A homochiral triple helix constructed from an axially chiral bipyridine

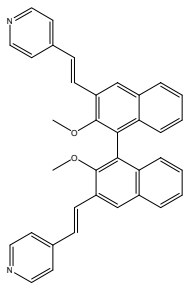
Yong Cui, Helen L. Ngo, and Wenbin Lin*

Department of Chemistry, CB#3290, University of North Carolina, Chapel Hill, NC 27599, USA E-mail: wlin@unc.edu

Experimental.

Materials and Methods. All of the chemicals were purchased from Aldrich or Fisher Scientific, and used without further purification. The IR spectra were recorded as KBr pellets on a Nicolet Magna-560 FT-IR spectrometer. X-ray powder diffraction data (XRPD) were recorded on a Rigaku MultiFlex diffractometer at 40 kV, 40 mA for CuKα (λ = 1.5406 Å), with a scan speed of 0.05-0.2 deg/min. The calculated XRPD patterns were produced using the SHELXTL-XPOW program and single crystal reflection data. TGA experiments were carried out at a heating rate of 4 °C/min in air. The CD spectra were recorded as KBr pellets on a JASCO J-810 Circular Dichroism system.

Synthesis of 2,2'-dimethoxy-1,1'-binaphthyl-3,3'-bis(divinylpyridine). A mixture of 3,3'-diiodo-2,2'-dimethoxy-1,1'-binaphthalene (2.0 g, 3.5 mmol), 4-vinylpyridine (1.14 mL, 10.6 mmol), palladium (II) acetate(71 mg, 0.177 mmol), and tris(o-tolyl)phosphine (108 mg, 0.355 mmol) was dissolved in 30 mL of anhydrous DMF and 20 mL of NEt_3. The mixture was heated to reflux for 48 hrs. Upon cooling to r.t., the organic volatiles were removed under reduced pressure, and the residue was dissolved in EtOAc and washed with water 3 times. The organic layer was dried with $MgSO_4$. The crude product was purified by silica gel column chromatography with EtOAc:actone (v/v 1:1) to give 900 mg of pure product (yield: 50%). ^{1}H NMR ($CDCl_3$) : δ 8.60 (d, 4H, $^3J_{H\text{-}H}$ = 5.2 Hz, H_{10}), 8.29 (s, 2H, H_4), 7.95 (d, 2H, $^3J_{H\text{-}H}$ = 8.5 Hz, H_5), 7.82 (d, 2H, $^3J_{H\text{-}H}$ = 17 Hz, H_a), 7.43 (m, 6H, H_9 & H_6), 7.34 (d, 2H, $^3J_{H\text{-}H}$ = 17 Hz, H_b), 7.27 (t, 2H, $^3J_{H\text{-}H}$ = 7.8 & 7.2 Hz, H_7), 7.16 (d, 2H, $^3J_{H\text{-}H}$ = 9.2 Hz, H_8), 3.41 (s, 6H, -OCH_3). ^{13}C NMR($CDCl_3$): δ 154.5 (s), 150.2 (s), 144.9 (s), 134.1 (s), 130.7 (s), 129.9 (s), 128.6 (s), 128.4 (s), 127.9 (s), 127.2 (s), 126.9 (s), 125.7 (s), 125.4 (s), 125.2 (s), 120.9 (s), 61.4 (s, OCH_3).



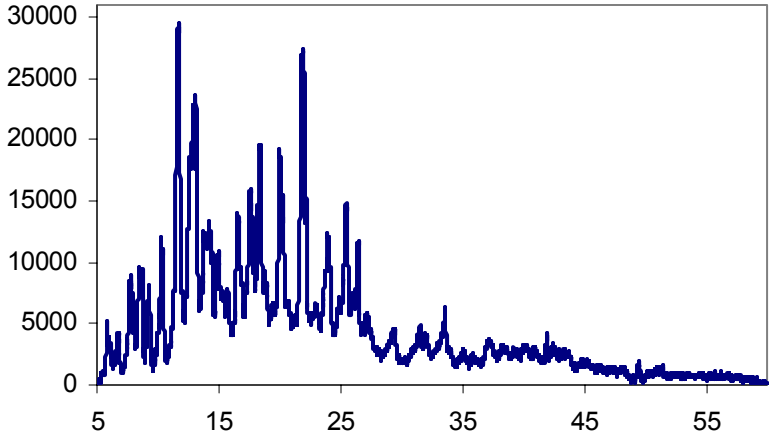


Figure S1. Powder XRD pattern of **1**.

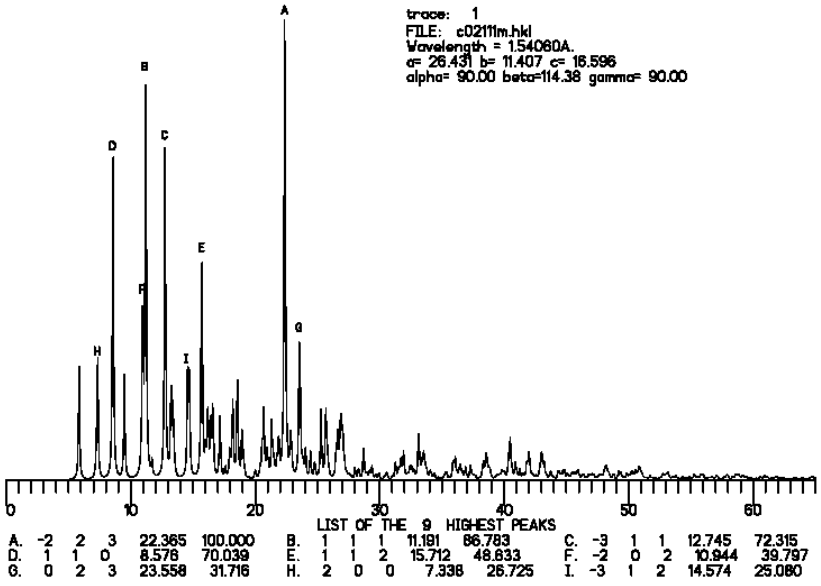


Figure S2. Simulated powder XRD pattern of **1**.

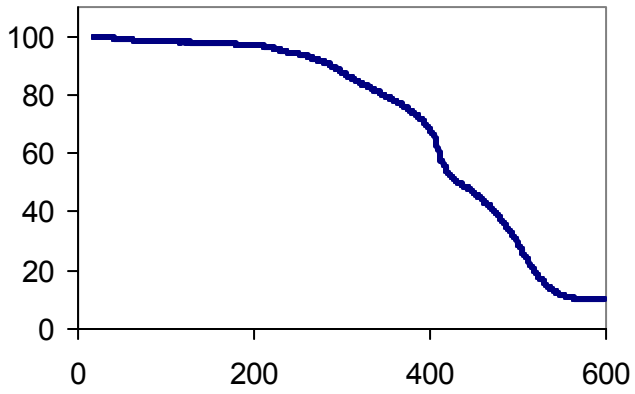


Figure S3. TGA curve of **1**.

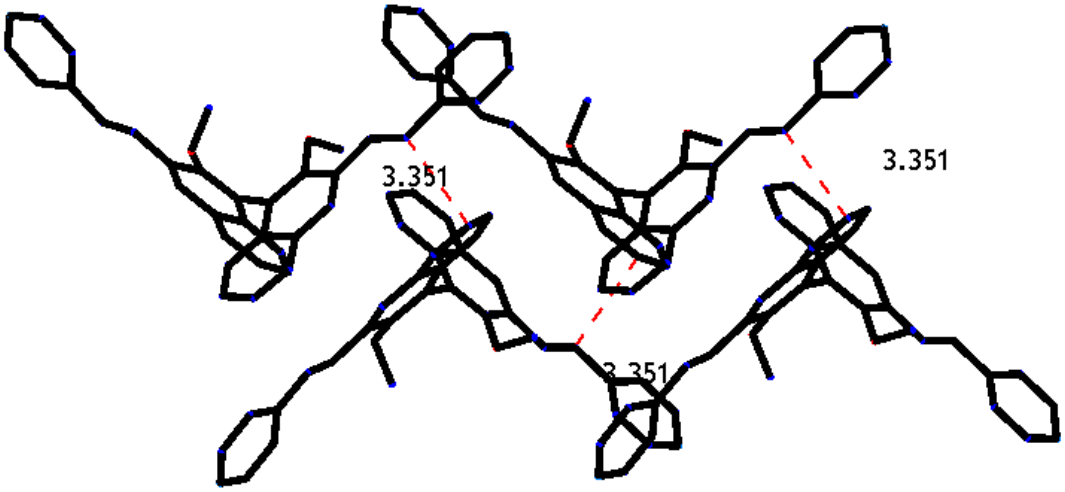


Figure S4. A schematic showing the π-π interactions in **1**.