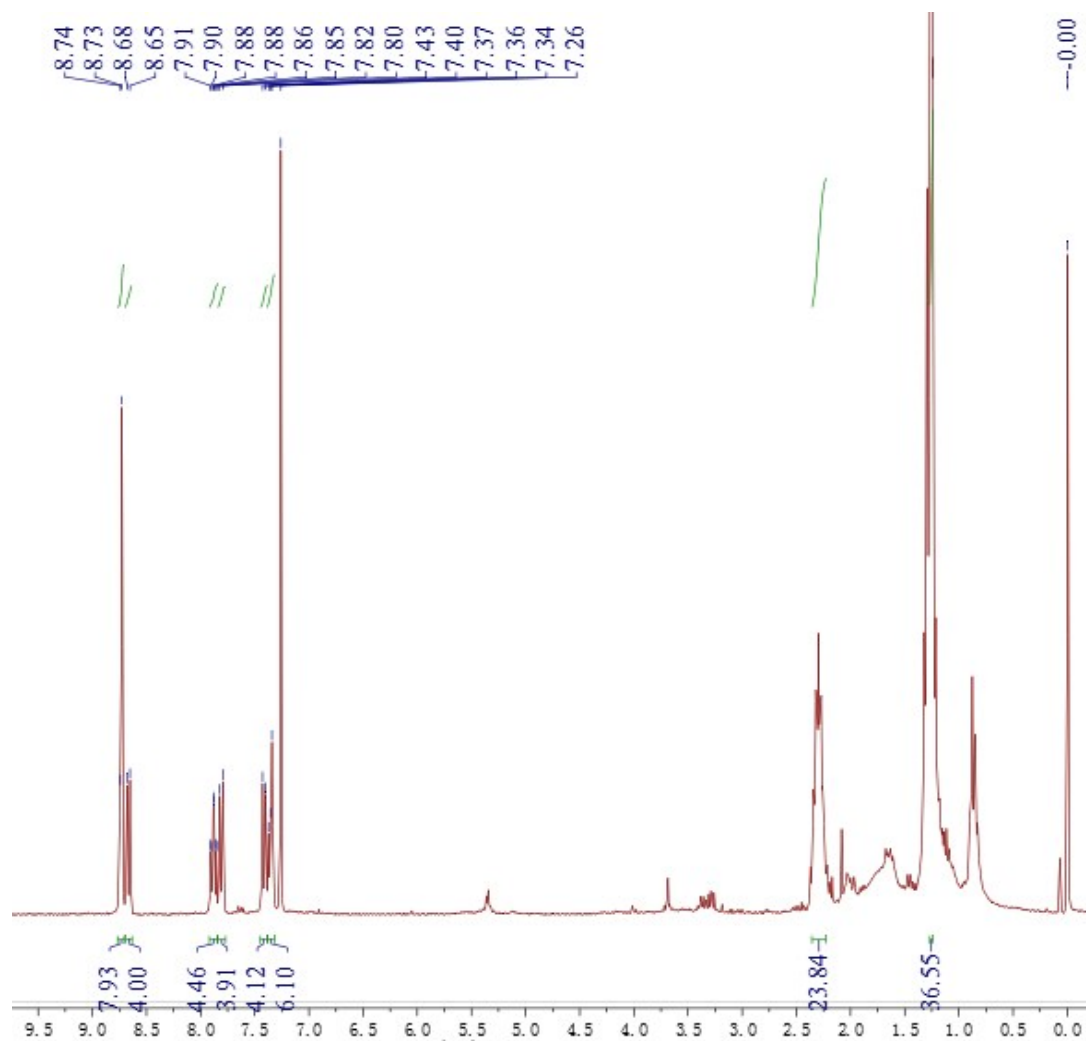


Figure S2. ^1H NMR spectrum (300 MHz, CDCl_3 , room temperature) of monomer

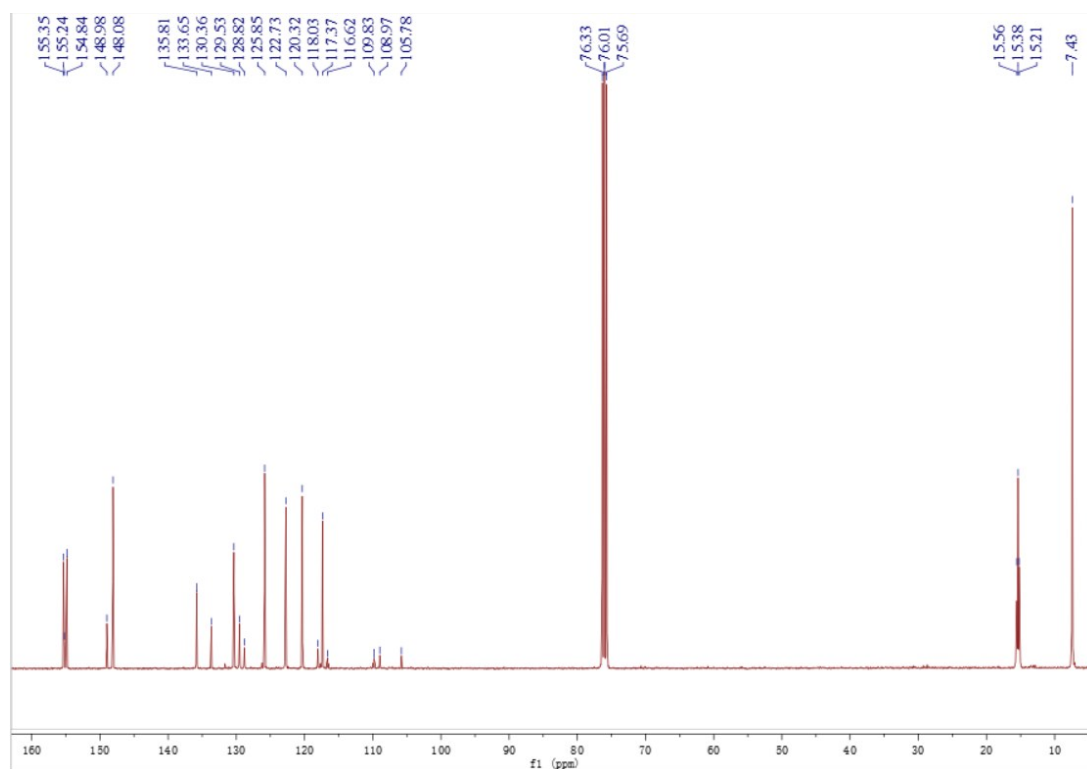


Figure S3. ^{13}C NMR spectrum (100 MHz, CDCl_3 , room temperature) of monomer

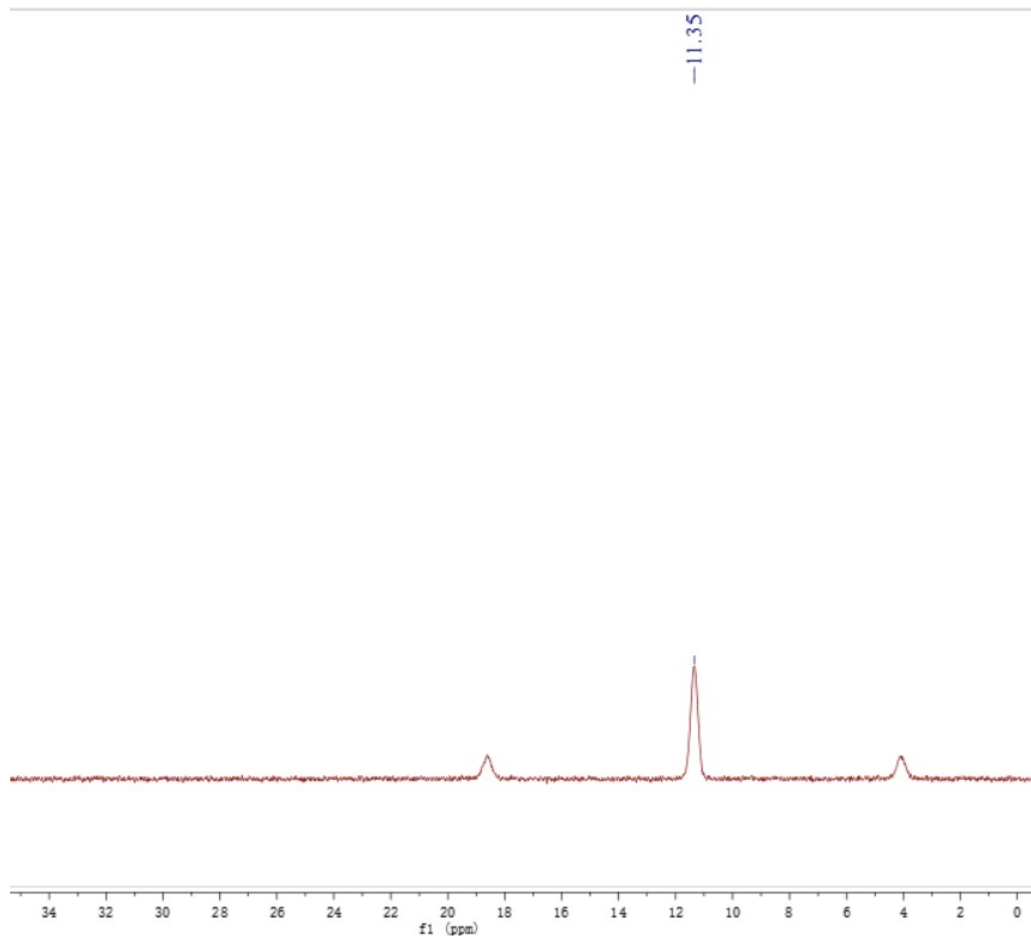


Figure S4. ^{31}P NMR spectrum (162 MHz, CDCl_3 , room temperature) of monomer

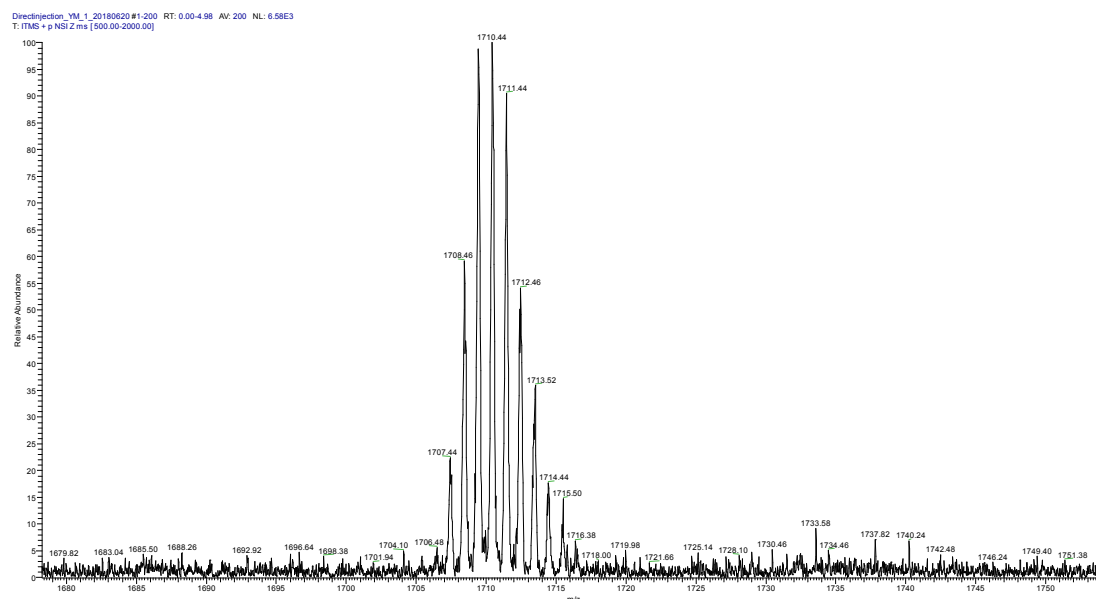


Figure S5. Electrospray ionization spectrum of monomer

3. ITC data for the consecutive injecting of $\text{Zn}(\text{OTf})_2$ into the monomer

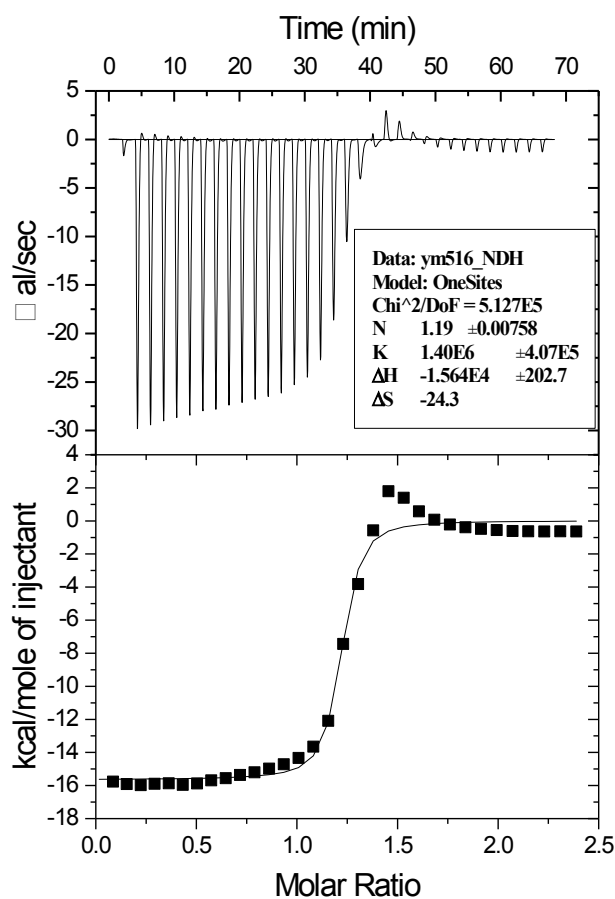


Figure S6. ITC data for the consecutive injecting of $\text{Zn}(\text{OTf})_2$ (5 mmol/L) into the $\text{CHCl}_3:\text{CH}_3\text{OH}=2:1$ solution of monomer (0.4 mmol/L)

4. The emission lifetime of monomer

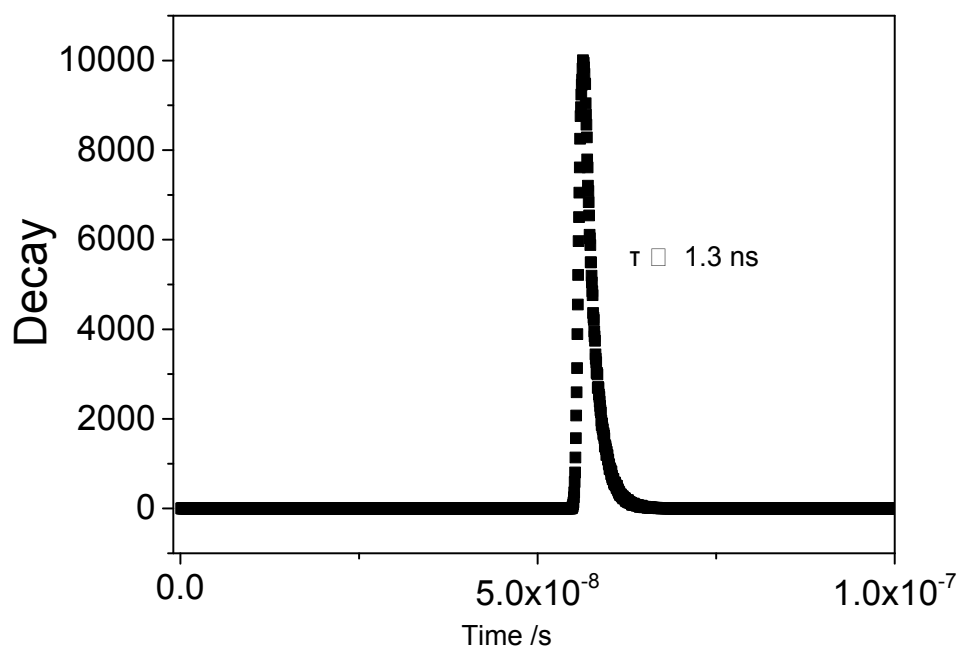


Figure S7. The emission lifetime of monomer

5. Concentration-dependent ^1H NMR spectra of supramolecular polymer **1**

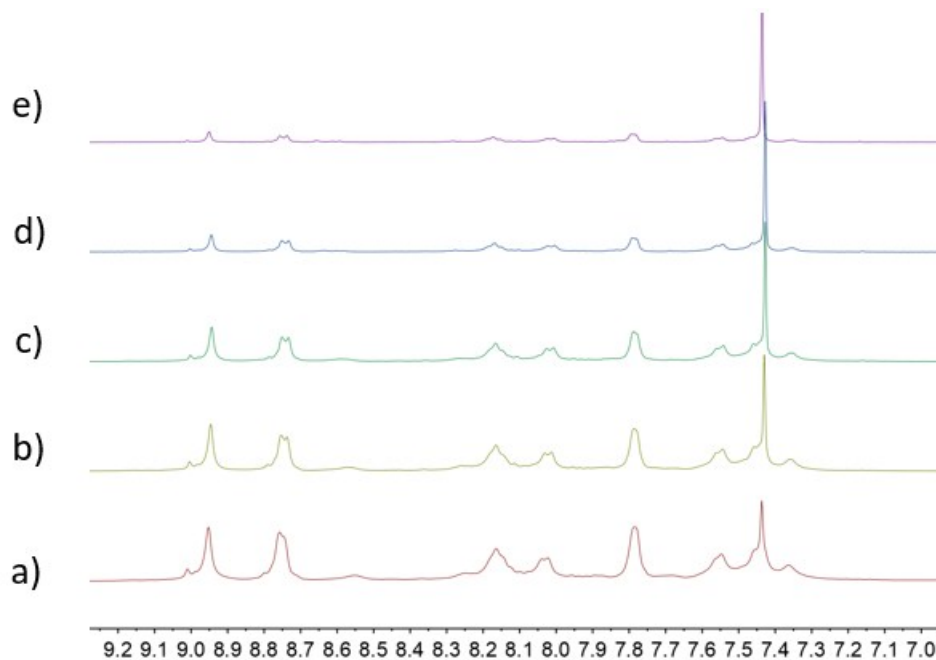


Figure S8. Concentration-dependent ^1H NMR (300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OH}$ (2/1, v/v), 25 $^\circ\text{C}$) spectra of supramolecular polymer **1**

6. DOSY spectra of supramolecular polymer **1**

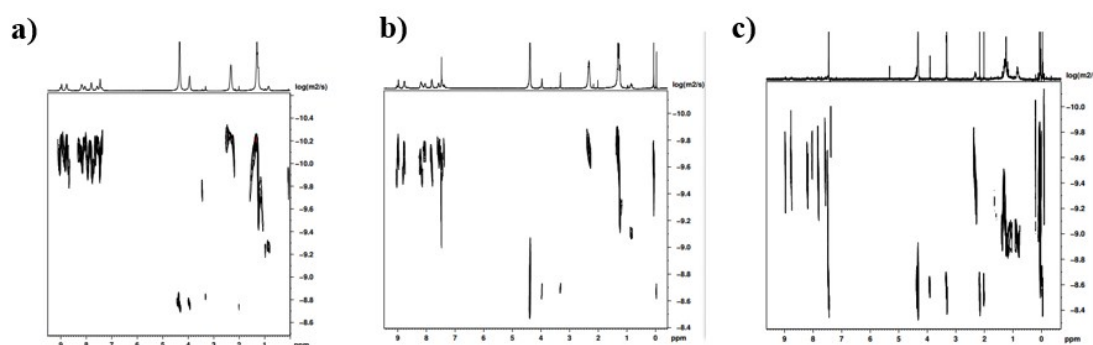


Figure S9. Two-dimensional diffusion-ordered NMR (DOSY) spectra of supramolecular polymer **1** in $\text{CDCl}_3/\text{CD}_3\text{OD}$ (2/1, v/v) at different concentrations: (a) 35.0 mM; (b) 11.7 mM; (c) 0.5 mM.

7. Responsiveness of supramolecular polymer **1** upon addition of cyclen, then Zn^{2+} .

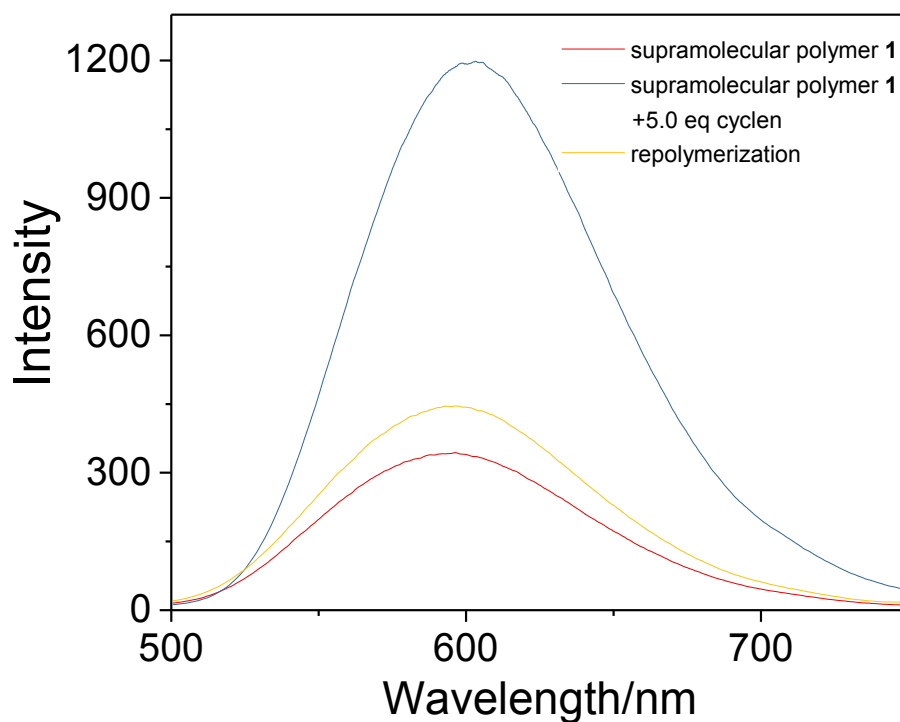


Fig S10 Fluorescence spectral changes of supramolecular polymer **1** upon addition of cyclen and then $\text{Zn}(\text{OTf})_2$.

8. ^1H -NMR responsiveness of supramolecular polymer **1** upon addition of cyclen, Zn^{2+}

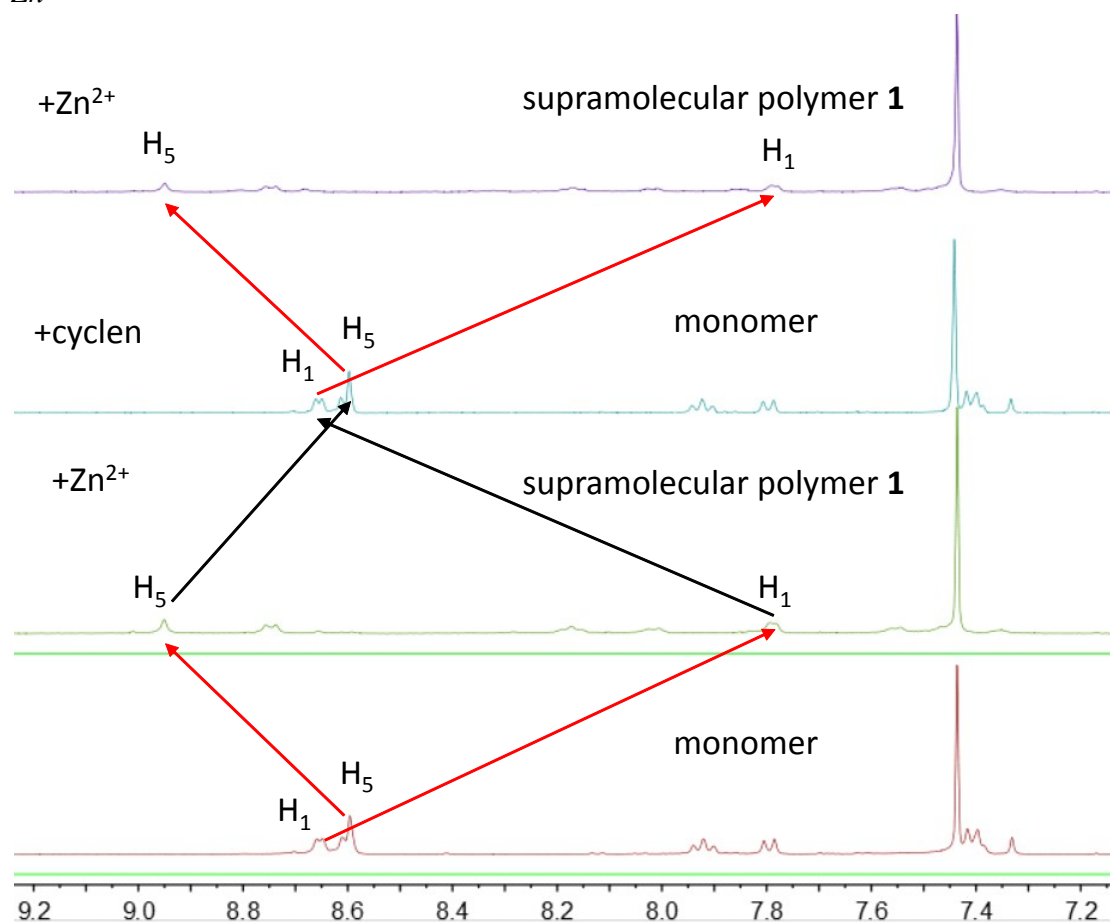


Fig S11. ^1H NMR of monomer, supramolecular polymer **1**, monomer, supramolecular polymer **1** (from bottom to top is monomer, supramolecular polymer **1**, monomer, supramolecular polymer **1**)

References:

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