

Reversal Aggregation-Induced Circular Dichroism from Axial Chirality Transfer via Self-Assembled Helical Nanowires

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ESI 1. Synthesis procedures of the compounds

Synthesis of 3,5-Dimethylpyrrole-2-carbaldehyde

The compound was synthesized using a similar method to that reported in the literature. Freshly distilled POCl₃ (5.1 mL, 0.055 mol) was added dropwise to anhydrous DMF (4.3 mL, 0.055 mol) at 0 °C. The resulting solution was stirred for 15 min after the ice-bath was removed. Dry 1,2-dichloroethane (10 mL) was then added, and the solution was cooled to 0-5 °C. 2,4-Dimethylpyrrole (4.78 g, 0.05 mol) dissolved in dry 1,2-dichloroethane (10 mL) was added. Following the addition, the yellow mixture was heated to reflux for 15 min before being cooled to room temperature. Aqueous sodium acetate (45 g in 100 mL water) was then added (slowly at first then as rapidly as possible). The mixture was again refluxed for 15 min with vigorous stirring. The layers were separated, and the aqueous layer was washed three times with dichloromethane. The combined organic layer was washed three times with saturated brine, dried over Na₂SO₄, and evaporated under vacuum to give an oily liquid. The crude product was purified via chromatography on Al₂O₃ (petroleum ether/ethyl acetate, 4/1, v/v), to yield light yellow crystals (10.2 g, 82%). ¹H NMR (300 MHz, CDCl₃) δ 10.27 (s, 1H), 9.44 (s, 1H), 5.83 (s, 1H), 2.30 (s, 3H), 2.29 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 175.89, 138.74, 134.86, 128.75, 112.01, 13.13, 10.58.

Synthesis of 3

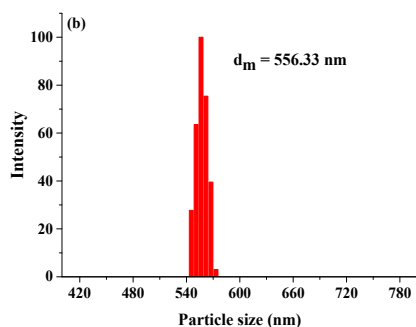
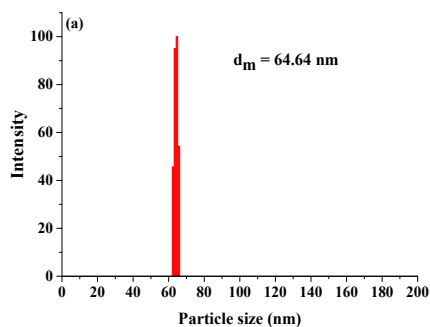
To 20 mL ethanol in a 100 ml round-bottomed flask were added 3,5-dimethylpyrrole-2-carbaldehyde (2.33g, 18.9 mmol) and 80% hydrazine (570 μL, 9.4 mmol) and a few drops of acetic acid. The reaction mixture turned to yellow, and yellow precipitates were observed. It was left stir at room temperature, and TLC was used to follow the reaction. The yellow precipitates were collected by filtration and washed with small amount of cold alcohol, and then dried at room temperature to give **2** as yellow powder, and can be directly used for the synthesis of the compound **3** without further purification. **2** (1.21 g, 10.0 mmol), 12 mL of triethylamine were added in 25 mL

toluene and the resulting mixture was stirred for 10 min. Then $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (15 mL) was added by syringe and the mixture was heated to reflux overnight. After cooling to room temperature, the residue was concentrated to dryness under reduced pressure and the product was purified by column chromatography on silica gel (petroleum ether/ethyl acetate, 6/1, v/v) to get **3** ($R_f = 0.8$) as an orange solid (1.23 g, 73%). ^1H NMR (300 MHz, CDCl_3): δ 7.94 (s, 2H), 6.18 (s, 2H), 2.50 (s, 6H), 2.33 (s, 6H); MS (EI, m/z): 361.13 $[\text{M}+\text{H}]^+$.

Synthesis of **4**

Iodic acid (352 mg, 2.0 mmol) dissolved in a minimal amount of water was added dropwise to a solution of **3** (338 mg, 1.0 mmol) and iodine (316 mg, 2.5 mmol) in 150 mL ethanol. The resulting mixture was stirred at 60 °C for 3 h. After cooling to room temperature, the yellow precipitate was collected by filtration and further recrystallized from ethanol to afford **4** as an orange solid (520 mg, 88%). ^1H NMR (300 MHz, CDCl_3): δ 8.00 (s, 2H), 2.55 (s, 6H), 2.31 (s, 6H).

ESI 2. Optical data



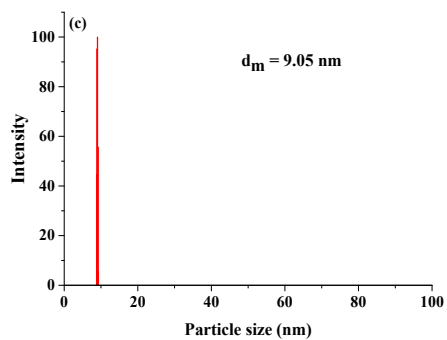


Figure S1. DLS data of (*R/S*)-**7** in THF/water mixed solvents (a) 40/60 v/v; (b) 5/95 v/v and dichloromethane/hexane mixed solvents (c) 5/95 v/v, where d_m is the mean diameter (1.0×10^{-5} mol/L).

ESI 3. NMR spectra

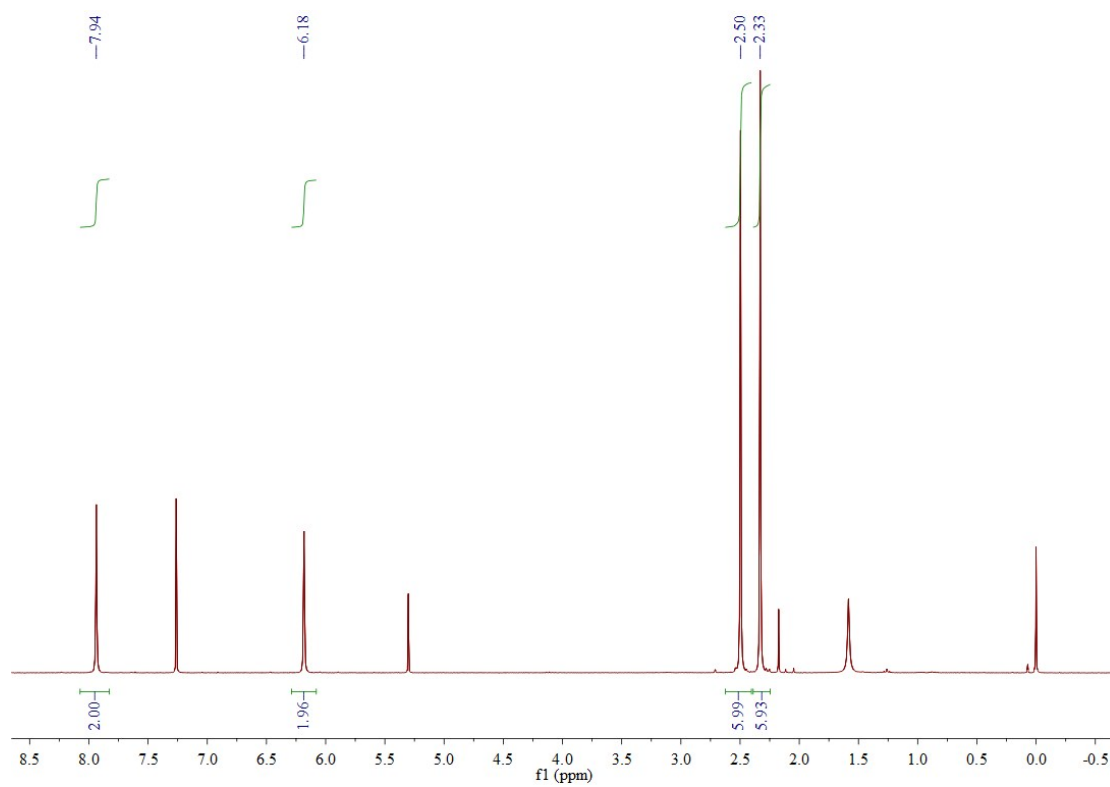


Figure S2. ¹H NMR spectra of **3**

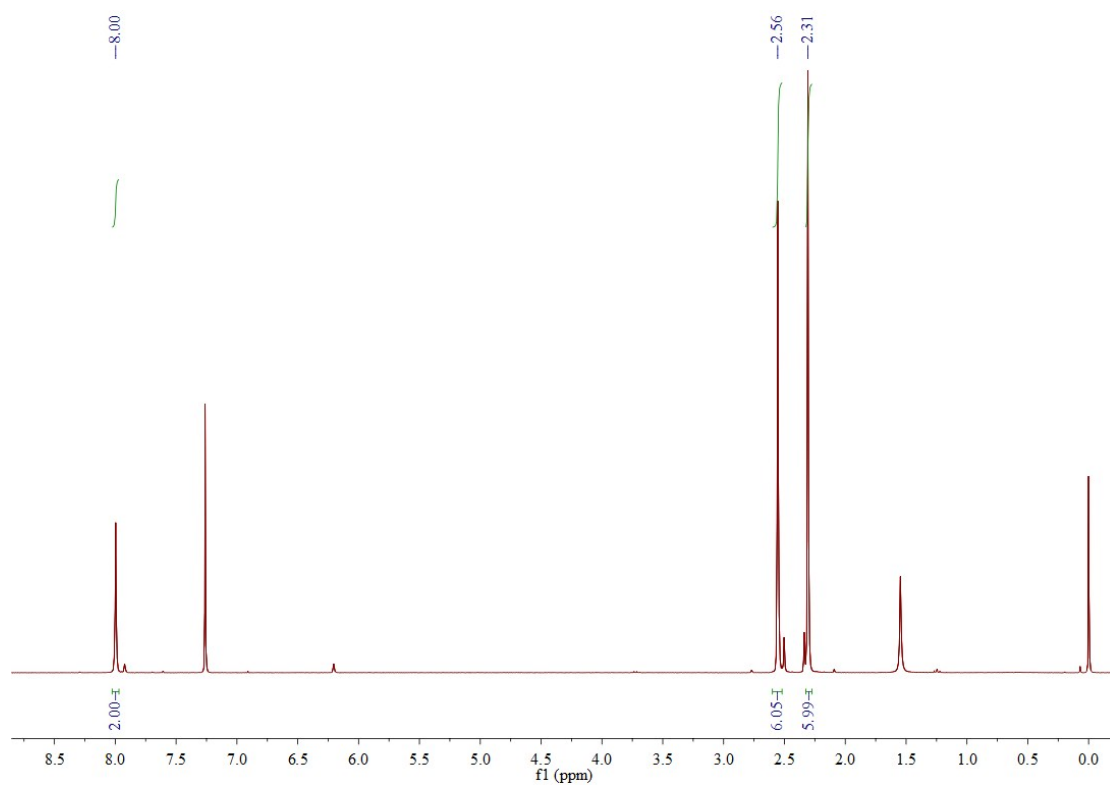


Figure S3. ¹H NMR spectra of **4**

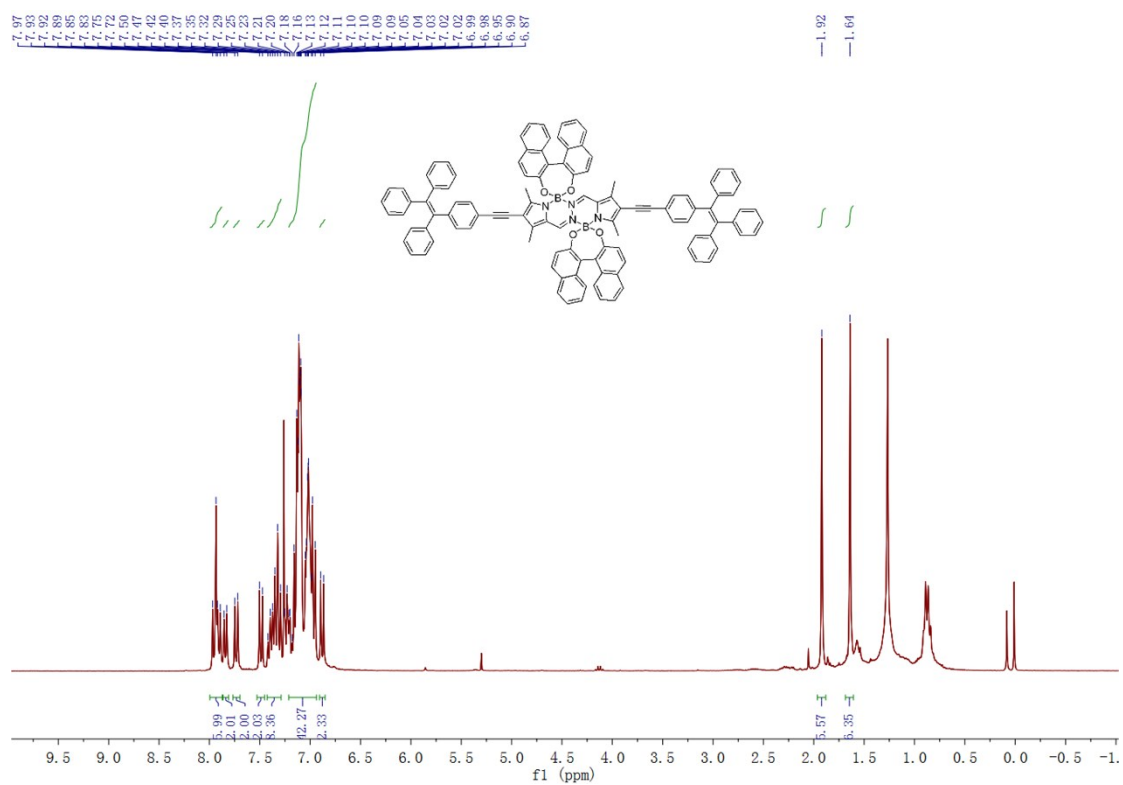


Figure S4. ¹H NMR spectra of 7

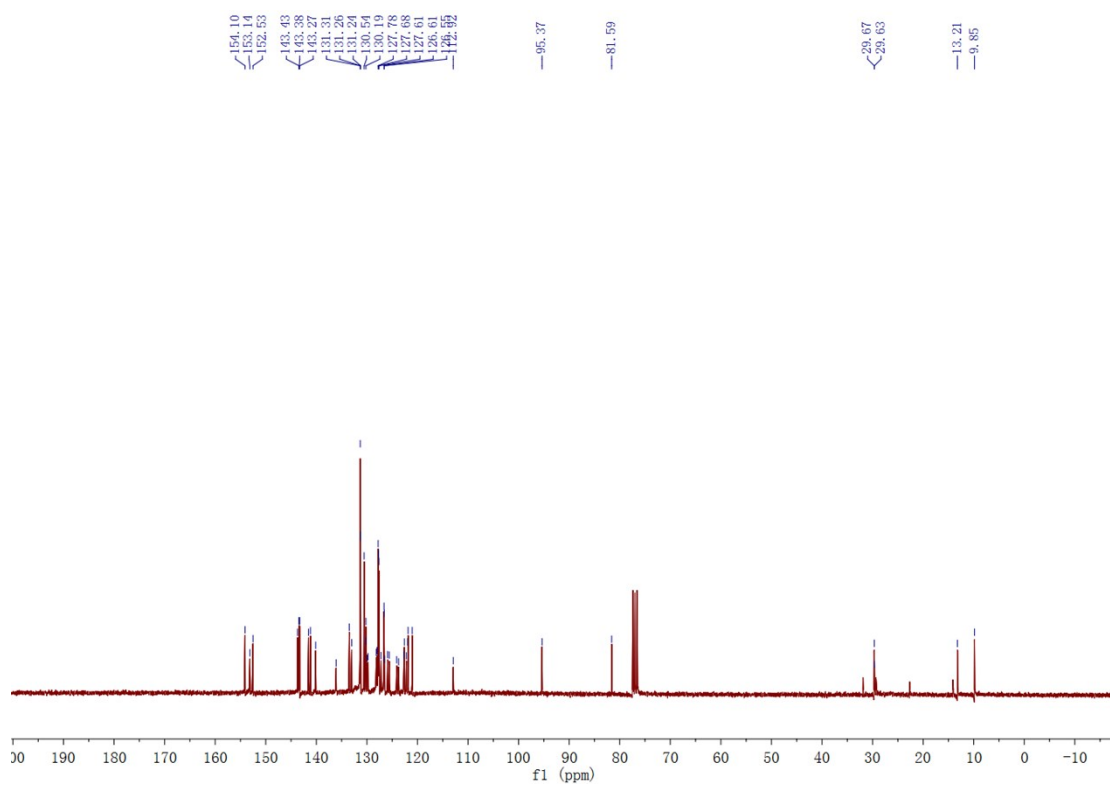


Figure S5. ¹³C NMR spectra of 7