Synthesis, luminescence enhancement, and self-assembly behaviours of platinum(II)-containing ABC triblock metallopolymers

Weisheng Meng,[a] Xiandeng Qiu,[a] Ran Wang,[a] Qun He*[a] and Weifeng Bu*[ab]

[a] *Key Laboratory of Nonferrous Metals Chemistry and Resources Utilization of Gansu Province, State Key Laboratory of Applied Organic Chemistry, and College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou, 730000, China. E-mail: buwf@lzu.edu.cn*
[b] *State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, China.*
*E-mail: hequn@lzu.edu.cn; buwf@lzu.edu.cn

EXPERIMENTAL SECTION

General Considerations. ^{1}H and ^{13}C NMR spectra were performed on a *JNM-ESC400* spectrometer. All of the samples were dissolved in *d*-chloroform ($CDCl_3$), in which tetramethylsilane was utilized as an internal standard. Fourier transformation infrared (FT-IR) spectra were recorded on a *Nicolet NEXUS 670* spectrometer with a resolution of 4 cm^{-1}, where the samples were jammed into thin KBr pellet. For the metallopolymers, C, H, and N elemental analyses were obtained with *Elementar Analysensysteme GmbH VarioEL* element analyser (Germany). UV-vis absorption and emission spectra was carried out by using a *Shimadzu 2550* spectrophotometer and a *Hitachi F-7000* spectrofluorimeter, respectively. Transmission electron microscopy (BF-TEM) was collected on a *JEM-2100* operating at 200 kV. For these microscopic measurements, the sample solutions (1.0×10^{-4} mol/L, 6 μL) were cast on carbon-coated copper grids and the excess liquids were blotted with filter papers.

4-Hydroxy-2,6-bis(*N*-methylbenzimidazol-2′-yl)pyridine,[S1] 4-(12-bromododecyloxy)phenyl acetylene,[S2] and 2-(*N*-dodecylbenzimidazol-2′-yl)-6-(*N*-{[(4-ethynylphenyl)oxy]dodecyl} benzimidazol-2′-yl)pyridine (**L-I**)[S3] were prepared according to the previously reported procedures, respectively. *N*,*N*-dimethylformamide (DMF) was dried over CaH_2 for 6 h and then used as a reaction medium for click reactions. Other chemicals were commercially used without any further purification. All reaction procedures were operated under an atmosphere of Ar.

Synthetic Procedures and Characterization Details

SE_{10}-N_3: SE_{10}-O_3SCH_3 (2.00 g, 2.48 mmol), and tetrabutylammonium iodide (0.0092 g, 0.025 mmol) were dissolved in anhydrous DMF (50 mL) in a Schlenk flask. The resulting mixture was cooled at 0 °C and sodium azide (NaN_3, 0.81 g, 12.40 mmol) was added. After warming to room temperature, the reaction mixture was then placed in an oil bath preheated at 50 °C for 48 h. During this reaction process, the solution slowly turned yellowish. After cooling the reaction mixture to room temperature, the solvent of DMF was removed under reduced pressure. The crude product was dissolved in CH_2Cl_2 and then washed by water three times. After drying over anhydrous $MgSO_4$, the organic layer was concentrated under reduced pressure and precipitated by addition of cold diethyl ether. The resulting white solid was collected *via* vacuum filtration and then dried under vacuum (Yield: 1.93 g, 97%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.68-3.38 (polyethylene glycol peak, m, 42H), 1.57 (m, 2H), 1.36-1.20 (m, 30H), 0.87 (t, $J = 8$ Hz, 3H).

SE_{20}-N_3 and **SE_{100}-N_3** were synthesized in an analogous procedure described for **SE_{10}-N_3**, but using **SE_{20}-O_3SCH_3** and **SE_{100}-O_3SCH_3** as reactants, respectively. The isolated yields were equal to or larger than 95% on the basis of the corresponding **SE_n-O_3SCH_3**.

4-[(4′-Ethynylphenyl)oxy]dodecyloxy-2,6-bis(*N*-methylbenzimidazol-2′-yl)pyridine (L-II): 4-Hydroxy-2,6-bis(*N*-methylbenzimidazol-2′-yl)pyridine (0.71 g, 2 mmol), 4-(12-bromododecyloxy)phenylacetylene (0.84 g, 2.4 mmol), K_2CO_3 (0.69 g, 5 mmol), and KI (0.33 g, 2 mmol) were added to 2-butanone in an oven dried Schlenk flask under an argon atmosphere. The resulting mixture was stirred at 80 °C for 72 h. After cooling the reaction mixture to room temperature, the insoluble solid was removed by filtration, and then the solvent was removed under reduced pressure. Further purification was performed by column chromatography on silica gel using petroleum ether/ethyl acetate (v/v = 2:1). The resulting pale pink solid was collected *via* vacuum filtration and then dried under vacuum (Yield: 1.03 g, 82%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (s, 2H), 7.87 (d, J = 8 Hz, 2H), 7.45 (d, J = 4 Hz, 2H), 7.39 (d, J = 8 Hz, 2H), 7.36 (m, J = 8 Hz, 4H), 6.83 (d, J = 4 Hz, 4H), 4.24 (m, 8H), 3.94 (t, J = 8 Hz, 2H), 2.98 (s, 1H), 1.86 (m, 2H), 1.78 (m, 2H), 1.47-1.29 (m, 16H).

SE_{10}L-I: CuBr (0.035 g, 0.24 mmol), **L-I** (0.180 g, 0.24 mmol), **SE_{10}-N_3** (0.15 g, 0.20 mmol), and PMDETA (0.10 mL, 0.48 mmol) were dissolved in anhydrous DMF (5 mL) in a round-bottom flask. The resulting mixture was degassed by three freeze-pump-thaw cycles, sealed under vacuum, and placed in an oil bath preheated at 90 °C for 72 h. During this reaction process, the solution changed gradually from brown to deep green. The brown colour should originate from a trace amount of oxidized 1,2-diaminobenzene impurities in the ligand of **L-I**. After the click reaction, the solution was changed into a green colour because of the formation of Cu(II)/PMDETA and Cu(II)/**L-I** complexes. After cooling the reaction mixture to room temperature, the solvent of DMF was removed under reduced pressure. The crude product was dissolved in CH_2Cl_2, which then passed through a neutral alumina column to remove the copper catalyst. The filtrate was concentrated to 5 mL and precipitated into 50 mL of cold ether. This dissolution-precipitation process was repeated for three times. The resulting white solid was collected *via* vacuum filtration and then dried under vacuum (Yield: 0.27 g, 83%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.31 (d, J = 8 Hz, 2H), 8.05 (t, J = 8 Hz, 1H), 7.89 (s, 1H), 7.88 (d, J = 8 Hz, 2H), 7.73 (d, J = 8 Hz, 2H), 7.45 (d, J = 8 Hz, 2H), 7.35 (m, J = 4 Hz, 4H), 6.94 (d, J = 8 Hz, 2H), 4.72 (t, J = 8 Hz, 4H), 4.57 (t, J = 8 Hz, 2H), 3.96 (t, J = 8 Hz, 2H), 3.90 (t, J = 8 Hz, 2H), 3.84~3.44 (polyethylene glycol peak, m+p, 38H), 1.73 (m, 6H), 1.57~1.00 (m, 66H), 0.87 (t, J = 8 Hz, 6H).

SE_{20}L-I: SE_{20}L-I was synthesized by following the reaction procedure depicted for **SE_{10}L-I** except that **SE_{20}-N_3** (0.29 g, 0.25 mmol) was utilized instead of **SE_{10}-N_3** (Yield: 0.39 g, 83%). ^{1}H NMR (400 MHz, $CDCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.31 (d, J = 8 Hz, 2H), 8.05 (t, J = 8 Hz, 1H), 7.89 (s, 1H), 7.88 (d, J = 8 Hz, 2H), 7.73 (d, J = 8 Hz, 2H), 7.45 (d, J = 8 Hz, 2H), 7.35 (m, J = 4 Hz, 4H), 6.94 (d, J = 8 Hz, 2H), 4.72 (t, J = 8 Hz, 4H), 4.57 (t, J = 8 Hz, 2H), 3.96 (t, J = 8 Hz, 2H), 3.90 (t, J = 8 Hz, 2H), 3.84~3.44 (polyethylene glycol peak, m+p, 78H), 1.73 (m, 6H), 1.57~1.00 (m, 66H), 0.87 (t, J = 8 Hz, 6H).

SE_{100}L-I: SE_{100}L-I was synthesized by following the reaction procedure depicted for **SE_{10}L-I** except that **SE_{100}-N_3** (1.18 g, 0.25 mmol) was utilized instead of **SE_{10}-N_3** (Yield: 1.01 g, 86%). ^{1}H NMR (400 MHz, CDCl3): ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.31 (d, J = 8 Hz, 2H), 8.05 (t, J = 8 Hz, 1H), 7.89 (s, 1H), 7.88 (d, J = 8 Hz, 2H), 7.73 (d, J = 8 Hz, 2H), 7.45 (d, J = 8 Hz, 2H), 7.35 (m, *J* = 4 Hz, 4H), 6.94 (d, *J* = 8 Hz, 2H), 4.72 (t, *J* = 8 Hz, 4H), 4.57 (t, *J* = 8 Hz, 2H), 3.96 (t, *J* = 8 Hz, 2H), 3.90 (t, *J* = 8 Hz, 2H), 3.84~3.44 (polyethylene glycol peak, m+p, 398H), 1.73 (m, 6H), 1.57~1.00 (m, 66H), 0.87 (t, *J* = 8 Hz, 6H).

$SE_{10}L$-II: $CuSO_4 \cdot 5H_2O$ (0.125 g, 0.5 mmol), **L-II** (0.35 g, 0.54 mmol), **SE_{10}-N_3** (0.38 g, 0.52 mmol), and ascorbate sodium (0.30 mg, 0.75 mmol) were dissolved in a liquid mixture of water (5 mL), tertiary butanol (20 mL), and dichloromethane (5 mL). The resulting mixture was degassed by three freeze-pump-thaw cycles, sealed under vacuum, and placed in an oil bath preheated at 90 °C for 72 h. During this reaction process, the solution changed gradually from blue to brown. After the click reaction, the brown colour should originate from a trace amount of oxidized 1,2-diaminobenzene impurities in the ligand of **L-II**. After cooling the reaction mixture to room temperature, the solvents were removed under reduced pressure. The crude product was dissolved in CH_2Cl_2, which then passed through a neutral alumina column to remove the copper catalyst. The filtrate was concentrated to 5 mL and precipitated into 50 mL of cold ether. This dissolution-precipitation process was repeated for three times. The resulting white solid was collected *via* vacuum filtration and then dried under vacuum (Yield: 0.63 g, 86%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (s, 2H), 7.88 (s, 1H), 7.86 (d, *J* = 8 Hz, 2H), 7.73 (d, *J* = 8 Hz, 2H), 7.46 (d, *J* = 8 Hz, 2H), 7.37 (m, *J* = 4 Hz, 4H), 6.95 (d, *J* = 8 Hz, 4H), 4.56 (t, *J* = 8 Hz, 2H), 4.23 (m, 8H), 3.98 (t, *J* = 8 Hz, 2H), 3.90 (t, *J* = 4 Hz, 2H), 3.84~3.35 (polyethylene glycol peak, m, 38H), 1.87 (m, 2H), 1.82 (m, 2H), 1.56~1.00 (m, 48H), 0.87 (t, *J* = 8 Hz, 3H).

$SE_{20}L$-II: **$SE_{20}L$-II** was synthesized by following the reaction procedure depicted for **$SE_{10}L$-II** except that **SE_{20}-N_3** (0.61 g, 0.52 mmol) was utilized instead of **SE_{10}-N_3** (Yield: 0.84 g, 88%). ^{1}H NMR (400 MHz, CDCl3): δ 7.98 (s, 2H), 7.88 (s, 2H), 7.86 (d, *J* = 8 Hz, 2H), 7.73 (d, *J* = 8 Hz, 2H), 7.46 (d, *J* = 8 Hz, 2H), 7.37 (m, *J* = 4 Hz, 4H), 6.95 (d, *J* = 8 Hz, 4H), 4.58 (t, *J* = 8 Hz, 2H), 4.23 (m, 8H), 3.98 (t, *J* = 8 Hz, 2H), 3.90 (t, *J* = 4 Hz, 2H), 3.84~3.35 (polyethylene glycol peak, m, 78H), 1.87 (m, 2H), 1.82 (m, 2H), 1.56~1.00 (m, 48H), 0.88 (t, *J* = 8 Hz, 3H).

$SE_{100}L$-II: **$SE_{100}L$-II** was synthesized by following the reaction procedure depicted for **$SE_{10}L$-II** except that **SE_{100}-N_3** (2.45 g, 0.52 mmol) was utilized instead of **SE_{10}-N_3** (Yield: 2.24 g, 80%). ^{1}H NMR (400 MHz, CDCl3): δ 7.92 (s, 2H), 7.87 (s, 2H), 7.86 (d, *J* = 8 Hz, 2H), 7.73 (d, *J* = 8 Hz, 2H), 7.44 (d, *J* = 8 Hz, 2H), 7.35 (m, *J* = 4 Hz, 4H), 6.91 (d, *J* = 8 Hz, 4H), 4.55 (t, *J* = 8 Hz, 2H), 4.22 (m, 8H), 3.96 (t, *J* = 8 Hz, 2H), 3.89 (t, *J* = 4 Hz, 2H), 3.80~3.33 (polyethylene glycol peak, m, 398H), 1.84 (m, 2H), 1.82 (m, 2H), 1.56~1.00 (m, 48H), 0.87 (t, *J* = 8 Hz, 3H).

SE_{10}Pt-I: Red solid K_2PtCl_4 (0.083 g, 0.20 mmol) was first added to a DMSO solution of **$SE_{10}L$-I** (0.30 g, 0.20 mmol, 20 mL). The resulting mixture was heated and stirred at 90 °C for 3 days, where an orange red solution formed. After removing the solvent of DMSO under reduced pressure, the crude product was dissolved in CH_2Cl_2, and stirred vigorously with an aqueous solution of NH_4PF_6 (0.17 g, 0.74 mmol, 20 mL) over night. The organic phase was collected and washed with cold water for five times (20 × 5 mL). After evaporating the solvent of CH_2Cl_2, a dark red solid was isolated with a yield of 94% (0.36 g) based on **$SE_{10}L$-I**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.61 (broad, 1H), 8.01 (broad, 2H), 7.90 (s, 1H), 7.78 (broad, 2H), 7.71 (d, *J* = 8 Hz, 2H), 7.21 (broad, 4H), 7.04 (broad, 2H), 6.90 (d, *J* = 8 Hz, 2H), 4.56 (broad, 2H), 4.33 (broad, 4H), 3.94~3.90 (m, broad, 4H), 3.80~3.44 (polyethylene glycol peak, m, 38H), 1.80 (m, 6H), 1.57~1.00 (m, broad, 66H), 0.88 (broad, 6H). FT-IR (KBr): 3435, 2922, 2887, 1643, 1604, 1522, 1467, 1352, 1299, 1106, 951, 844, 748, 558 cm^{-1}. Elemental analysis calcd. for **SE_{10}Pt-I**

($C_{89}H_{142}ClF_6N_8O_{11}PPt$, 1875.62 g mol^{-1}): C 56.99, H 7.63, N 5.97. Found:C 57.33, H 7.95, N 6.04.

SE$_{20}$Pt-I: Similarly, **SE$_{20}$-Pt-I** was prepared as a dark red solid, but using **SE$_{20}$L-I** (0.30 g, 0.15 mol) and K_2PtCl_4 (0.062 g, 0.15 mmol) as reactants. The yield was 91% (0.33 g) based on **SE$_{20}$L-I**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.62 (broad, 1H), 8.01 (broad, 2H), 7.89 (s, 1H), 7.75 (broad, 2H), 7.71 (d, J = 8 Hz, 2H), 7.21 (broad, 4H), 7.02 (broad, 2H), 6.90 (d, J = 8 Hz, 2H), 4.55 (broad, 2H), 4.32 (broad, 4H), 3.95~3.90 (m, broad, 4H), 3.80~3.44 (polyethylene glycol peak, m, 78H), 1.80 (m, 6H), 1.57~1.00 (m, broad, 66H), 0.88 (broad, 6H). FT-IR (KBr): 2887, 2741, 2698, 1470, 1346, 1282, 1243, 1149, 1107, 1061, 958, 843, 750, 556, 528 cm^{-1}. Elemental analysis calcd. for **SE$_{20}$Pt-I** ($C_{109}H_{182}ClF_6N_8O_{21}PPt$, 2316.15 g mol^{-1}): C 56.52, H 7.92, N 4.84. Found: C 56.78, H 7.65, N 5.23.

SE$_{100}$Pt-I: Similarly, **SE$_{100}$Pt-I** was prepared as a dark red solid, but using **SE$_{100}$L-I** (0.30 g, 0.05 mol) and K_2PtCl_4 (0.021 g, 0.05 mmol) as reactants. The yield was 90% (0.29 g) based on **SE$_{100}$L-I**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.66 (broad, 1H), 8.08 (broad, 2H), 7.89 (s, 1H), 7.78 (broad, 2H), 7.69 (d, J = 8 Hz, 2H), 7.23 (broad, 4H), 7.06 (broad, 2H), 6.90 (d, J = 8 Hz, 2H), 4.55 (broad, 2H), 4.41 (broad, 4H), 3.95~3.90 (m, broad, 4H), 3.80~3.44 (polyethylene glycol peak, m, 398H), 1.80 (m, 6H), 1.57~1.00 (m, broad, 66H), 0.88 (broad, 6H). FT-IR (KBr): 3430, 2888, 2741, 2694, 1617, 1467, 1360, 1281, 1242, 1150, 1100, 1060, 958, 842, 750, 557, 529 cm^{-1}. Elemental analysis calcd. for **SE$_{100}$Pt-I** ($C_{269}H_{502}ClF_6N_8O_{101}PPt$, 5840.35 g mol^{-1}): C 55.32, H 8.66, N1.92. Found: C 55.62, H 8.34, N 1.76.

SE$_{10}$Pt-II: Red solid K_2PtCl_4 (0.10 g, 0.24 mmol) was first added to a DMSO solution of **SE$_{10}$L-II** (0.3 g, 0.21 mmol, 20 mL). The resulting mixture was heated and stirred at 90 °C for 3 days, where an orange red solution formed. After removing the solvent of DMSO under reduced pressure, the crude product was dissolved in CH_2Cl_2, and stirred vigorously with an aqueous solution of NH_4PF_6 (0.24g, 1.48 mmol, 20 mL) over night. The organic phase was collected and washed with cold water for five times (20 × 5 mL). After evaporating the solvent of CH_2Cl_2, an orange solid was isolated with a yield of 96% (0.38 g) based on **SE$_{10}$L-II**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (broad, 2H), 8.09 (broad, 2H), 7.88 (broad, 2H), 7.72 (broad, 2H), 7.08 (broad, 2H), 7.01~6.87 (m, broad, 6H), 4.67 (broad, 2H), 4.52 (broad, 2H), 4.30 (broad, 4H), 4.06~3.88 (m, broad, 4H), 3.75~3.38 (polyethylene glycol peak, m, 36H), 3.33 (broad, 2H), 1.87 (m, 2H), 1.84 (broad, 2H), 1.56~1.00 (m, broad, 48H), 0.87 (broad, 3H). IR (KBr): 3443, 2921, 2856, 1653, 1467, 1343, 1280, 1248, 1113, 1036, 951, 842, 748, 557 cm^{-1}.Elemental analysis calcd. for **EG$_{45}$-Pt-II** ($C_{61}H_{104}ClF_6N_8O_{12}PPt$, 1611.68 g mol^{-1}): C 51.38, H 6.50, N 6.95. Found: C 51.21, H 6.54, N 6.63.

SE$_{20}$Pt-II: Similarly, **SE$_{20}$Pt-II** was prepared as a dark red solid, but using **SE$_{20}$L-II** (0.30 g, 0.16 mol) and K_2PtCl_4 (0.06 g, 0.14 mmol) as reactants. The yield was 94% (0.34 g) based on **SE$_{20}$L-II**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (broad, 2H),8.09 (broad, 2H), 7.88 (broad, 2H), 7.72 (broad, 2H), 7.08 (broad, 2H), 7.01~6.87 (m, broad,6H), 4.67 (broad, 2H), 4.52 (broad, 2H), 4.30 (broad, 4H), 4.06~3.88 (m, broad, 4H), 3.75-3.38 (polyethylene glycol peak, m, 76H), 3.33 (broad, 2H), 1.87 (broad, 2H), 1.84 (broad, 2H), 1.56~1.00 (m, broad, 48H), 0.87 (broad, 3H). FT-IR (KBr): 3441, 2885, 2741, 2697, 1610, 1468, 1346, 1282, 1244, 1147, 1110, 1050, 958, 842, 749, 556, 529 cm^{-1}. Elemental analysis calcd. for **SE$_{20}$Pt-II** ($C_{89}H_{144}ClF_6N_8O_{22}PPt$, 2053.63 g mol^{-1}): C 52.05, H 7.07, N 5.46. Found: C 52.24, H 7.36, N 5.24.

SE$_{100}$Pt-II: Similarly, **SE$_{100}$Pt-II** was prepared as a dark red solid, but using **SE$_{100}$L-II** (0.30 g, 0.06 mol) and K_2PtCl_4 (0.024 g, 0.06 mmol) as reactants. The yield was 95% (0.31 g) based on **SE$_{100}$L-II**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.76-3.43 (polyethylene glycol peak, m, 396H), 3.38 (broad, 2H), 1.73 (broad, 4H), 1.56~1.00 (m, broad, 48H), 0.87 (broad, 3H). FT-IR (KBr): 3448, 2887, 2741, 2697, 1629, 1469, 1347, 1283, 1244, 1147, 1109, 1032, 958, 841, 748, 556, 532 cm^{-1}. Elemental analysis calcd. for **SE$_{100}$Pt-II** ($C_{249}H_{464}ClF_6N_8O_{102}PPt$, 5577.84 g mol^{-1}): C 53.62, H 8.38, N 2.01. Found: C 53.46, H 8.52, N 1.86.

References:
S1. S. J. Rowan and J. B. Beck, *Faraday Discuss.*, 2005, **128**, 43.
S2. J. W. Y. Lam, A. Qin, Y. Dong, L. M. Lai, M. Häussler, Y. Dong and B. Z. Tang, *J. Phys. Chem. B*, 2006, **110**, 21613.
S3. F. Qu, B. Yang, Q. He and W. Bu, *Polym. Chem.*, 2017, **8**, 4716.

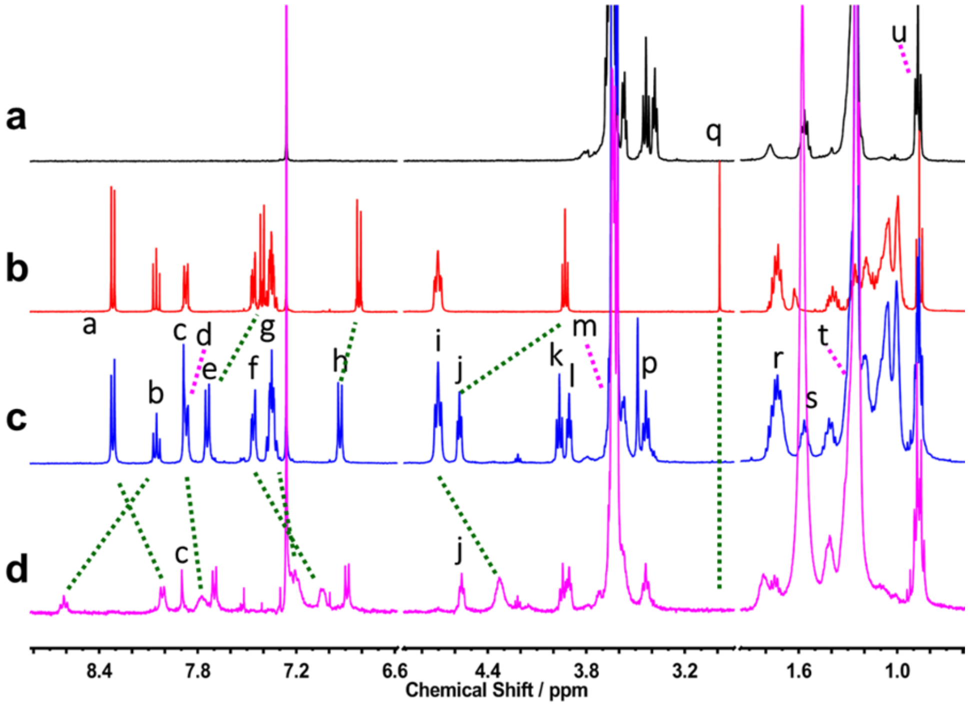

Fig. S1 ^{1}H NMR spectra of **SE$_{10}$-N$_3$** (a), **L-I** (b), **SE$_{10}$L-I** (c), and **SE$_{10}$Pt-I** in $CDCl_3$.

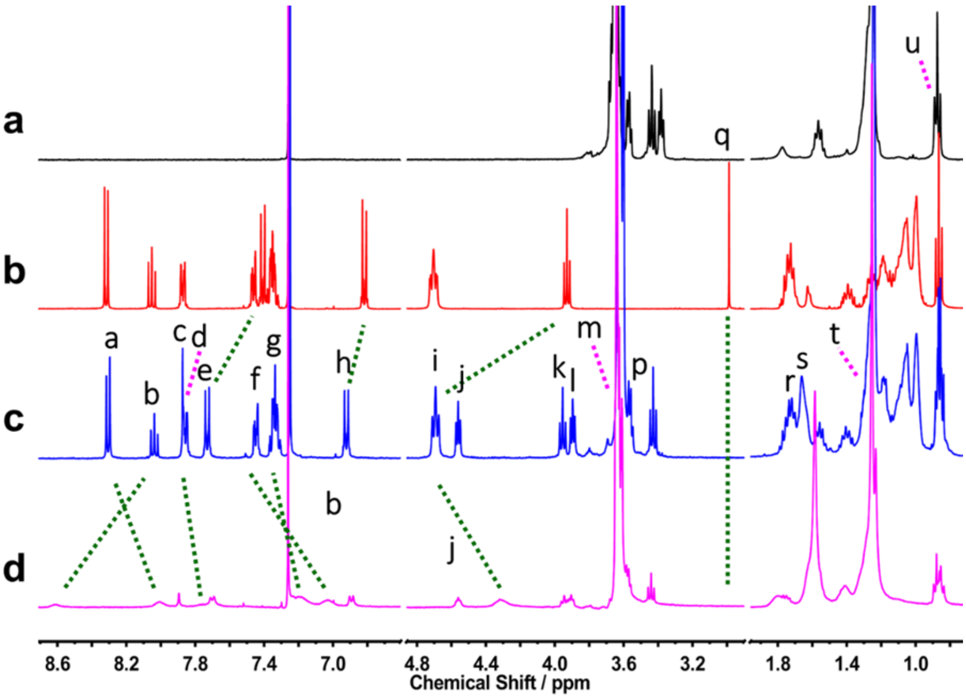

Fig. S2 ^{1}H NMR spectra of **SE$_{20}$-N$_3$** (a), **L-I** (b), **SE$_{20}$L-I** (c), and **SE$_{20}$Pt-I** in CDCl$_3$.

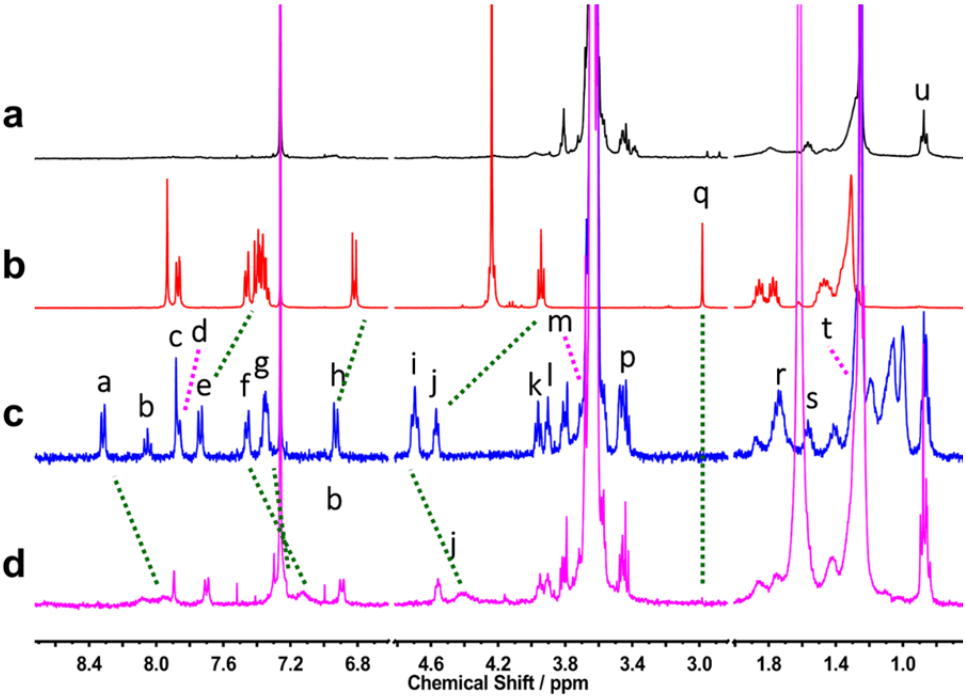

Fig. S3 ^{1}H NMR spectra of **SE$_{100}$-N$_3$** (a), **L-I** (b), **SE$_{100}$L-I** (c), and **SE$_{100}$Pt-I** in CDCl$_3$.

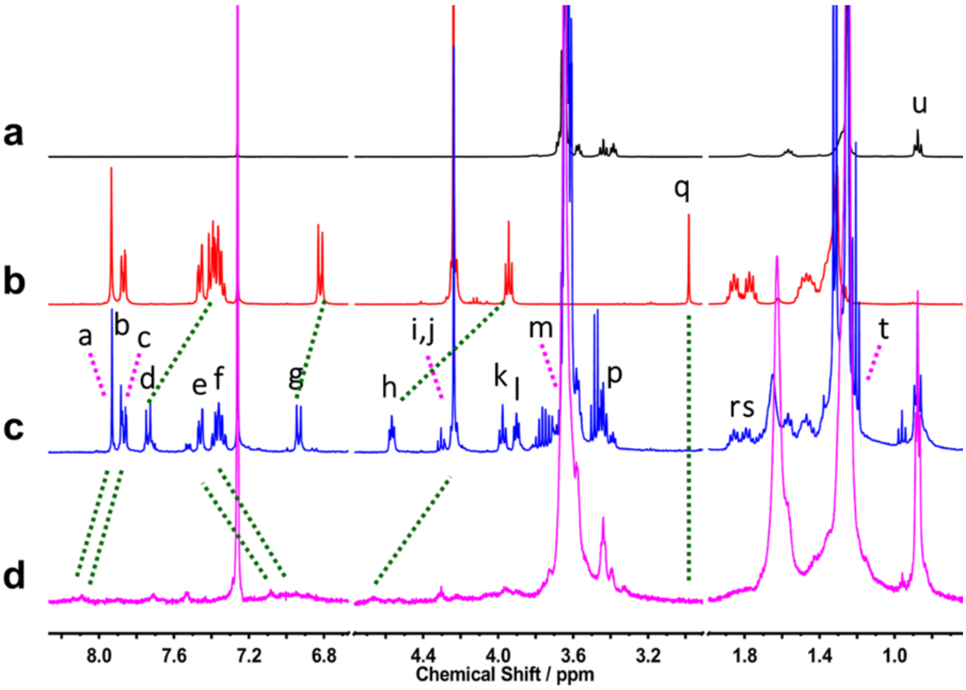

Fig. S4 ^{1}H NMR spectra of **SE_{20}-N_3** (a), **L-II** (b), **SE_{20}L-II** (c), and **SE_{20}Pt-II** in $CDCl_3$.

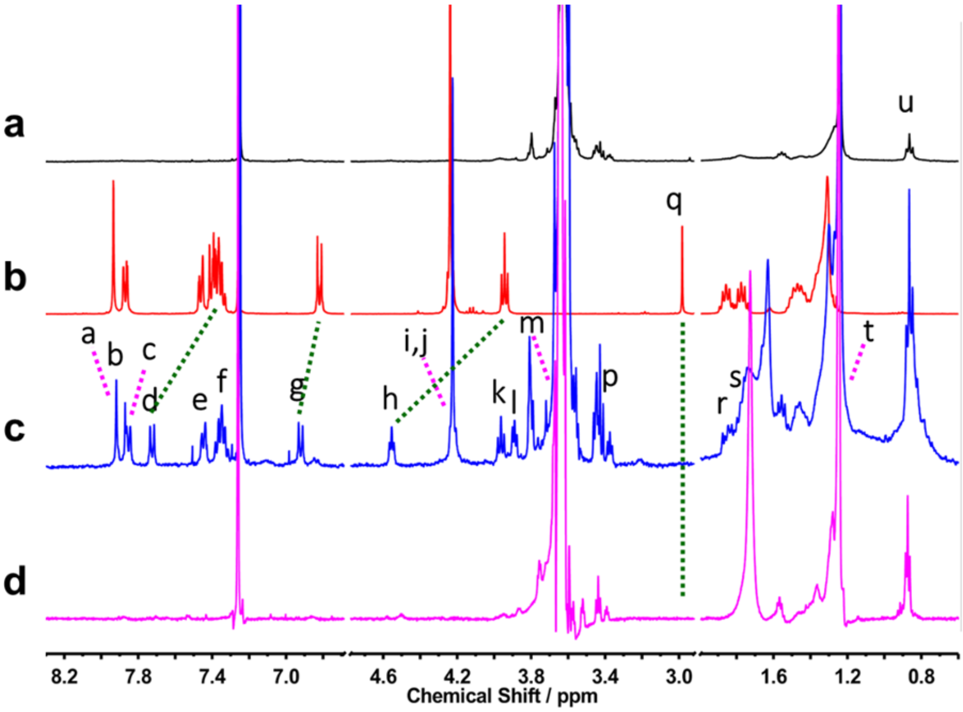

Fig. S5 ^{1}H NMR spectra of **SE_{100}-N_3** (a), **L-II** (b), **SE_{100}L-II** (c), and **SE_{100}Pt-II** in $CDCl_3$.

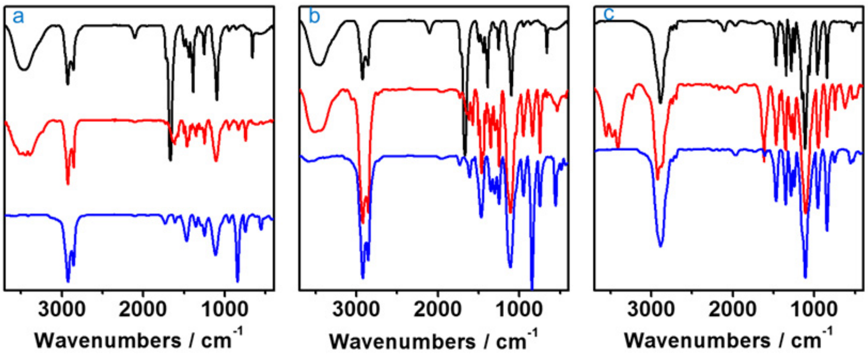

Fig. S6 FT-IR spectra of (a, **SE$_{10}$-N$_3$**, **SE$_{10}$L-I**, and **SE$_{10}$Pt-I**), (b, **SE$_{20}$-N$_3$**, **SE$_{20}$L-I**, and **SE$_{20}$Pt-I**), and (c, **SE$_{100}$-N$_3$**, **SE$_{100}$L-I**, and **SE$_{100}$Pt-I**).

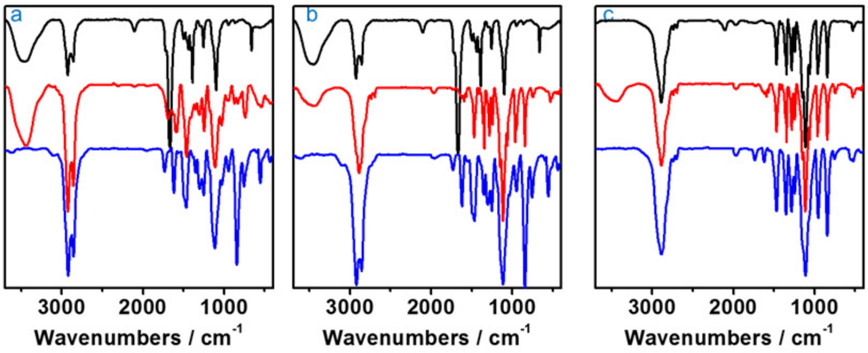

Fig. S7 FT-IR spectra of (a, **SE$_{10}$-N$_3$**, **SE$_{10}$L-I**, and **SE$_{10}$Pt-I**), (b, **SE$_{20}$-N$_3$**, **SE$_{20}$L-I**, and **SE$_{20}$Pt-I**), and (c, **SE$_{100}$-N$_3$**, **SE$_{100}$L-I**, and **SE$_{100}$Pt-I**).

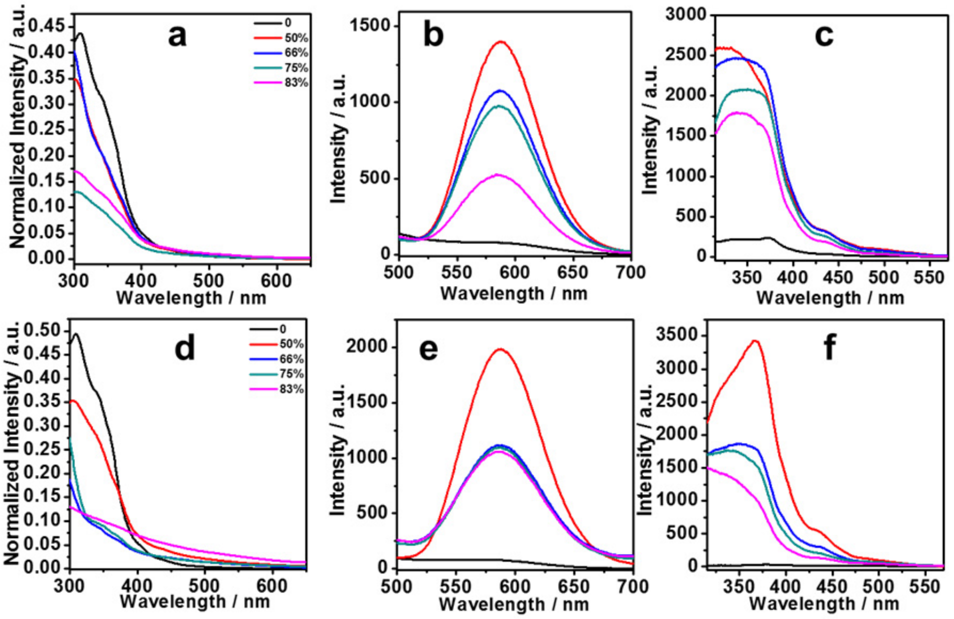

Fig. S8 (a and d) UV−vis absorption, (b and e) emission and (c and f) excited spectral changes of **SE$_{20}$Pt-II** (Top) and **SE$_{100}$Pt-II** (Bottom) upon increasing the *n*-hexane composition from 0 to 83 vol % in THF.

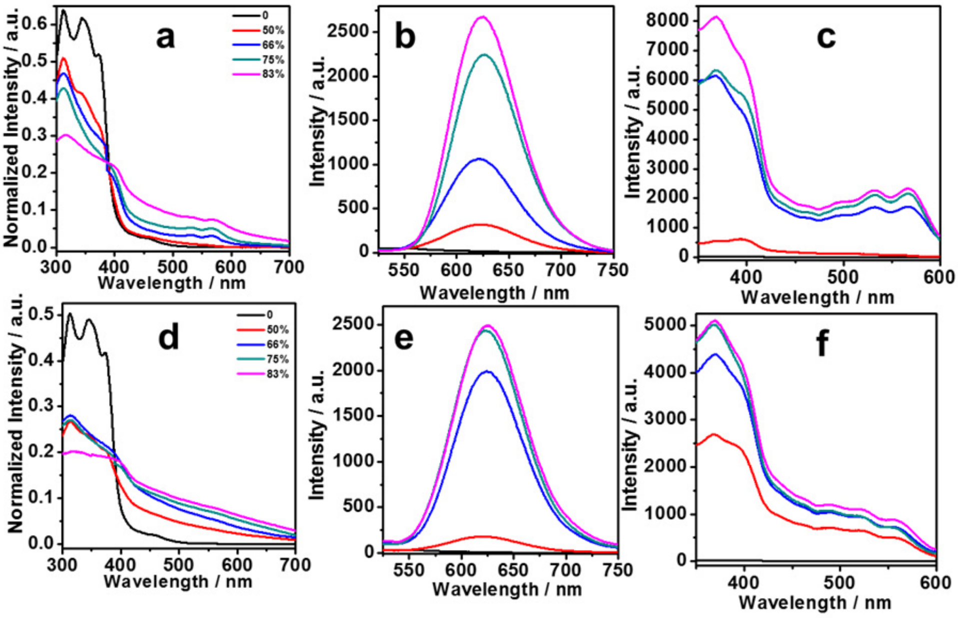

Fig. S9 (a and d) UV−vis absorption, (b and e) emission and (c and f) excited spectral changes of **SE$_{20}$Pt-I** (Top) and **SE$_{100}$Pt-I** (Bottom) upon increasing the *n*-hexane composition from 0 to 83 vol % in THF.

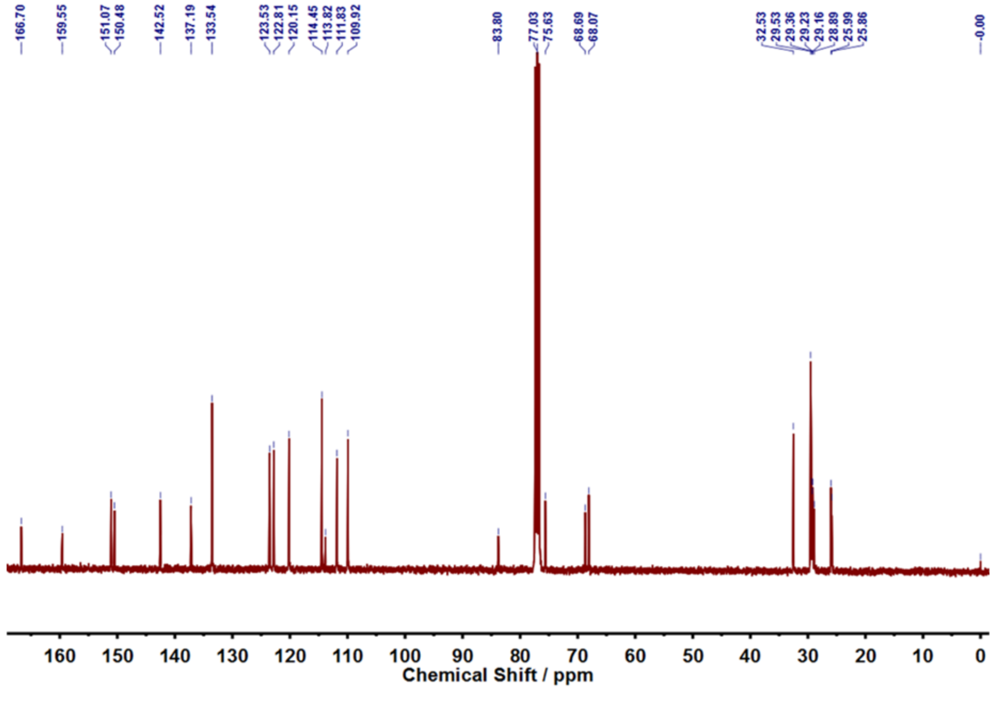

Fig. S10 ^{13}C NMR spectra of **L-II** in CDCl$_3$.

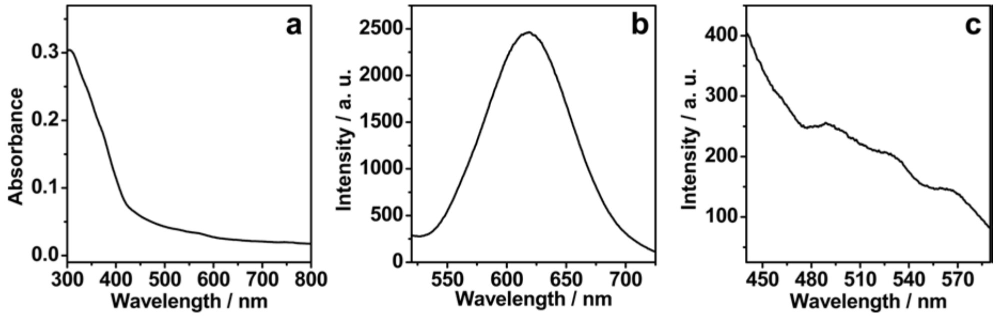

Fig. S11 (a) UV−vis absorption, (b) emission and (c) excited spectra of the mixture solution of **SE$_{10}$Pt-I** (5.0×10^{-5} mol L^{-1}) and **SE$_{10}$Pt-II** (5.0×10^{-5} mol L^{-1}). The mixture solution was prepared in the THF/*n*-hexane mixture solvent with an *n*-hexane content of 83 vol %, wherein the total concentration of the platinum(II) complexes was consistent with those of the isolated metallopolymers (1.0×10^{-4} mol L^{-1}, Fig. 4). The resulting emission spectrum was similar to the overlapping one from **SE$_{10}$Pt-I** (1.0×10^{-4} mol L^{-1}) and **SE$_{10}$Pt-II** (1.0×10^{-4} mol L^{-1}). Moreover, no luminescence energy transferring and no luminescence quenching were observed in the present study.