Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.
This journal is © The Royal Society of Chemistry 2019

Supporting Information

Structure-Tuned and Thermodynamically Controlled Mechanochromic Self-Recovery of AIE-Active Au(I) Complexes

Yubao Dong,[#a] Jing Zhang,*[#a,b] Aisen Li,[#c] Junyi Gong,[b] Benzhao He,[b] Shuping Xu,[c] Jun Yin,[a] Sheng Hua Liu*[a, d] and Ben Zhong Tang*[b]

[a] Key Laboratory of Pesticide and Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, China

[b] Department of Chemistry, The Hong Kong Branch of Chinese National Engineering Research Center for Tissue Restoration and Reconstruction, Institute for Advanced Study, Department of Chemical and Biological Engineering and Institute of Molecular Functional Material, The Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong, China

[c] State Key Laboratory of Supramolecular Structure and Materials, College of Chemistry, Jilin University, Changchun 130012, P. R. China

[d]State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou 350002, P. R. China

Materials and Methods

Materials: All starting materials and reagents were purchased from Shen Shi Hua Gong Company (China) and used without further purification. Dichloromethane was dried with calcium hydride then distilled. Compounds **1a**[1], **2a**[2], **3a**[3], **4a**[4], **5a**[5], **6a**[4], **7a**[6], **1b**[7], **3b**[7], **4b**[8], **5b**[9] and $C_6F_5Au(tht)$[10] (tht = tetrahydrothiophene) were synthesized according to the literatures.

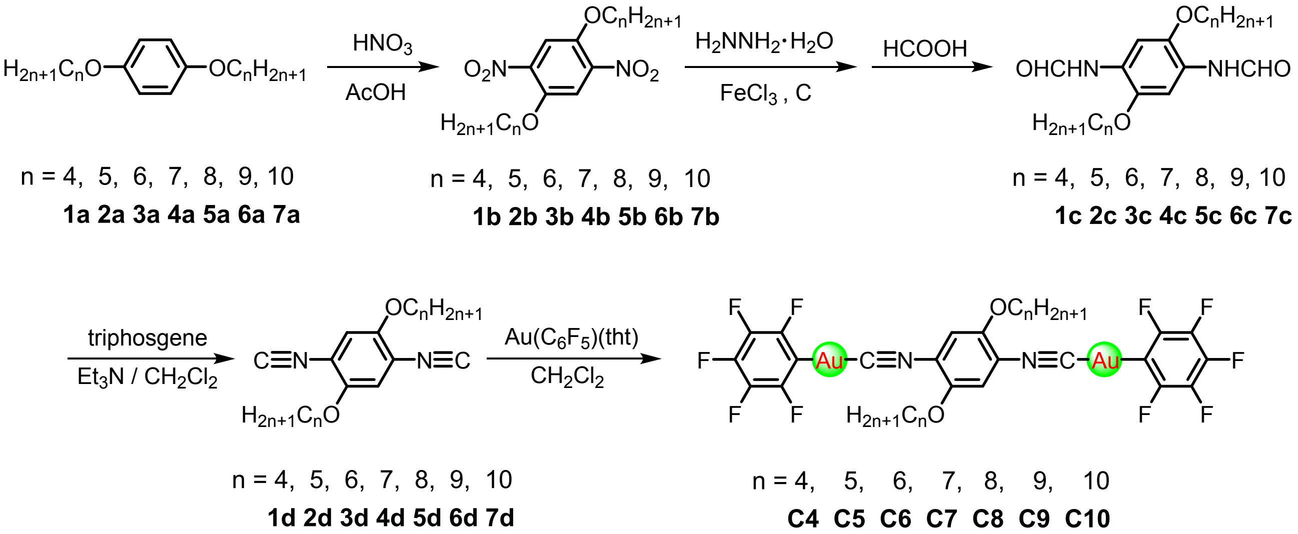
Characterization: Elemental analyses (C, H, N) were performed by the Microanalytical Services, College of Chemistry, CCNU. ^1H and ^{19}F NMR spectra were collected on American Varian Mercury Plus 400 spectrometer (400 MHz). ^1H NMR chemical shifts are relative to TMS. ^{19}F NMR chemical shifts are relative to C_6F_6 (δ = -163.00). Powder XRD patterns were performed with Shimadzu XRD-6000 diffractometer. Single-crystal structures were obtained by Bruker Apex CCD X-ray diffractometer. TGA traces were recorded on a NETZSCH STA 409 PG/PC. CCDC numbers for crystals **C6G** (green emission), **C6O** (orange emission), **C7**, **C8**, **C9** and **C10** are 1939844, 1940006, 1939845, 1939846, 1939847 and 1939848 respectively.

Photophysical experiments: The liquid and solid-state PL spectra were recorded on Horiba FluoroMax-P. The absolute fluorescence quantum yields were recorded on a FLS1000 Photoluminescence Spectrometer with an integrating sphere. Emission lifetime measurements were recorded on a FLS1000 Photoluminescence Spectrometer. The UV-visible absorption spectrum were studied on U-3310 UV spectrophotometer. Dynamic light scattering (DLS) measurements were performed on the Zetasizer instrument ZEN3600 (Malvern, UK) with a 173° back scattering angle and He-Ne laser (λ =633 nm).

High-pressure experiments: High-pressure experiments were carried out in diamond anvil cell (BGI-type DAC) with 500 μm in diameter. T301 stainless steel sheet was drilled a hole with 200 μm in diameter to serve as gasket. A small ruby chip in the sample chamber was used for in situ pressure calibration based on the fluorescence of the ruby R1 line. Silicone oil was used as a pressure-transmitting medium (PTM) to obtain the hydrostatic pressure. The measurements of the ruby chip were performed at a Horiba Jobin Yvon T64000 Raman spectrometer with a 1800 gr/mm holographic grating with a laser of 532 nm. The high-pressure PL spectra under hydrostatic pressure were measured using a fluorescence microscope equipped with a spectrometer (Horiba Jobin Yvon iHR320), and the excitation wavelength is 365 nm. The high-pressure Raman spectra were recorded by using a confocal Raman system (LabRAM Aramis, Horiba Jobin Yvon) and the excitation source was a 785 nm laser with power of 23 mW for

Raman[11-13].

Synthesis



Scheme S1. Synthetic route for complexes **C4~C10**.

Synthesis of compounds 2b, 6b, 7b.

Synthesis of **2b**: concentrated nitric acid (6 mL) was added dropwise to a solution of 1,4-dipentyloxybenzene (7.0 g, 20.6 mmol) in glacial acetic acid (120 mL) at room temperature. The mixture was stirred for 30 min at 80 °C. After cooling, iced water (200 mL) was added to the reaction mixture, and the yellow precipitate was filtered from the solution. The residue was subjected to column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) to give pure compound **2b**. Derivatives **6b** and **7b** were synthesized and purified according to the general procedure above.

1,4-Dipentyloxy-2,5-dinitrobenzene (2b). Yellow solid (3.0 g), 32% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.51 (s, 2H), 4.06 (t, J = 6.4 Hz, 4H), 1.88-1.69 (m, 4H), 1.52-1.22 (m, 8H), 0.92 (t, J = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 145.4, 141.9, 111.9, 71.0, 28.6, 27.8, 22.3, 13.9.

1,4-Dinonyloxy)-2,5-dinitrobenzene (6b). Yellow solid (3.3 g), 38% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.51 (s, 2H), 4.07 (t, J = 6.4 Hz, 4H), 1.92-1.71 (m, 4H), 1.53-1.16 (m, 24H), 0.88 (t, J = 6.8 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 145.5, 142.0, 112.0, 70.9, 31.9, 29.5, 29.3, 29.0, 28.9, 25.8, 22.8, 14.2.

1,4-Didecyloxy-2,5-dinitrobenzene (7b). Yellow solid (2.8 g), 33% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.48 (s, 2H), 4.05 (t, *J* = 6.6 Hz, 4H), 1.88-1.67 (m, 4H), 1.52-1.15 (m, 28H), 0.88 (t, *J* = 6.6 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 145.5, 142.0, 112.0, 70.9, 32.0, 29.6, 29.4, 29.3, 29.0, 28.9, 25.8, 22.8, 14.2.

Synthesis of compounds 1c~7c.

Synthesis of **1c**: A mixture of 1,4-dibutoxy-2,5-dinitrobenzene (2.5 g, 8.0 mmol), activated carbon (1.6 g) and anhydrous ferric chloride (0.32 g, 2.0 mmol) in ethanol (100 mL) was refluxed for 2 h under an argon atmosphere and then hydrazine monohydrate (32 mL) was added dropwise. The resulting solution was heated at 80 °C for 3 h. After cooling to room temperature, the catalyst was filtered out and ethanol was evaporated under reduced pressure. The residue was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic phase was dried over anhydrous magnesium sulfate and the solvent was evaporated. The residue was dissolved in formic acid (35 mL) and the mixture was refluxed for 12 h. After completion of present reaction, formic acid was removed from reaction system by distillation, the residual mixture was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic phase was dried over anhydrous magnesium sulfate and the solvent was evaporated. The residue was poured into methanol (100 mL), a large amount of solids separated out in the mixture. After filtration, the crude product was washed with methanol three times, and then dried to afford pure compound **1c**. Derivatives **2c~7c** were synthesized and purified according to the general procedures above.

N,N'-(2,5-dibutoxy-1,4-phenylene)bisformamide (1c). Pale purple solid (0.69 g), 28% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 8.67 (d, *J* = 11.4 Hz, 1H), 8.44 (d, *J* = 6.6 Hz, 1H), 8.15 (d, *J* = 17.4 Hz, 1H), 7.78 (s, 2H), 6.75 (s, 1H), 4.19-3.89 (m, 4H), 1.94-1.67 (m, 4H), 1.57-1.36 (m, 4H), 1.17-0.81 (m, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 158.8, 140.8, 122.8, 104.7, 69.2, 31.4, 19.4, 14.0.

N,N'-(2,5-dipentyloxy-1,4-phenylene)bisformamide (2c). Pale purple solid (0.62 g), 25% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 8.68 (d, *J* = 12.0 Hz, 1H), 8.44 (s, 1H), 8.15 (d, *J* = 17.4 Hz, 1H), 7.80 (s, 2H), 6.75 (s, 1H), 4.17-3.88 (m, 4H), 1.93-1.71 (m, 4H), 1.58-1.21 (m, 8H), 1.16-0.74 (m, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 158.8, 140.8, 122.8, 104.7, 69.5, 29.1, 28.3, 22.6, 14.1.

N,N'-(2,5-dihexyloxy-1,4-phenylene)bisformamide (3c). Pale purple solid (0.74 g), 30% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 8.67 (d, *J* = 12.0 Hz, 1H), 8.43 (s, 1H), 8.15 (d, *J* = 17.4

Hz, 1H), 7.76 (d, *J* = 23.4 Hz, 2H), 6.76 (d, *J* = 22.2 Hz, 1H), 4.15-3.86 (m, 4H), 1.98-1.67 (m, 4H), 1.58-1.13 (m, 12H), 1.06-0.76 (m, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 158.8, 140.8, 122.8, 104.8, 69.5, 31.7, 29.4, 25.9, 22.7, 14.2.

N,N'-(2,5-diheptyloxy-1,4-phenylene)bisformamide (4c). Pale purple solid (0.77 g), 31% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 8.67 (d, *J* = 12.0 Hz, 1H), 8.43 (s, 1H), 8.15 (d, *J* = 17.4 Hz, 1H), 7.76 (d, *J* = 23.4 Hz, 2H), 6.74 (s, 1H), 4.19-3.83 (m, 4H), 1.99-1.62 (m, 4H), 1.58-1.04 (m, 16H), 0.90 (t, *J* = 6.6 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 158.8, 140.8, 122.8, 104.8, 69.5, 31.9, 29.4, 29.2, 26.1, 22.7, 14.2.

N,N'-(2,5-dioctyloxy-1,4-phenylene)bisformamide (5c). Pale purple solid (0.86 g), 35% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 8.64 (d, *J* = 11.6 Hz, 1H), 8.40 (d, *J* = 1.6 Hz, 1H), 8.11 (d, *J* = 12.0 Hz, 1H), 7.78 (s, 2H), 6.72 (s, 1H), 4.11-3.91 (m, 4H), 1.88-1.72 (m, 4H), 1.57-1.10 (m, 20H), 0.89 (t, *J* = 6.8 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 158.7, 140.8, 122.8, 104.8, 69.5, 31.9, 29.5, 29.38, 29.36, 26.2, 22.8, 14.2.

N,N'-(2,5-dinonyloxy-1,4-phenylene)bisformamide (6c). Pale purple solid (0.71 g), 29% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 8.67 (d, *J* = 11.6 Hz, 1H), 8.43 (s, 1H), 8.14 (d, *J* = 11.6 Hz, 1H), 7.80 (s, 2H), 6.74 (s, 1H), 4.18-3.87 (m, 4H), 1.85-1.74 (m, 4H), 1.47-1.22 (m, 24H), 0.89 (t, *J* = 6.4 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 158.7, 140.8, 122.8, 104.7, 69.5, 32.0, 29.6, 29.5, 29.38, 29.35, 26.2, 22.8, 14.3.

N,N'-(2,5-didecyloxy-1,4-phenylene)bisformamide (7c). Pale purple solid (0.85 g), 34% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 8.68 (d, *J* = 11.6 Hz, 1H), 8.43 (d, *J* = 1.6 Hz, 1H), 8.15 (d, *J* = 11.6 Hz, 1H), 7.76 (d, *J* = 15.6 Hz, 2H), 6.74 (s, 1H), 4.08-3.92 (m, 4H), 1.89-1.70 (m, 4H), 1.47-1.24 (m, 28H), 0.88 (t, *J* = 6.8 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 158.7, 140.8, 122.8, 104.7, 69.5, 32.0, 29.7, 29.53, 29.46, 29.39, 29.36, 26.2, 22.8, 14.3.

Synthesis of compounds 1d~7d.

Synthesis of **1d**: a CH_2Cl_2 suspension (10 mL) of N,N'-(2,5-dibutoxy-1,4-phenylene)bisformamide (0.25 g, 0.81 mmol) and triethylamine (3 mL) was cooled to 0 °C. To the mixture was added dropwise a CH_2Cl_2 solution (2 mL) of triphosgene (0.53 g, 1.8 mmol). The mixture was refluxed for 3 h under an argon atmosphere. After cooling to room temperature, 10% aq. Na_2CO_3 (50 mL) was added dropwise. The mixture was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic phase was dried over anhydrous magnesium sulfate and the solvent was evaporated. The residue was subjected to column chromatography on silica gel (eluent:

petroleum ether/CH_2Cl_2 = 4:1) to give pure compound **1d**. Derivatives **2d~7d** were synthesized and purified according to the general procedure above.

1,4-Diisocyano-2,5-dibutoxybenzene (1d). White solid (0.18 g), 81% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 6.93 (s, 2H), 4.00 (t, *J* = 6.6 Hz, 4H), 1.90-1.76 (m, 4H), 1.57-1.46 (m, 4H), 0.99 (t, *J* = 7.2 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.0, 148.3, 117.0, 112.4, 69.8, 31.0, 19.1, 13.8.

1,4-Diisocyano-2,5-dipentyloxybenzene (2d). White solid (0.23 g), 86% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 6.92 (s, 2H), 3.99 (t, *J* = 6.6 Hz, 4H), 1.88-1.80 (m, 4H), 1.50-1.44 (m, 4H), 1.43-1.37 (m, 4H), 0.94 (t, *J* = 7.2 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.0, 148.4, 117.1, 112.5, 70.1, 28.7, 28.1, 22.5, 14.1.

1,4-Diisocyano-2,5-dihexyloxybenzene (3d). White solid (0.23 g), 85% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 6.90 (s, 2H), 3.97 (t, *J* = 6.4 Hz, 4H), 1.88-1.75 (m, 4H), 1.53-1.43 (m, 4H), 1.40-1.29 (m, 8H), 0.91 (t, *J* = 6.8 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.0, 148.4, 117.1, 112.5, 70.1, 31.5, 29.0, 25.6, 22.7, 14.1.

1,4-Diisocyano-2,5-diheptyloxybenzene (4d). White solid (0.24 g), 89% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 6.92 (s, 2H), 3.99 (t, *J* = 6.0 Hz, 4H), 1.96-1.76 (m, 4H), 1.56-1.14 (m, 16H), 0.90 (t, *J* = 6.0 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.0, 148.3, 117.0, 112.4, 70.1, 31.8, 29.00, 28.96, 25.9, 22.7, 14.1.

1,4-Diisocyano-2,5-dioctyloxybenzene (5d). White solid (0.24 g), 89% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 6.92 (s, 2H), 3.99 (t, *J* = 6.6 Hz, 4H), 1.93-1.74 (m, 4H), 1.54-1.18 (m, 20H), 0.89 (t, *J* = 6.6 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.0, 148.4, 117.1, 112.5, 70.1, 31.9, 29.34, 29.30, 29.0, 25.9, 22.8, 14.2.

1,4-Diisocyano-2,5-dinonyloxybenzene (6d). White solid (0.23 g), 83% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 6.92 (s, 2H), 3.99 (t, *J* = 6.6 Hz, 4H), 1.91-1.76 (m, 4H), 1.52-1.44 (m, 4H), 1.43-1.13 (m, 20H), 0.88 (t, *J* = 6.6 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.0, 148.4, 117.1, 112.5, 70.2, 32.0, 29.6, 29.40, 29.38, 29.0, 25.9, 22.8, 14.3.

1,4-Diisocyano-2,5-didecyloxybenzene (7d). White solid (0.22 g), 79% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 6.92 (s, 2H), 3.99 (t, *J* = 6.6 Hz, 4H), 1.91-1.76 (m, 4H), 1.52-1.44 (m, 4H), 1.39-1.20 (m, 24H), 0.88 (t, *J* = 6.6 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.0, 148.4, 117.1, 112.5, 70.2, 32.0, 29.7, 29.6, 29.43, 29.39, 29.0, 25.9, 22.8, 14.3.

Synthesis of compounds C4~C10.

Synthesis of **C4**: a mixture of $C_6F_5Au(tht)$ (0.34 g, 0.76 mmol) and 1,4-diisocyano-2,5-dibutoxybenzene (0.10 g, 0.37 mmol) was stirred in CH_2Cl_2 (20 mL) for overnight under an argon atmosphere at room temperature. The solvent was evaporated. Purification of the residue by recrystallization (CH_2Cl_2/hexane) gave pure compound **C4**. Derivatives **C4–C10** were synthesized and purified according to the general procedure above.

[μ-[1,4-di(isocyano-κ*C*)-2,5-dibutoxybenzene]]bis(2,3,4,5,6-pentafluorophenyl)digold(I) (C4). White solid (0.34 g), 92% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.18 (s, 2H), 4.12 (t, J = 6.6 Hz, 4H), 1.98-1.83 (m, 4H), 1.54-1.51 (m, 4H), 1.03 (t, J = 7.2 Hz, 6H). ^{19}F NMR ($CDCl_3$): δ (ppm) = -116.06, -156.92, -162.14. Anal. Calcd for $C_{28}H_{20}Au_2F_{10}N_2O_2$: C, 33.62; H, 2.02; N, 2.80. Found: C, 33.64; H, 2.06; N, 2.83.

[μ-[1,4-di(isocyano-κ*C*)-2,5-dipentyloxybenzene]]bis(2,3,4,5,6-pentafluorophenyl)digold(I) (C5). Pale yellow solid (0.47 g), 92% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.16 (s, 2H), 4.11 (t, J = 6.6 Hz, 4H), 2.01-1.83 (m, 4H), 1.53-1.41 (m, 8H), 0.97 (t, J = 7.2 Hz, 6H). ^{19}F NMR ($CDCl_3$): δ (ppm) = -115.99, -156.96, -162.20. Anal. Calcd for $C_{30}H_{24}Au_2F_{10}N_2O_2$: C, 35.04; H, 2.35; N, 2.72. Found: C, 35.07; H, 2.30; N, 2.73.

[μ-[1,4-di(isocyano-κ*C*)-2,5-dihexyloxybenzene]]bis(2,3,4,5,6-pentafluorophenyl)digold(I) (C6). Pale yellow solid (0.46 g), 91% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.16 (s, 2H), 4.11 (t, J = 6.6 Hz, 4H), 1.93-1.88 (m, 4H), 1.54-1.49 (m, 4H), 1.44-1.32 (m, 8H), 0.92 (t, J = 6.6 Hz, 6H). ^{19}F NMR ($CDCl_3$): δ (ppm) = -117.28, -157.88, -163.04. Anal. Calcd for $C_{32}H_{28}Au_2F_{10}N_2O_2$: C, 36.38; H, 2.67; N, 2.65. Found: C, 36.37; H, 2.68; N, 2.68.

[μ-[1,4-di(isocyano-κ*C*)-2,5-diheptyloxybenzene]]bis(2,3,4,5,6-pentafluorophenyl)digold(I) (C7). Pale yellow solid (0.45 g), 87% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.16 (s, 2H), 4.10 (t, J = 6.6 Hz, 4H), 2.02-1.80 (m, 4H), 1.54-1.48 (m, 4H), 1.44-1.29 (m, 12H), 0.88 (t, J = 6.6 Hz, 6H). ^{19}F NMR ($CDCl_3$): δ (ppm) = -116.06, -156.91, -162.17. Anal. Calcd for $C_{34}H_{32}Au_2F_{10}N_2O_2$: C, 37.65; H, 2.97; N, 2.58. Found: C, 37.65; H, 2.97; N, 2.59.

[μ-[1,4-di(isocyano-κ*C*)-2,5-dioctyloxybenzene]]bis(2,3,4,5,6-pentafluorophenyl)digold(I) (C8). Pale yellow solid (0.47 g), 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.16 (s, 2H), 4.11 (t, J = 6.4 Hz, 4H), 1.95-1.86 (m, 4H), 1.54-1.48 (m, 4H), 1.44-1.23 (m, 16H), 0.87 (t, J = 6.8 Hz, 6H). ^{19}F NMR ($CDCl_3$): δ (ppm) = -117.32, -157.85, -163.04. Anal. Calcd for $C_{36}H_{36}Au_2F_{10}N_2O_2$: C, 38.86; H, 3.26; N, 2.52. Found: C, 38.89; H, 3.29; N, 2.56.

[μ-[1,4-di(isocyano-κ*C*)-2,5-dinonyloxybenzene]]bis(2,3,4,5,6-pentafluorophenyl)digold(I)

(C9). Pale yellow solid (0.40 g), 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.16 (s, 2H), 4.10 (t, *J* = 6.4 Hz, 4H), 1.95-1.85 (m, 4H), 1.54-1.47 (m, 4H), 1.41-1.20 (m, 20H), 0.86 (t, *J* = 6.8 Hz, 6H). ^{19}F NMR ($CDCl_3$): δ (ppm) = -115.83, -156.94, -162.17. Anal. Calcd for $C_{38}H_{40}Au_2F_{10}N_2O_2$: C, 40.01; H, 3.53; N, 2.46. Found: C, 40.17; H, 3.56; N, 2.44.

[μ-[1,4-di(isocyano-*κC*)-2,5-didecyloxybenzene]]bis(2,3,4,5,6-pentafluorophenyl)digold(I) (C10). Pale yellow solid (0.25 g), 93% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.17 (s, 2H), 4.10 (t, *J* = 6.6 Hz, 4H), 1.96-1.83 (m, 4H), 1.54-1.47 (m, 4H), 1.43-1.36 (m, 4H), 1.33-1.20 (m, 20H), 0.86 (t, *J* = 6.6 Hz, 6H). ^{19}F NMR ($CDCl_3$): δ (ppm) = -115.99, -157.02, -162.19. Anal. Calcd for $C_{40}H_{44}Au_2F_{10}N_2O_2$: C, 41.11; H, 3.79; N, 2.40. Found: C, 41.19; H, 3.78; N, 2.45.

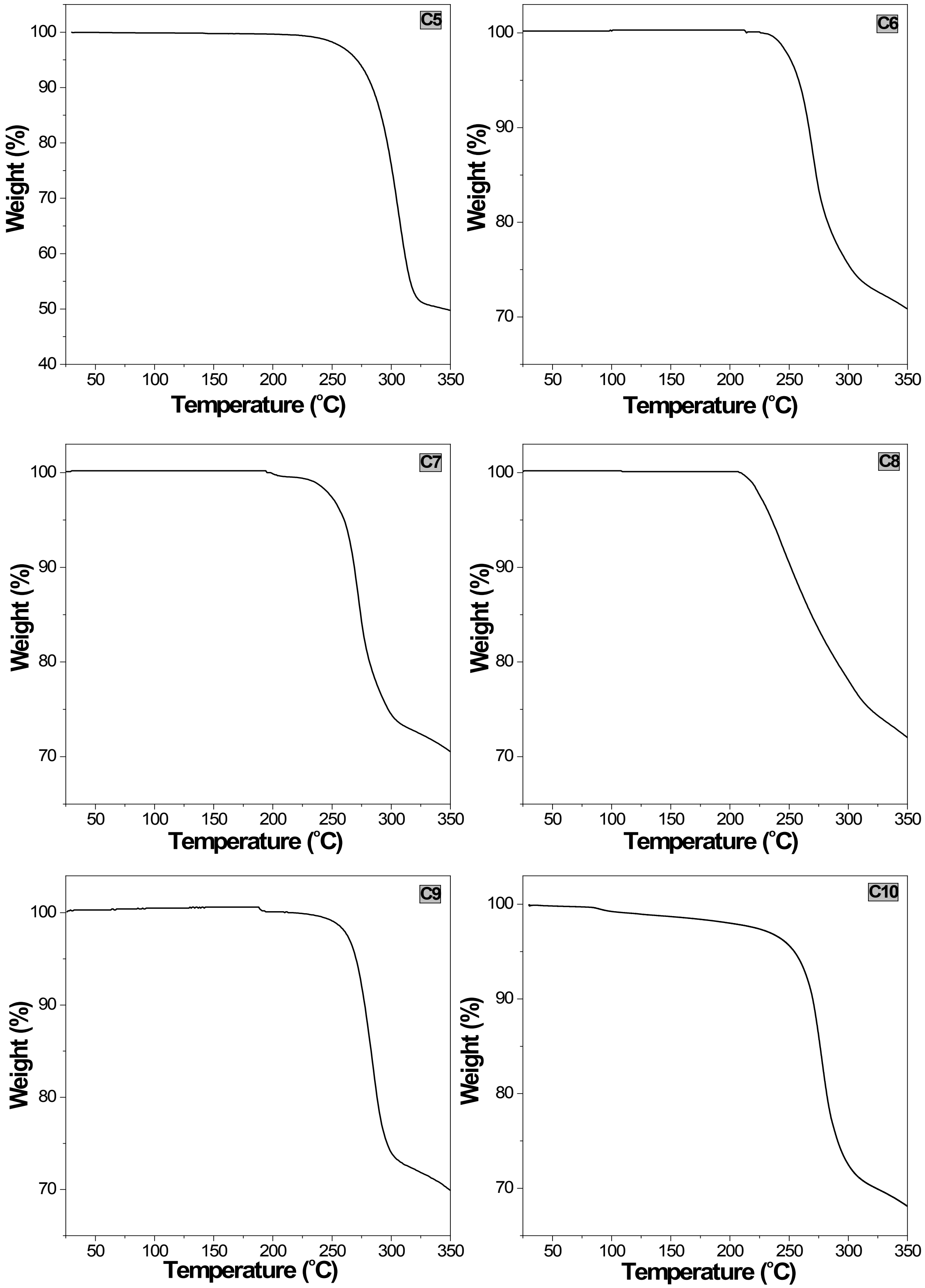


Fig. S1 TGA profiles of **C5~C10** at a heating rate of 10 °C min^{-1}. The thermal decomposition temperatures (T_d) corresponding to 5% weight loss under a nitrogen atmosphere are 271, 258, 260, 235, 271 and 254 °C for **C5~C10**, respectively.

Aggregation-induced emission (AIE) properties of AuIB-Cn

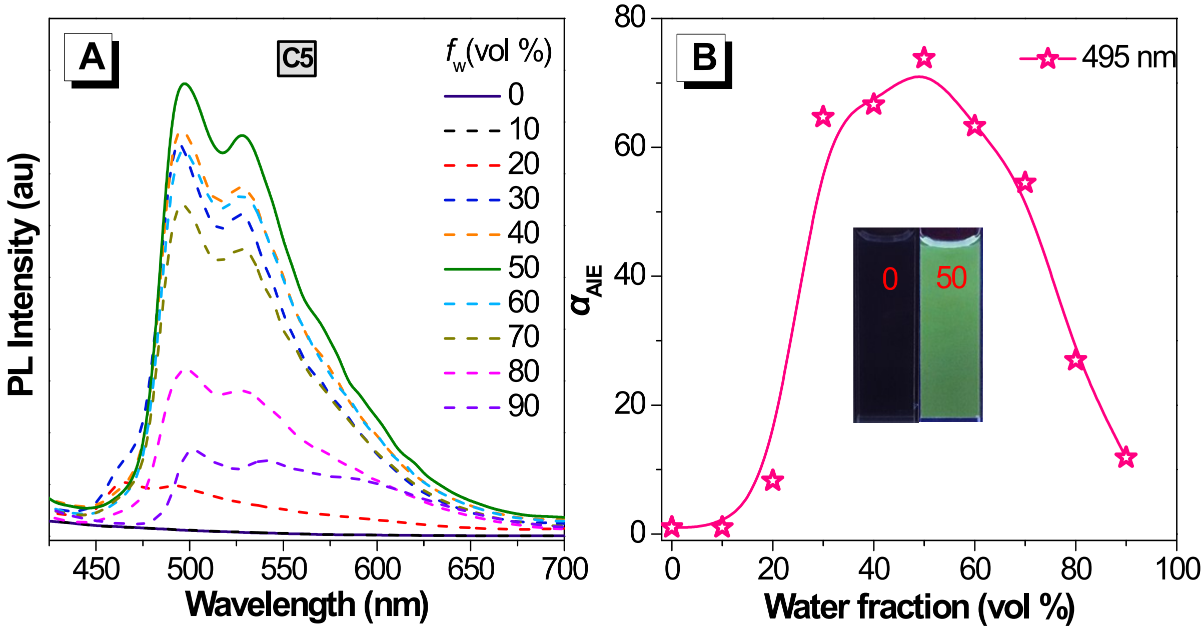


Fig. S2 (A) PL spectra of **C5** (c = 2.0 × 10^{-5} M) in DMF/water mixtures with different water fractions (f_w), λ_{ex} = 365 nm. (B) Plot of relative maximum emission peak intensity α_{AIE}) at 495 nm versus f_w of the DMF/water mixture, where α_{AIE} = I/I_0, I = emission intensity and I_0 = emission intensity in DMF solution. Inset: photos taken under 365 nm UV light of **C5** in DMF-water mixtures.

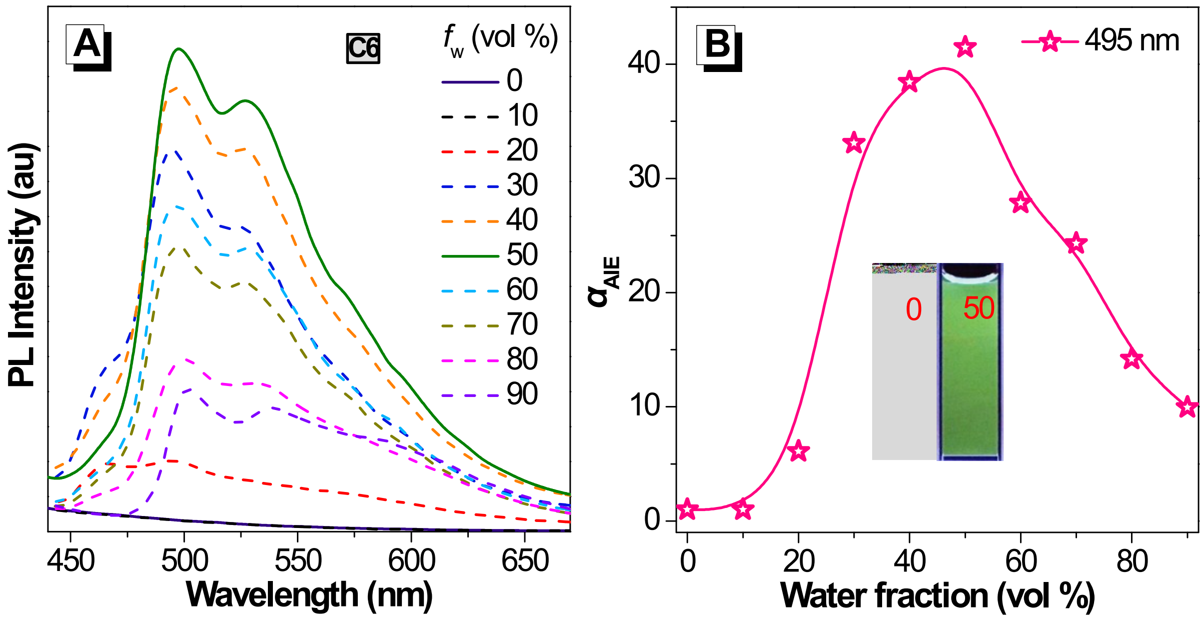


Fig. S3 (A) PL spectra of **C6** ($c = 2.0 \times 10^{-5}$ M) in DMF/water mixtures with different water fractions (f_w), λ_{ex} = 365 nm. (B) Plot of relative maximum emission peak intensity (α_{AIE}) at 495 nm versus f_w of the DMF/water mixture, where $\alpha_{AIE} = I/I_0$, I = emission intensity and I_0 = emission intensity in DMF solution. Inset: photos taken under 365 nm UV light of **C6** in DMF-water mixtures.

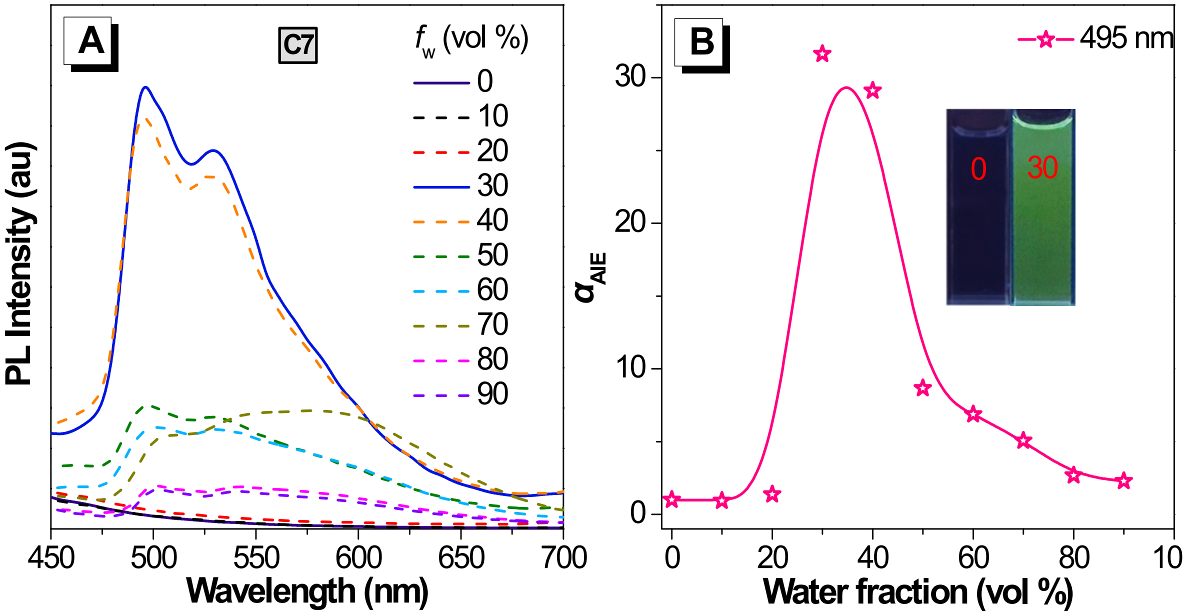


Fig. S4 (A) PL spectra of **C7** ($c = 2.0 \times 10^{-5}$ M) in DMF/water mixtures with different water fractions (f_w), λ_{ex} = 365 nm. (B) Plot of relative maximum emission peak intensity (α_{AIE}) at 495 nm versus f_w of the DMF/water mixture, where $\alpha_{AIE} = I/I_0$, I = emission intensity and I_0 = emission intensity in DMF solution. Inset: photos taken under 365 nm UV light of **C7** in DMF-water mixtures.

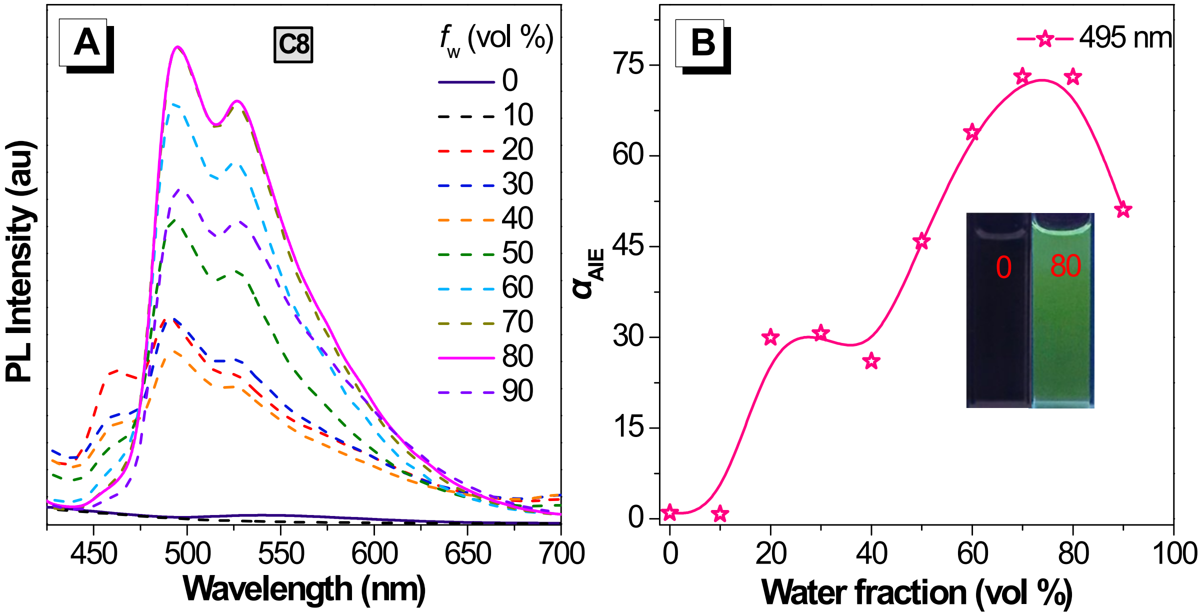


Fig. S5 (A) PL spectra of **C8** ($c = 2.0 \times 10^{-5}$ M) in DMF/water mixtures with different water fractions (f_w), λ_{ex} = 365 nm. (B) Plot of relative maximum emission peak intensity (α_{AIE}) at 495 nm versus f_w of the DMF/water mixture, where $\alpha_{AIE} = I/I_0$, I = emission intensity and I_0 = emission intensity in DMF solution. Inset: photos taken under 365 nm UV light of **C8** in DMF-water mixtures.

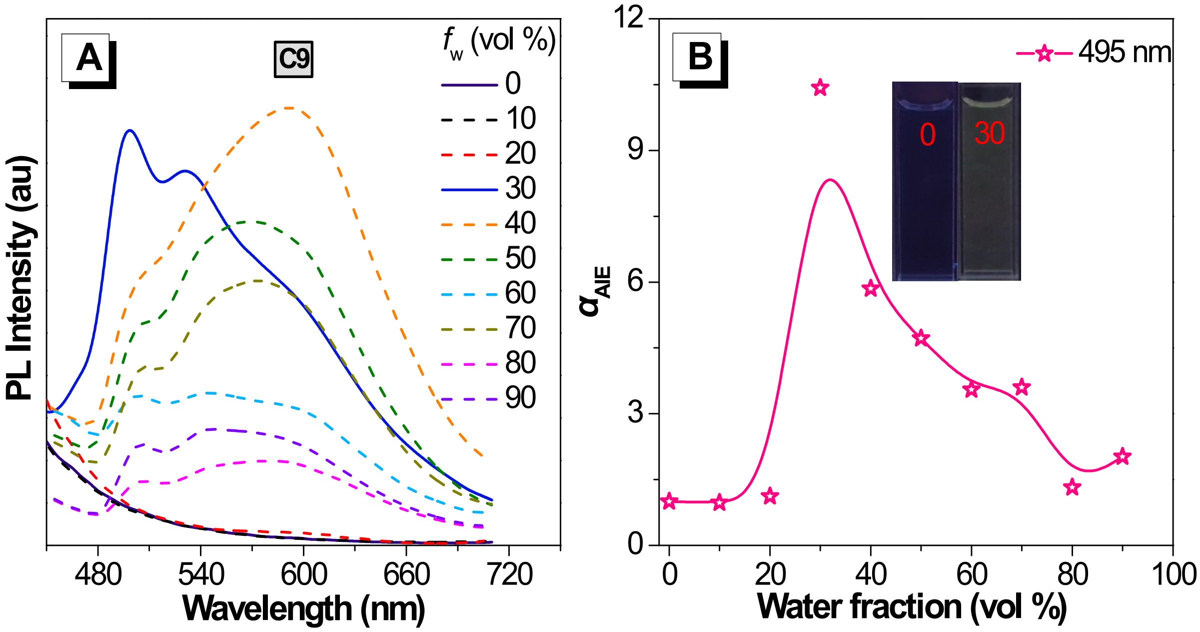


Fig. S6 (A) PL spectra of **C9** ($c = 2.0 \times 10^{-5}$ M) in DMF/water mixtures with different water fractions (f_w), $\lambda_{ex} = 365$ nm. (B) Plot of relative maximum emission peak intensity (α_{AIE}) at 495 nm versus f_w of the DMF/water mixture, where $\alpha_{AIE} = I/I_0$, I = emission intensity and I_0 = emission intensity in DMF solution. Inset: photos taken under 365 nm UV light of **C9** in DMF-water mixtures.

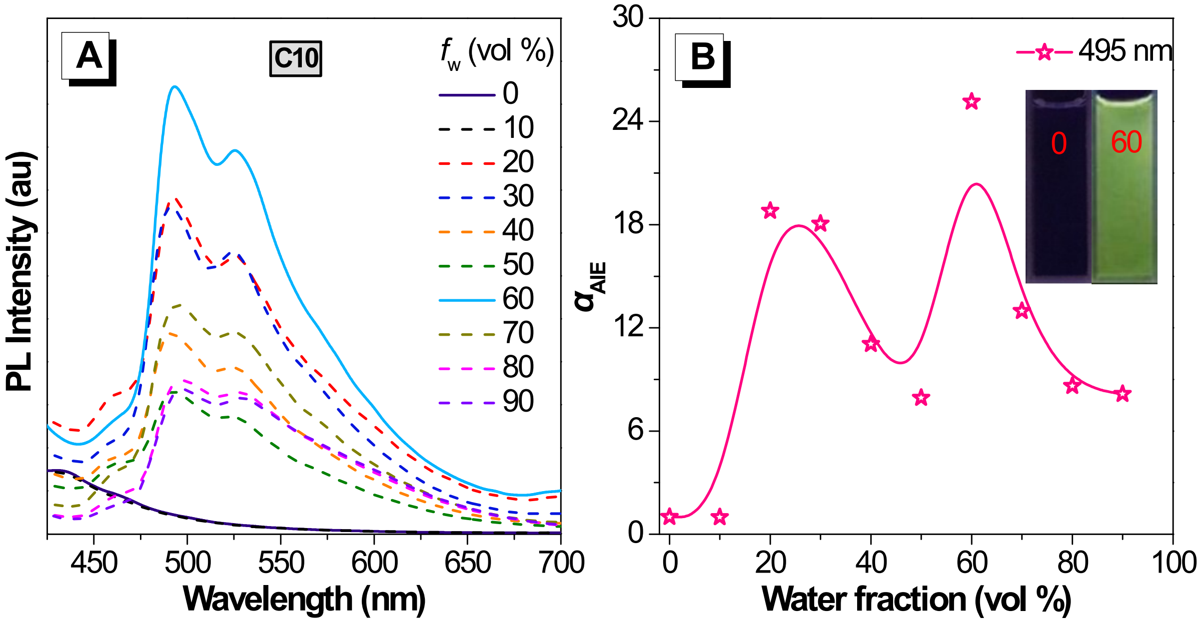


Fig. S7 (A) PL spectra of **C10** ($c = 2.0 \times 10^{-5}$ M) in DMF/water mixtures with different water fractions (f_w), λ_{ex} = 365 nm. (B) Plot of relative maximum emission peak intensity (α_{AIE}) at 495 nm versus f_w of the DMF/water mixture, where $\alpha_{AIE} = I/I_0$, I = emission intensity and I_0 = emission intensity in DMF solution. Inset: photos taken under 365 nm UV light of **C10** in DMF-water mixtures.

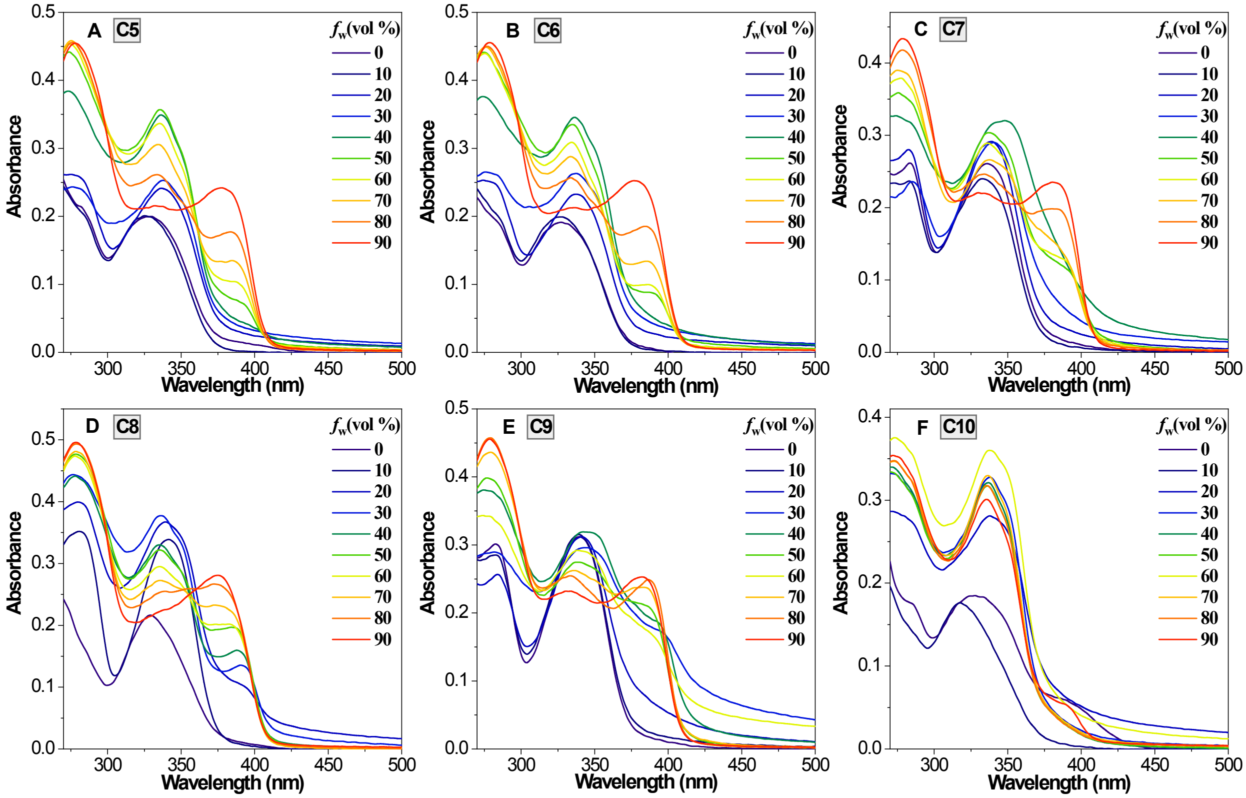


Fig. S8 UV-Vis spectra of **C5-C10** in DMF/water mixtures with different water fractions (f_w).

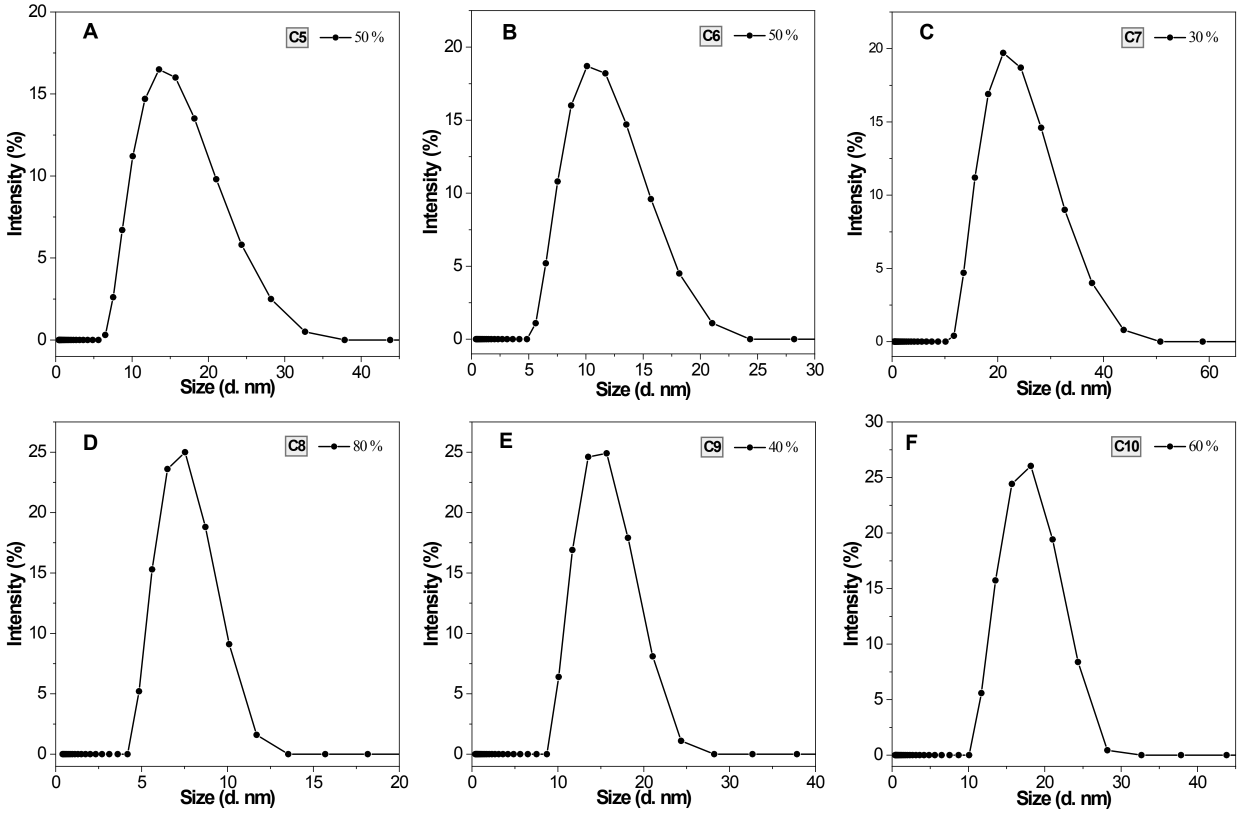


Fig. S9 Dynamic light scattering (DLS) curves of **C5-C10** in DMF/water mixtures with f_w = 50%, 50%, 30%, 80%, 40% and 60%.

Photophysical properties of AuIB-Cn solids

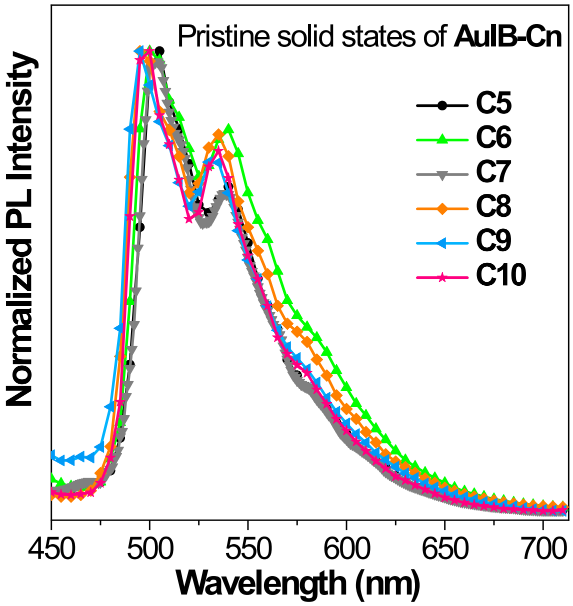


Fig. S10 Emission spectra of pristine **C5~C10** solids (λ_{ex} = 365 nm).

Table S1. Photophysical properties of **C4~C10** at solid states.

Complex		Pristine state		Ground state	
	Φ / %	τ_{av} / μs (λ_{em} / nm)		Φ / %	τ_{av} / μs (λ_{em} / nm)
C4	6	31.13 (505)	30.97 (540)	43	14.73 (595)
C5	5	18.37 (505)	16.00 (540)	43	6.59 (595)
C6	4	22.43 (500)	22.72 (540)	--[a]	--
C7	4	15.97 (505)	16.72 (540)	--	--
C8	68	201.68 (500)	184.05 (535)	--	--
C9	64	206.00 (500)	201.48 (535)	--	--
C10	16	127.47 (500)	124.61 (535)	--	--

[a] Because the self-recovery process is too fast, the data cannot be collected.

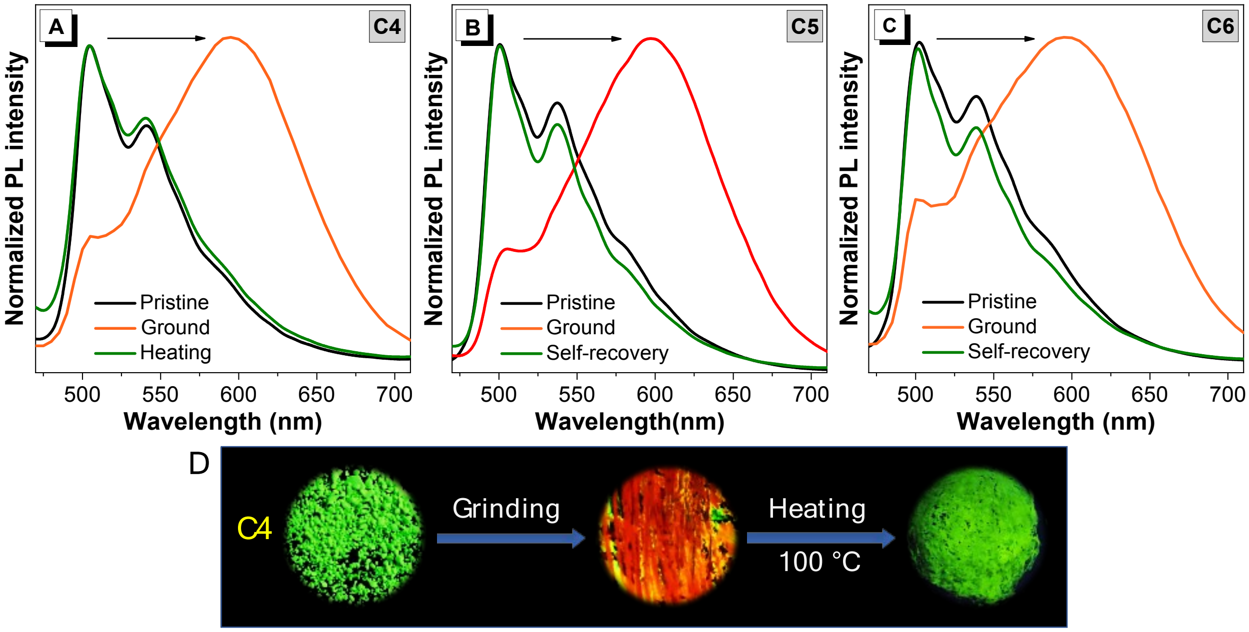


Fig. S11 Emission spectra of **C4** (A), **C5** (B) and **C6** (C) at different states. (D) photograph showing the emission color change of **C4** powder with grinding and after heating the ground powder.

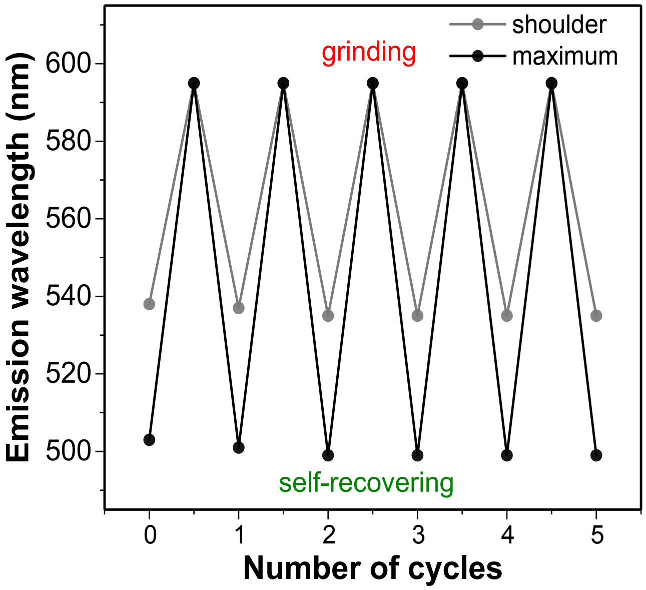


Fig. S12 Wavelength changes of the main emission peaks for **C5** during reversible grinding and self- recovering processes.

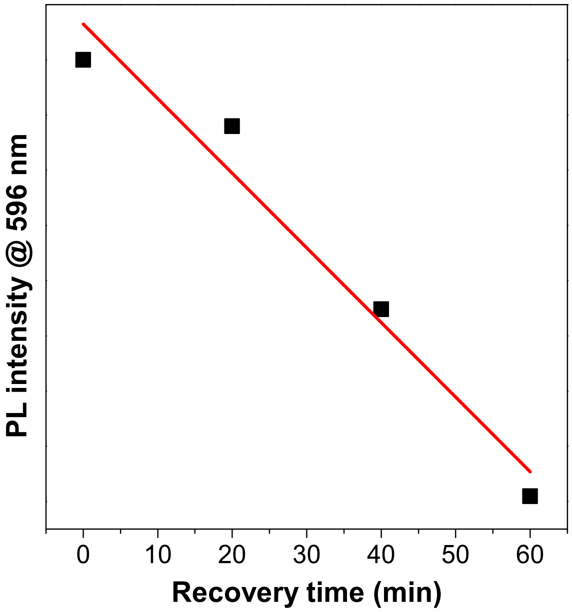


Fig. S13 Plot of PL intensity for **C5** at 596 nm after grinding with the corresponding recovery time (y = -0.01 x + 1.06, R^2 = 0.95).

Photographs of self-recovery process of C6~C10 at low temperature

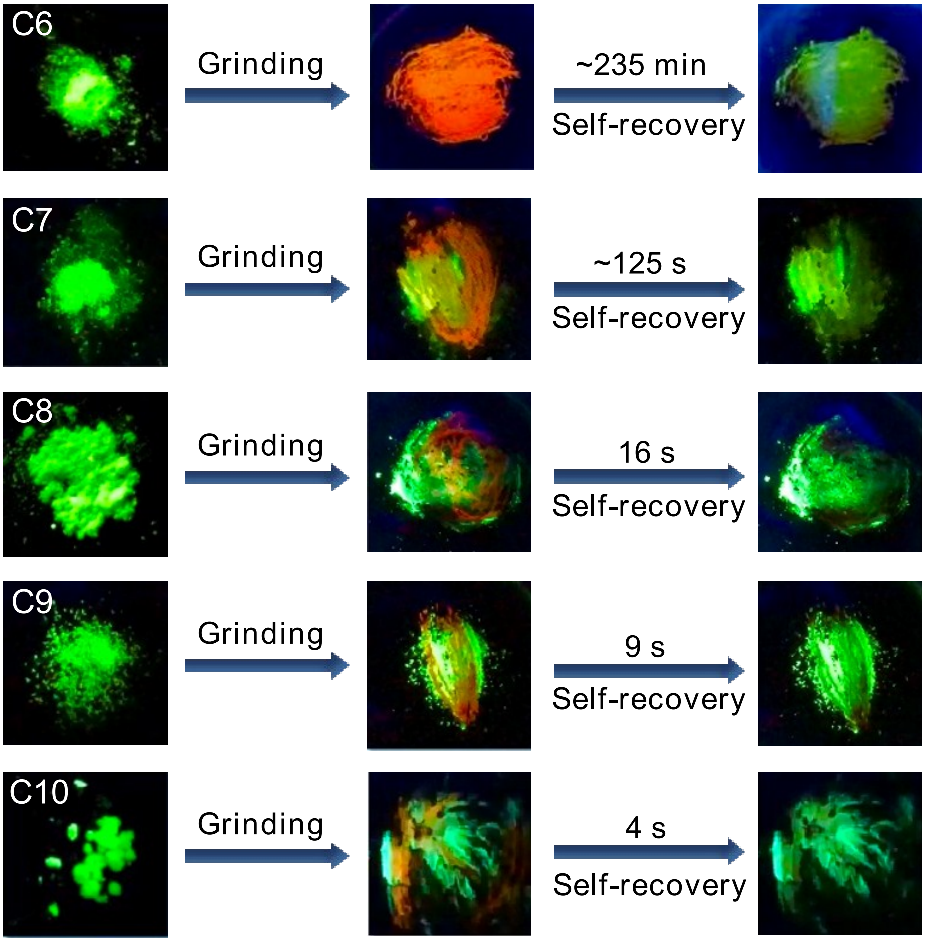


Fig. S14 Mechanochromic luminescence and corresponding self-recoveries of **C5~C10** powders at 8 °C.

PXRD analysis of C4~C6

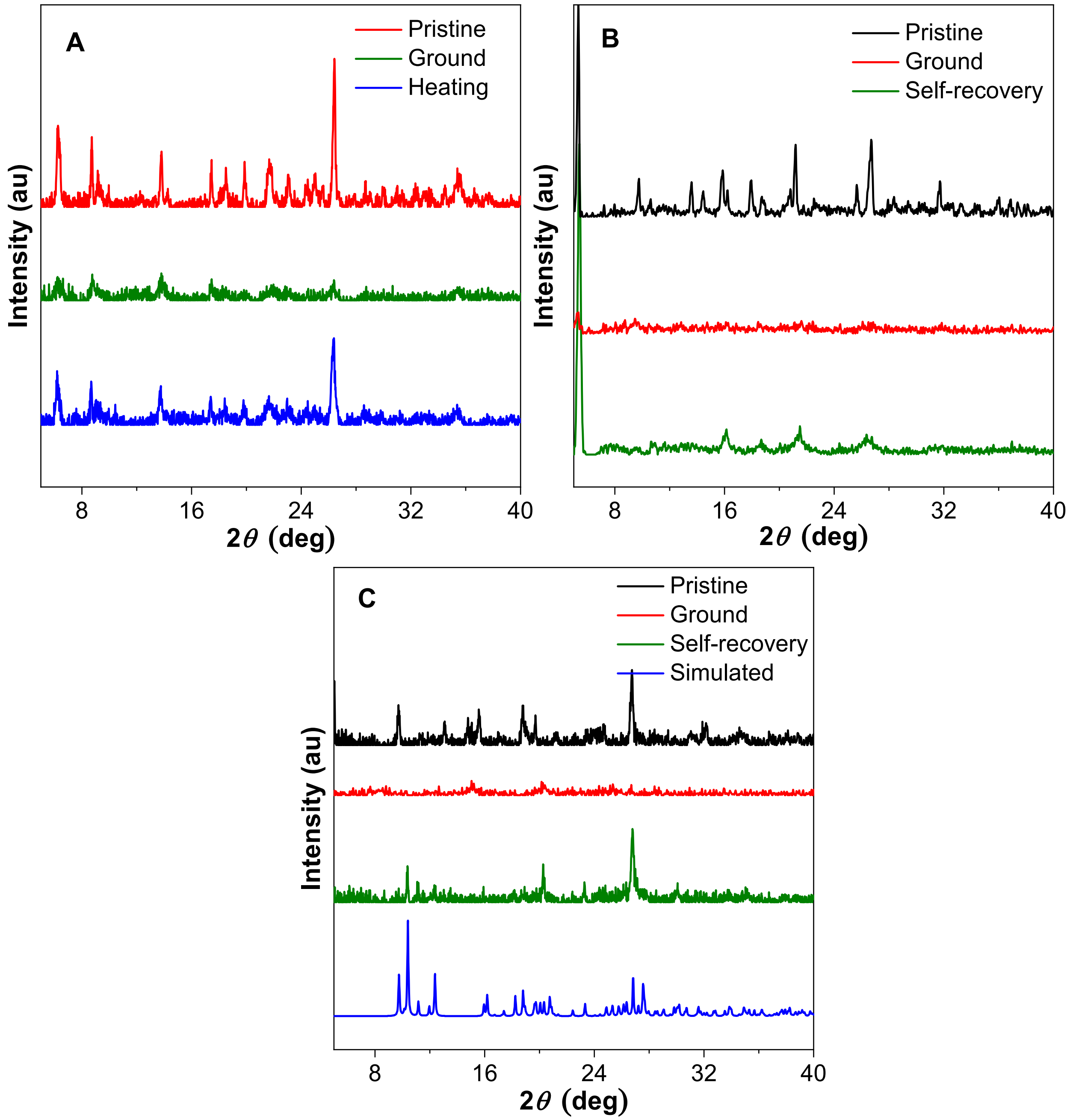


Fig. S15 PXRD patterns of **C4** (A), **C5** (B) and **C6** (C) powders at different states.

X-ray crystallographic analysis of AuIB-Cn

Table S2. Selected crystallographic data of **C6**.

Compound	**C6**
Formula	$C_{32}H_{28}Au_2F_{10}N_2O_2$
Formula weight	1056.50
Temperature (K)	298(2)
Crystal system	Triclinic
Space group	*P*-1
a (Å)	9.759(2)
b (Å)	10.239(2)
c (Å)	10.396(3)
α (deg)	64.173(3)
β (deg)	66.984(3)
γ (deg)	64.063(3)
V (Å^3)	814.7(3)
Z	2
Density$_{calcd}$ (Mg/m^3)	2.153
Absorption coefficient (mm^{-1})	9.084
F(000)	498
Theta range for data collection (deg)	2.247 to 29.000
Index ranges	$-13 \leq h \leq 13$, $-13 \leq k \leq 13$, $-14 \leq l \leq 14$
Reflections collected	7623
Independent reflections	4241 [*R*(int) = 0.0351]
Data / restraints / parameters	4241 / 0 / 218
Final *R* indices [*I*>2sigma(*I*)]	*R*1 = 0.0413, *wR*2 = 0.1156
R indices (all data)	*R*1 = 0.0496, *wR*2 = 0.1376
Goodness of fit on F^2	1.079
Largest diff. peak and hole (e Å^{-3})	2.897 and -1.879

Table S3. Selected crystallographic data of **C7**.

Compound	**C7**
Formula	$C_{34}H_{32}Au_2F_{10}N_2O_2$
Formula weight	1084.55
Temperature (K)	296(2)
Crystal system	Triclinic
Space group	*P*-1
a (Å)	8.8498(16)
b (Å)	9.9117(18)
c (Å)	10.5130(18)
α (deg)	93.537(3)
β (deg)	96.872(3)
γ (deg)	95.434(3)
V (Å^3)	908.9(3)
Z	1
Density$_{calcd}$ (Mg/m^3)	1.981
Absorption coefficient (mm^{-1})	8.145
F(000)	514
Theta range for data collection (deg)	1.956 to 25.997
Index ranges	-10≤h≤10, -12≤k≤12, -11≤l≤12
Reflections collected	6948
Independent reflections	3522 [*R*(int) = 0.0259]
Data / restraints / parameters	3522 / 122 / 301
Final *R* indices [*I*>2sigma(*I*)]	*R*1 = 0.0329, *wR*2 = 0.0933
R indices (all data)	*R*1 = 0.0458, *wR*2 = 0.1165
Goodness of fit on F^2	1.082
Largest diff. peak and hole (e Å^{-3})	0.869 and -0.677

Table S4. Selected crystallographic data of **C8**.

Compound	**C8**
Formula	$C_{36}H_{36}Au_2F_{10}N_2O_2$
Formula weight	1112.60
Temperature (K)	100(2)
Crystal system	Triclinic
Space group	*P*-1
a (Å)	8.3142(7)
b (Å)	10.4543(8)
c (Å)	10.7962(9)
α (deg)	101.9030(10)
β (deg)	99.0170(10)
γ (deg)	92.6020(10)
V (Å^3)	903.93(13)
Z	1
Density$_{calcd}$ (Mg/m^3)	2.044
Absorption coefficient (mm^{-1})	8.193
F(000)	530
Theta range for data collection (deg)	1.96 to 26.00
Index ranges	$-10\leq h\leq 10$, $-12\leq k\leq 12$, $-9\leq l\leq 13$
Reflections collected	5396
Independent reflections	3487 [*R*(int) = 0.0197]
Data / restraints / parameters	3487 / 18 / 236
Final *R* indices [*I*>2sigma(*I*)]	*R*1 = 0.0245, *wR*2 = 0.0846
R indices (all data)	*R*1 = 0.0311, *wR*2 = 0.1352
Goodness of fit on F^2	1.219
Largest diff. peak and hole (e Å^{-3})	2.578 and -3.503

Table S5. Selected crystallographic data of **C9**.

Compound	**C9**
Formula	$C_{38}H_{40}Au_2F_{10}N_2O_2$
Formula weight	1140.65
Temperature (K)	294(2)
Crystal system	Triclinic
Space group	*P*-1
a (Å)	8.828(3)
b (Å)	10.637(3)
c (Å)	11.035(3)
α (deg)	104.086(4)
β (deg)	94.677(4)
γ (deg)	95.957(4)
V (Å^3)	993.5(5)
Z	1
Density$_{calcd}$ (Mg/m^3)	1.907
Absorption coefficient (mm^{-1})	7.457
F(000)	546
Theta range for data collection (deg)	1.914 to 25.491
Index ranges	$-10 \le h \le 10$, $-12 \le k \le 12$, $-13 \le l \le 12$
Reflections collected	7094
Independent reflections	3650 [*R*(int) = 0.0478]
Data / restraints / parameters	3650 / 49 / 246
Final R indices [I>2sigma(I)]	*R*1 = 0.0529, *wR*2 = 0.1410
R indices (all data)	*R*1 = 0.0674, *wR*2 = 0.1502
Goodness of fit on F^2	1.018
Largest diff. peak and hole (e Å^{-3})	1.536 and -1.163

Table S6. Selected crystallographic data of **C10**.

Compound	**C10**
Formula	$C_{40}H_{44}Au_2F_{10}N_2O_2$
Formula weight	1168.72
Temperature (K)	293(2)
Crystal system	Triclinic
Space group	*P*-1
a (Å)	9.024
b (Å)	10.678
c (Å)	11.524
α (deg)	107.92
β (deg)	98.21
γ (deg)	93.26
V (Å^3)	1039.8
Z	2
Density$_{calcd}$ (Mg/m^3)	1.860
Absorption coefficient (mm^{-1})	7.127
F(000)	558
Theta range for data collection (deg)	1.88 to 27.00
Index ranges	$-11\leq h\leq 11$, $-13\leq k\leq 13$, $-14\leq l\leq 14$
Reflections collected	11806
Independent reflections	4525 [*R*(int) = 0.0212]
Data / restraints / parameters	4525 / 0 / 281
Final R indices [*I*>2sigma(*I*)]	*R*1 = 0.0275, *wR*2 = 0.0695
R indices (all data)	*R*1 = 0.0341, *wR*2 = 0.0718
Goodness of fit on F^2	1.050
Largest diff. peak and hole (e Å^{-3})	0.821 and -0.314

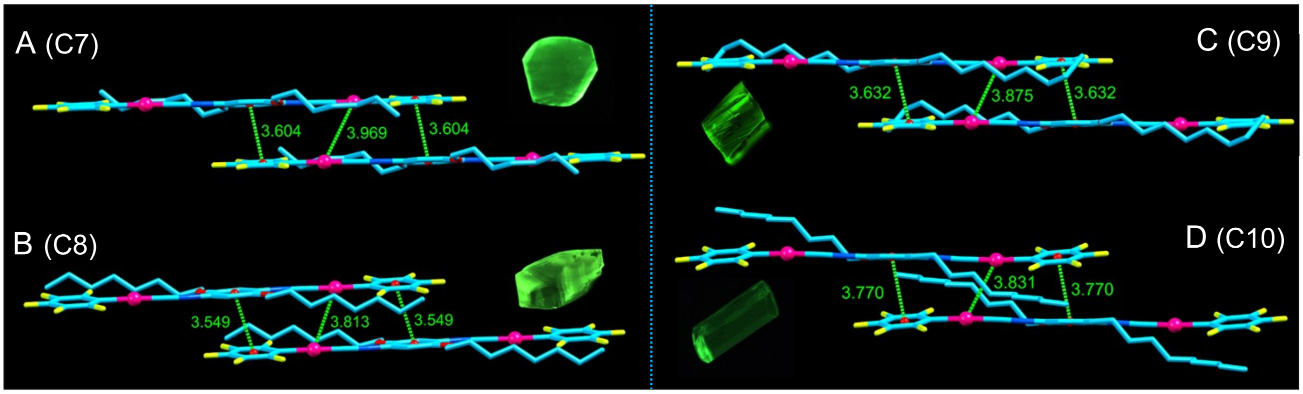


Fig. S16 Intermolecular interactions of **C7~C10**. Insert: photos of corresponding crystal taken under 365 nm UV light.

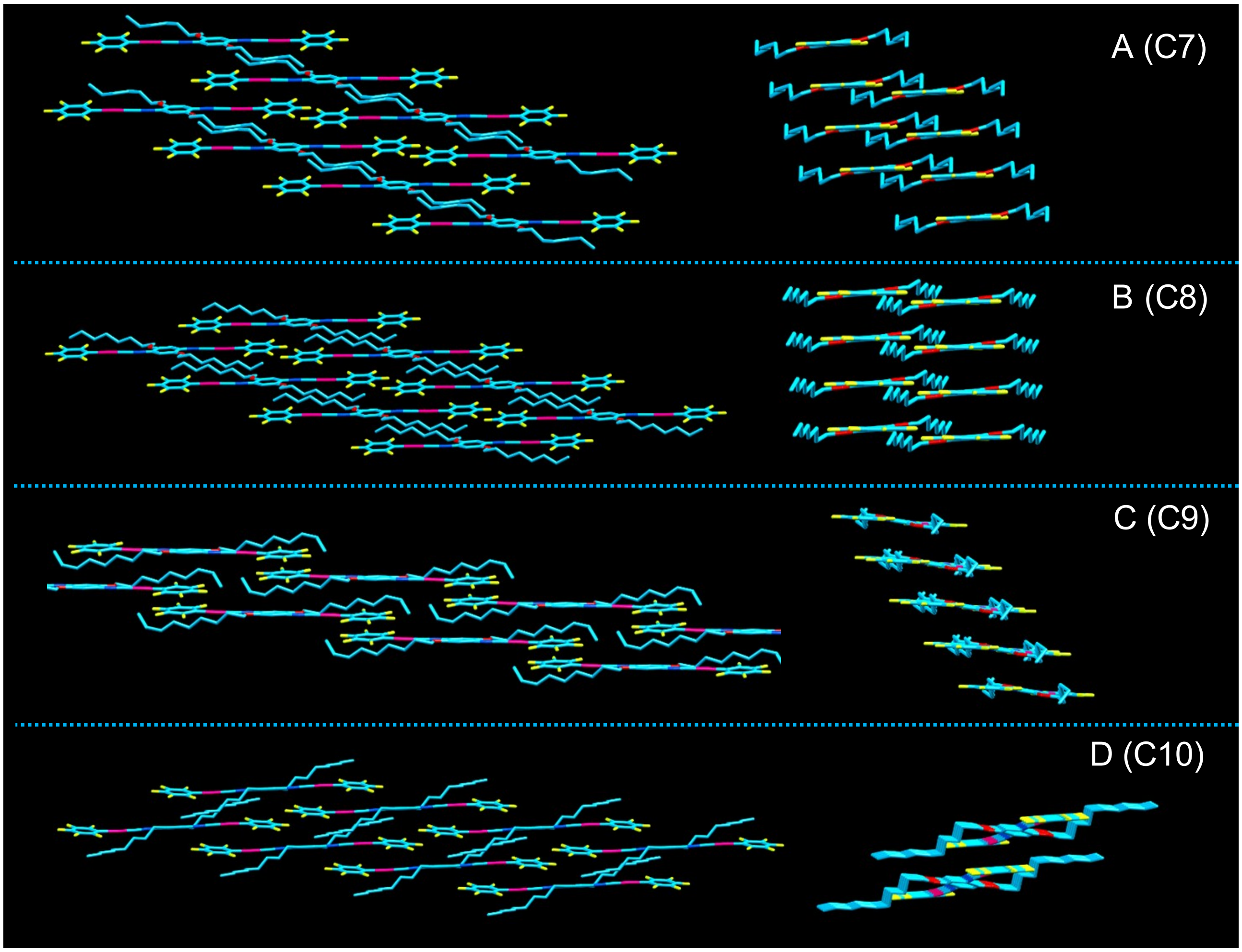


Fig. S17 Molecular packings of **C7~C10**.

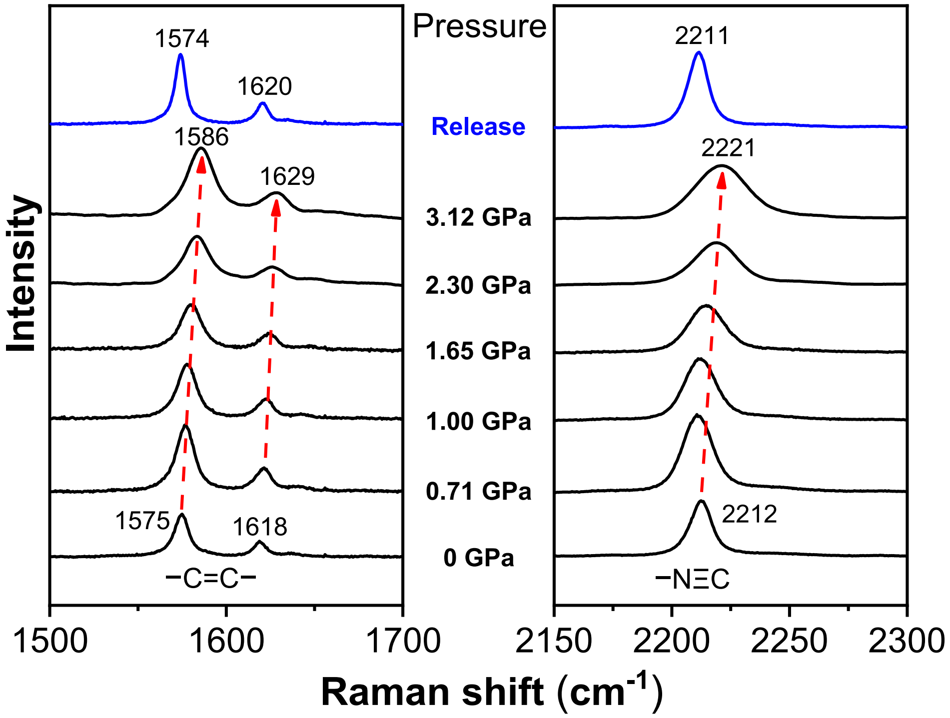


Fig. S18 Raman spectra of **C6** recorded in the region of ν(–C=C–) and ν(N≡C) stretching modes under hydrostatic pressure at room temperature.

Raman spectra were recorded in the range of 550–2500 cm^{-1} at various pressures structural change during the above piezochromic luminescent process. As shown in Fig. S15, the peaks observed at 1618 and 2212 cm^{-1} could be assigned to the aromatic olefins bond stretching vibration (–C=C–) and cyano group stretching vibration (N≡C), respectively. The relatively stronger peaks located at 1575 cm^{-1} was assigned to the vibrations of phenyl rings. The aromatic C=C and N≡C stretching vibrations showed gradual blue-shift with the increase of pressure from 1 atm to 3.12 GPa, indicative of shortened bond lengths and decreased intermolecular distances caused by the enhanced intermolecular interactions including aurophilic bonding. When the pressure was released, the Raman profile completely recovered to the initial state.

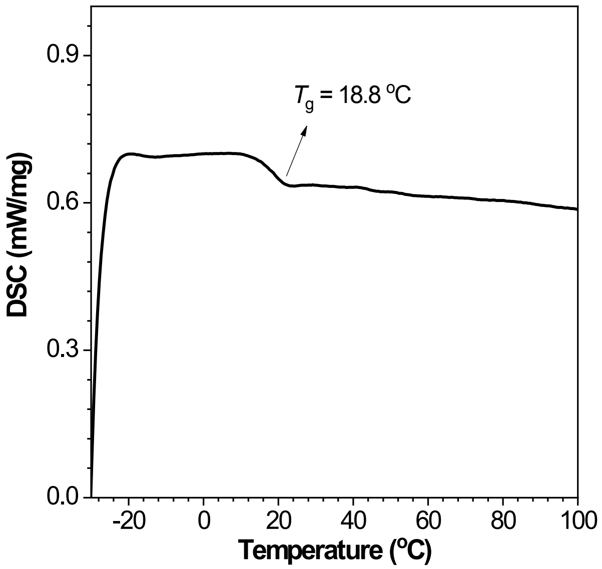


Fig. S19 DSC curve of poly(β-aminoacrylate).

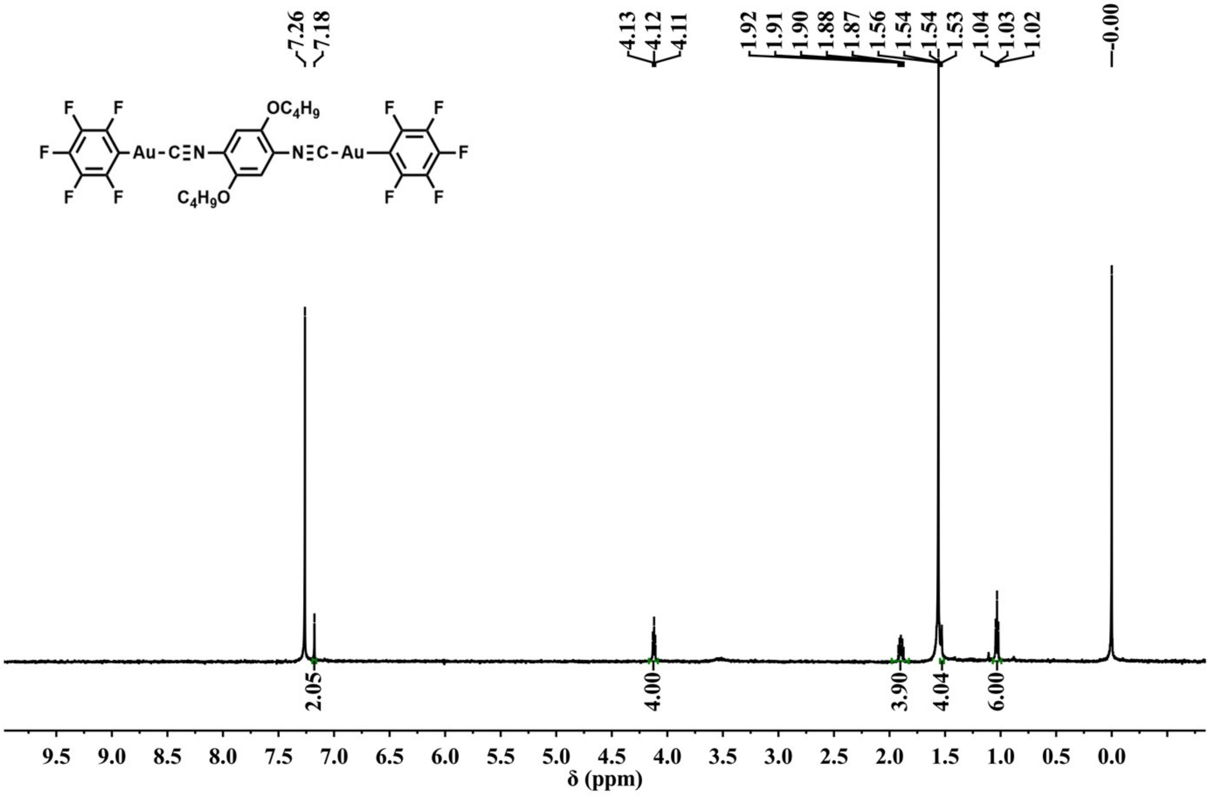


Fig. S20 ^{1}H NMR spectra of **C4** in $CDCl_3$ at room temperature.

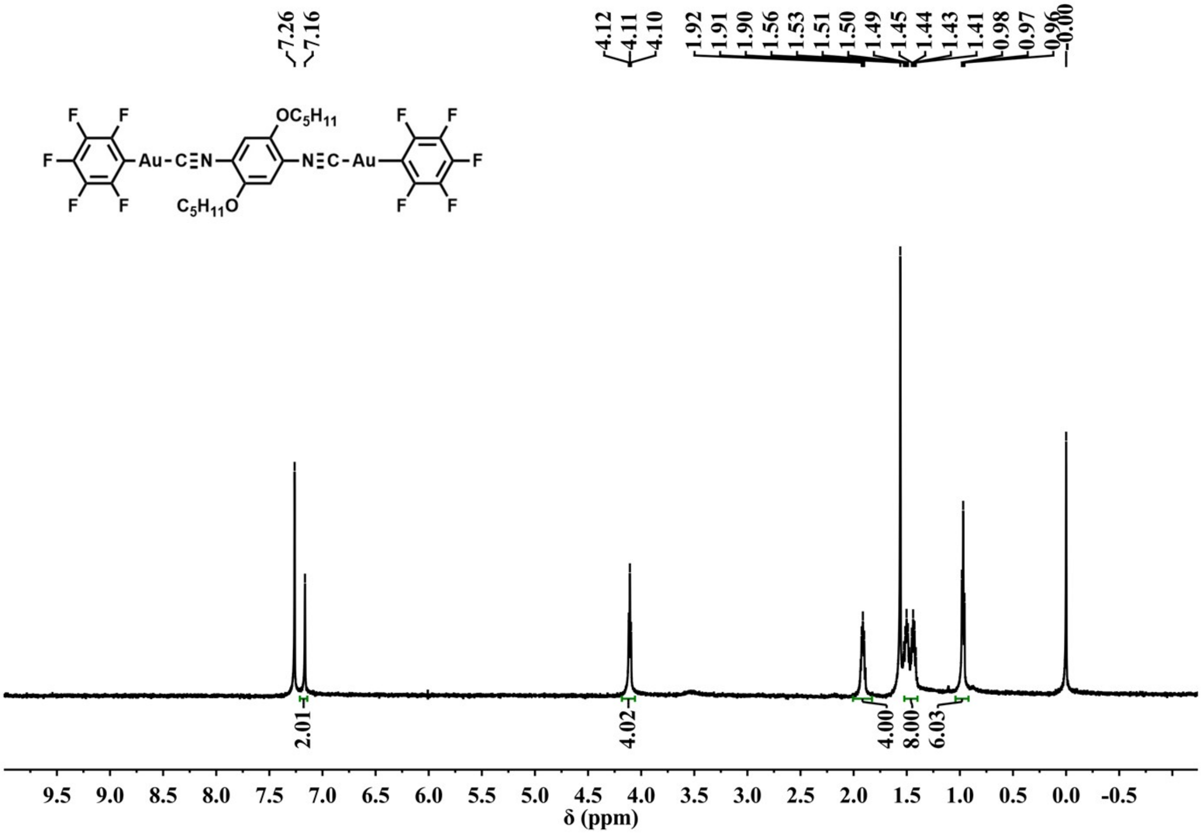


Fig. S21 ^{1}H NMR spectra of **C5** in $CDCl_3$ at room temperature.

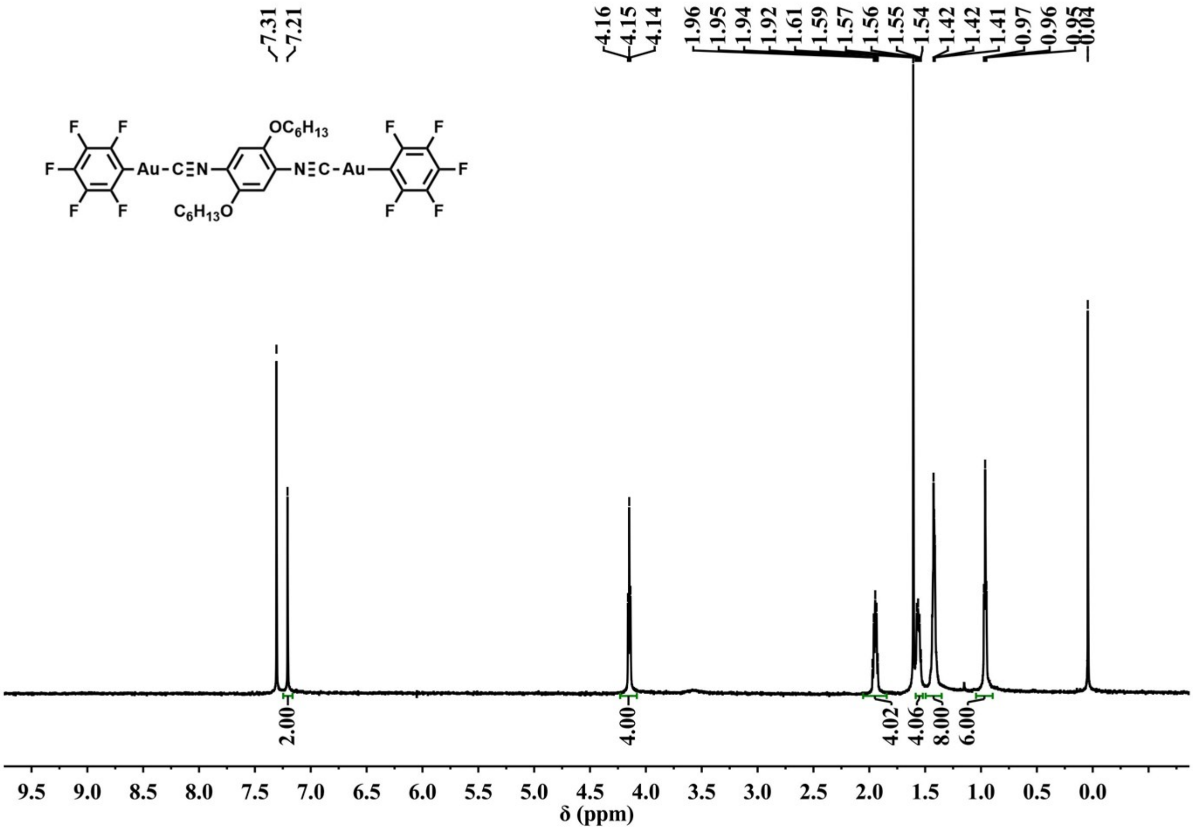


Fig. S22 ^{1}H NMR spectra of **C6** in $CDCl_3$ at room temperature.

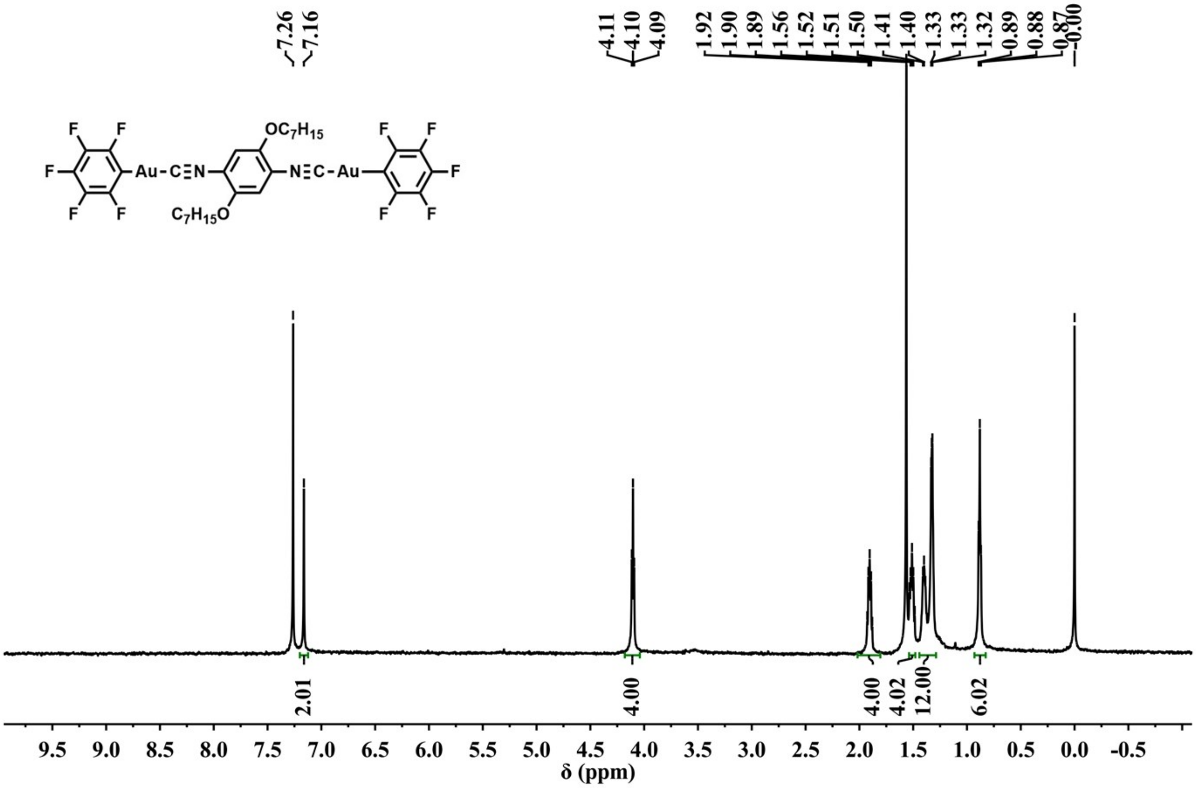


Fig. S23 ^{1}H NMR spectra of **C7** in $CDCl_3$ at room temperature.

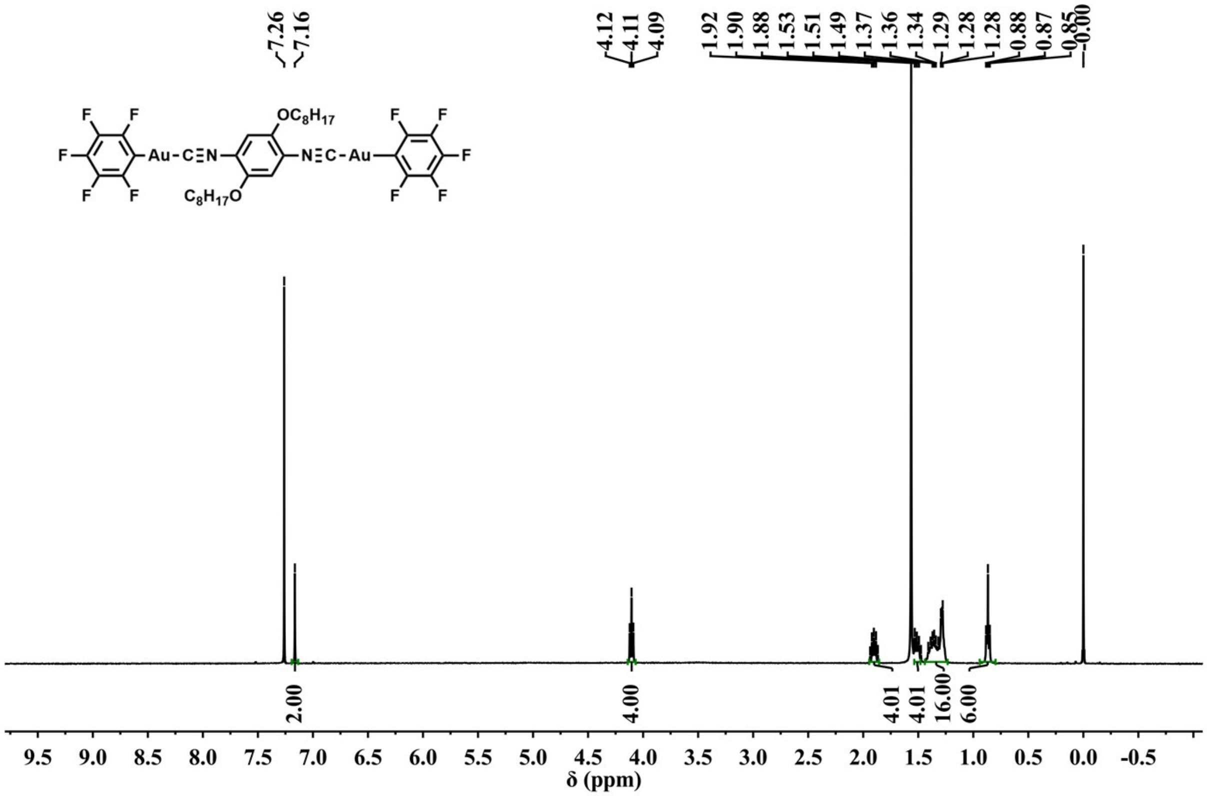


Fig. S24 ^{1}H NMR spectra of **C8** in $CDCl_3$ at room temperature.

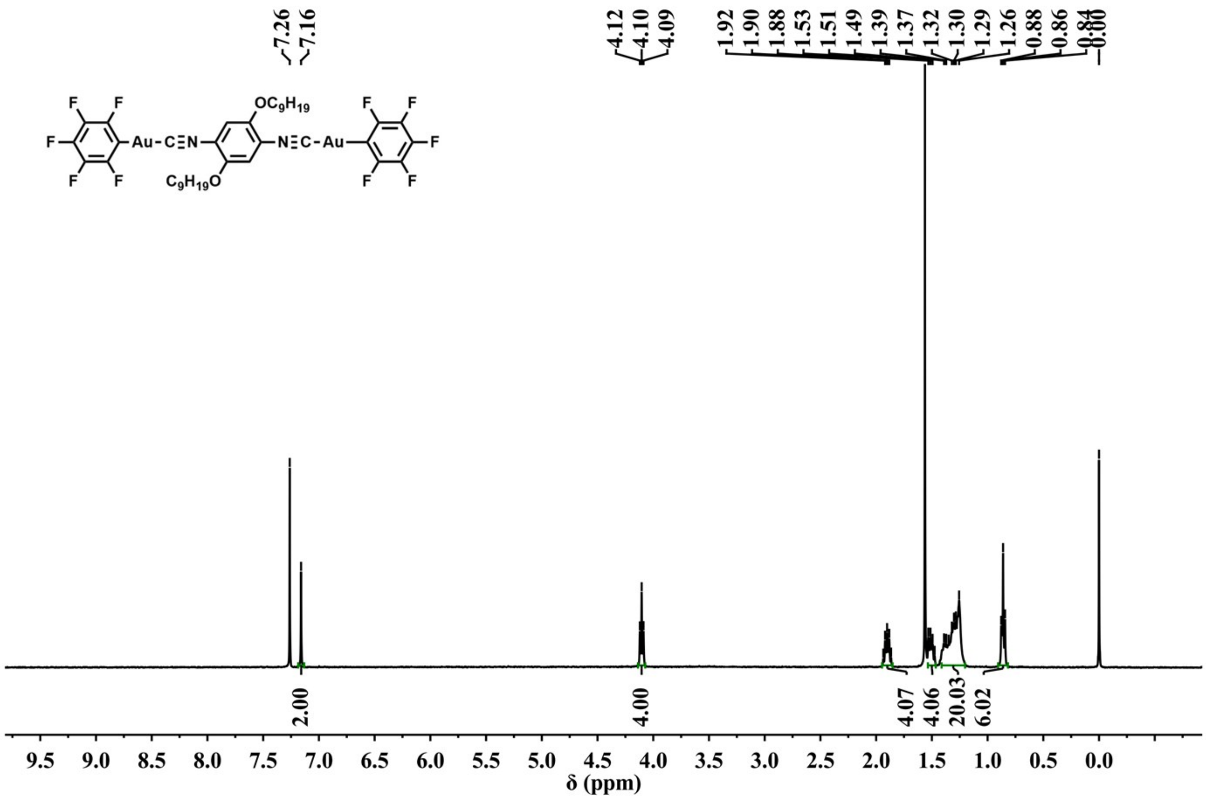


Fig. S25 ^{1}H NMR spectra of **C9** in $CDCl_3$ at room temperature.

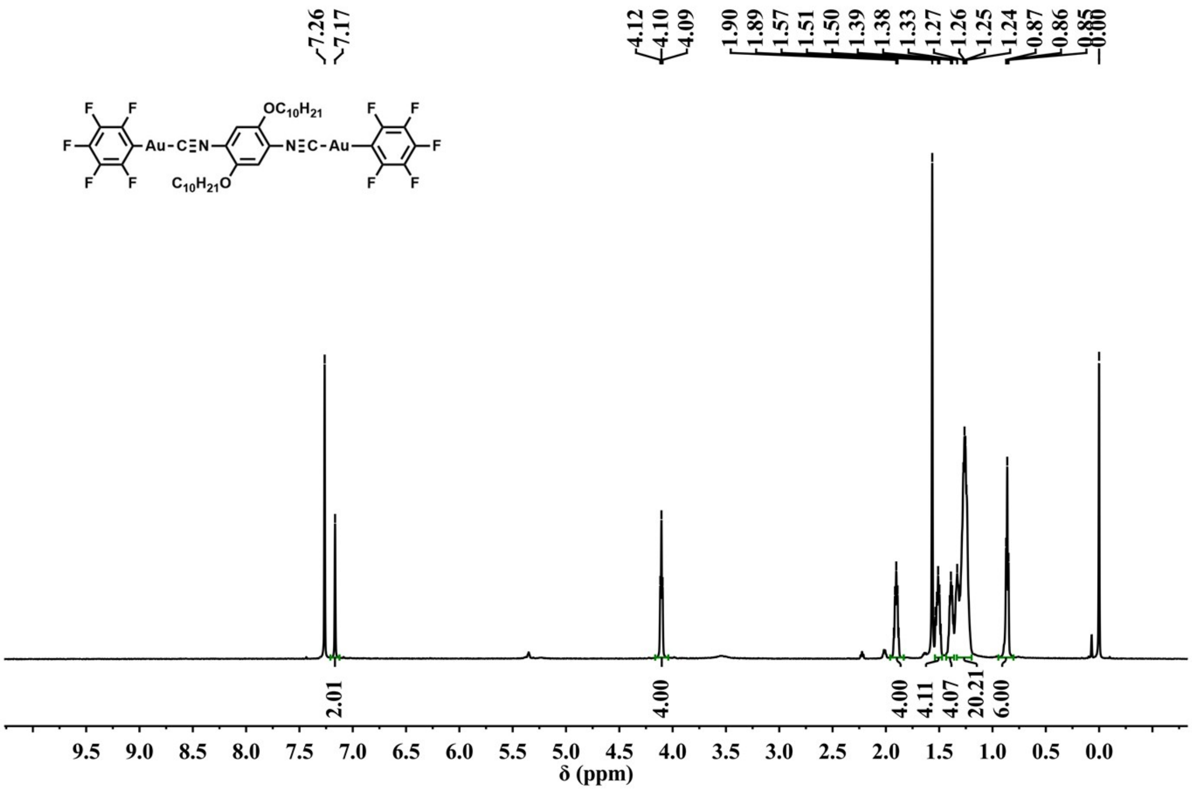


Fig. S26 ^{1}H NMR spectra of **C10** in $CDCl_3$ at room temperature.

References

1. J. Li, F. Li, X. Jiang, G. Wei, Y. Cheng, C. Zhu, *Macromol. Rapid Commun.* **2013**, *34*, 1312.
2. Y. Liu, R. Gao, Y. Hao, X. Sun, A. Ouyang, *Chinese J. Polym. Sci.* **2012**, *30*, 443.
3. M. V. Ivanov, V. J. Chebny, M. R. Talipov, R. Rathore, *J. Am. Chem. Soc.* **2017**, *139*, 4334.
4. A. M. E. Daniel, Christoph Ulbricht, Thomas Orgis, Benjamin Carbonnier, Thomas Kietzke, Micha Peip, Manuela Metzner, Martin Gericke, Eckhard Birckner, Tadeusz Pakula, *Chem. Mater.* **2005**, *17*, 6022.
5. M. Shellaiah, M. Ramakrishnamraju, A. Singh, H. C. Lin, K. H. Wei, H. C. Lin, *J. Mater. Chem. A* **2014**, *2*, 17463.
6. R. Singh, J. S. Meena, C. S. Wu, F. H. Ko, *Phys. Chem. Chem. Phys.* **2015**, *17*, 5227.
7. K. Shopsowitz, F. Lelj, M. J. Maclachlan, *J. Org. Chem.* **2011**, *76*, 1285.
8. O. A. Blackburn, B. J. Coe, R. Futhey, M. Helliwell, *Acta Cryst.* **2010**, *66*, o36.
9. J. Wu, F. Fang, Wenya Lu, Junli Hou, Chuang Li, Zongquan Wu, Xikui Jiang, A. Zhanting

Li, Yihua Yu, *J. Org. Chem.* **2007**, *72*, 2897.

10. R. Uson, A. Laguna, M. Laguna, *Inorg. Synth.* **1989**, *26*, 85.
11. G. J. Piermarini, S. Block, *Rev. Sci. Instrum.* **1975**, *46*, 973.
12. Y. K. Vohra, C. A. Vanderborgh, S. Desgreniers, A. L. Ruoff, *Phys. Rev. B* **1990**, *42*, 9189.
13. H. Li, H. Wang, D. Huang, L. Liang, Y. Gu, C. Liang, S. Xu, W. Xu, *Rev. Sci. Instrum.* **2014**, *85*, 301.