# New Types of Homochiral Helical Coordination Polymers Constructed by *exo*-Bidentate Binaphthol Derivatives

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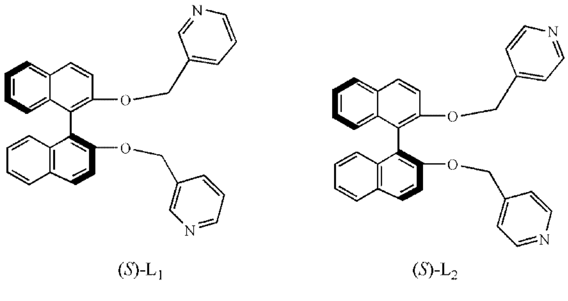
Assembly of axially chiral (*S*)-2,2′-bis(3-pyridylmethyleneoxy)-1,1′-binaphthalene [(*S*)-$L_1$] and (*S*)-2,2′-bis(4-pyridylmethyleneoxy)-1,1′-binaphthalene [(*S*)-$L_2$] with $AgNO_3$ resulted in a double-stranded helical coordination polymer encapsulating guest water molecules and a helix-like complex with inter-immersion clefts, respectively, in which helical chirality is due to axial chirality of the 1,1′-bi-2-naphthol scaffold.

## Introduction

The designed construction of supramolecular frameworks with helical morphology has made tremendous progress because of their practical application in functional materials and in the understanding of molecular self-assembly procedures.[1−6] The well-documented single-, double-, triple- and quadruple-stranded as well as circular and cylindrical helical complexes have been prepared from organic ligands and cleverly selected metal ions using metal-directed programmed self-assembly.[1−5] Recently, the stereoselective synthesis of homochiral helical complexes from enantiopure ligands has become an area of interest owing to their potential application in molecular recognition, asymmetric catalysis, optical materials and biomimetic chemistry.[3−5] In general, the metallo-helicated chirality in these complexes may be defined either by the chirality of at least one optically active carbon center[3d,3e] or by terminal axially chiral scaffolds in which transformation of chiral information on the helix can take place at a distance of up to 2 nm.[4] Thus, the axially chiral ligands may be used as starting materials for the design of new chiral ligands. Several helical frameworks have been constructed by introducing oligopyridine chelating groups into the axially chiral scaffolds,[4,5] but monopyridine-donor groups have been seldom employed.[6]

Optically active 1,1′-bi-2-naphthol (BINOL) has been widely used as a starting material and auxiliary in asymmetric synthesis[7] and chiral recognition[8] owing to its axial chirality and configurational stability. Based on chiral BINOL-derived ligands, Lin and co-workers have recently prepared a series of novel homochiral metallo-supramolecular complexes.[9] Several helical metal complexes based on binaphthyl chiral twisting have been also constructed by introducing the oligopyridine group into the 3,3′-[5a] and 6,6′-positions[5b] of BINOL, but helical metal-organic complexes from 2,2′-positions substituted BINOL derivatives have seldom been reported despite the fact that BINOL may form infinite helical chains through intermolecular O−H···O hydrogen bonds.[10] As an extension of our previous work[11] and with the aim of introducing valuable functional chiral spacers into pyridine-donor ligands, we have designed optically active *exo*-bidentate ligands (*S*)-2,2′-bis(3-pyridylmethyleneoxy)-1,1′-binaphthalene [(*S*)-$L_1$] and (*S*)-2,2′-bis(4-pyridylmethyleneoxy)-1,1′-binaphthalene [(*S*)-$L_2$]. Herein, we wish to report the syntheses and characterization of a double-stranded helical coordination polymer encapsulating guest water molecules $[Ag\{(S)\text{-}L_1\}(NO_3)\cdot H_2O]_n$ (**1**) and a helix-like complex with in-



Scheme 1

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terimmersion clefts $[Ag\{(S)\text{-}L_2\}(NO_3)]_n$ (**2**), assembled from (*S*)-$L_1$ and (*S*)-$L_2$ with $AgNO_3$, respectively (Scheme 1).

## Results and Discussion

The chiral ligands (*S*)-$L_1$ or (*S*)-$L_2$ are readily prepared from commercially available (*S*)-BINOL and 3-picolyl chloride hydrochloride or 4-picolyl chloride hydrochloride in the presence of NaOH. (*S*)-$L_1$ and (*S*)-$L_2$ have five interesting characteristics as a result of incorporating the axially chiral 1,1′-binaphthol spacer into the pyridine-donor group: 1) the chirality of the ligands permits the formation of homochiral compounds; 2) the chiral twist of the binaphthyl group can be used to control the helical chirality; 3) the conformational rigidity of the binaphthyl group restricts its free rotation; 4) the divergency of the *exo*-bidentate pyridyl group prevents mononucleation of this ligand which is inclined to form a multinuclear complex or polymer; 5) the flexible rotation of the methylenepyridine unit may meet the requirement of the coordination environment of metal ions. Therefore, the linkage of the pyridyl group and the optically active BINOL scaffold affords the possibility of forming homochiral helical complexes.

(*S*)-$L_1$ and (*S*)-$L_2$ are soluble in most organic solvents; treating (*S*)-$L_1$ and (*S*)-$L_2$ with $AgNO_3$ in MeCN/$H_2O$ at 60 °C afforded colorless plate crystals of complexes **1** and **2**, respectively, in nearly quantitative yield. Complexes **1** and **2** are stable in air, and are insoluble in water and common solvents.

Single-crystal X-ray diffraction analysis reveals that complex **1** crystallizes in an orthorhombic space group $P2_12_12$ and has a double-helical structure with *M* (left-handed) configuration. As shown in Figure 1, each $Ag^I$ center is coordinated by two nitrogen atoms from different (*S*)-$L_1$ ligands in a distorted linear geometry with the N(1)−Ag−N(2A) bond angle of 164.9(3)°. Bond lengths of Ag−N are 2.130(1) and 2.164(7) Å, which are typical values for $Ag^I{-}N_{py}$ coordination distances.[11,12] Each (*S*)-$L_1$ ligand behaves in an *exo*-bidentate mode bridging a pair of adjacent $Ag^I$ centers. The naphthyl rings are twisted with correspondingly linked pyridyl rings with dihedral angles between them of 8.6° and 33.4°. A dramatic twisting is observed between two naphthyl rings with a dihedral angle between them of 85.7°, which places the rings almost perpendicular to each other. The combination of these twistings results in the formation of the helical framework. The pyridyl and adjacent naphthyl rings from the other helical chain are approximately parallel with a dihedral angle between them of 7.5° and a distance between their centroids of 3.84 Å, indicating significant $\pi{-}\pi$ interaction.[13] The intermolecular $\pi{-}\pi$ interaction stabilizes the helical framework and produces a double-helical structure with an adjacent Ag···Ag separation of 4.29 Å between different helical chains. It is interesting to note that water molecules are encapsulated into the double helix as guest molecules (Figure 2). To the best of our knowledge, such a homochiral double-helical structure encapsulating guest water molecules is unprecedented.

Complex **2** crystallizes in the monoclinic space group $C_2$ and is a helix-like structure with *M* configuration. Similar to complex **1**, each $Ag^I$ center is coordinated by two nitrogen atoms from different (*S*)-$L_2$ groups [Ag−N 2.195(4),

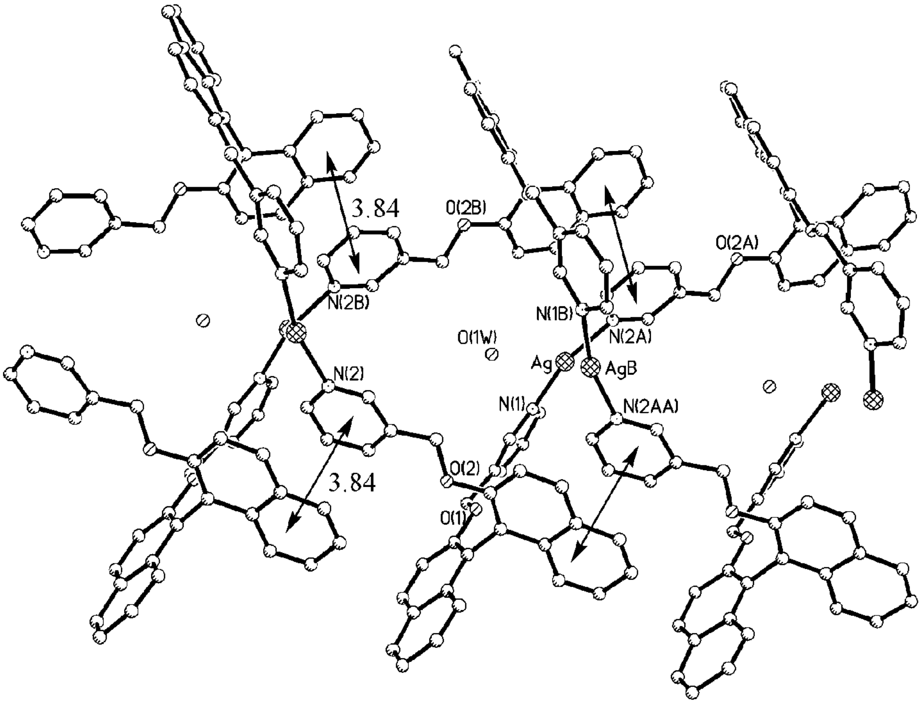

Figure 1. View of the double-helical chains encapsulating water molecules with counter anions, periphery water molecules and hydrogen atoms are omitted for clarity

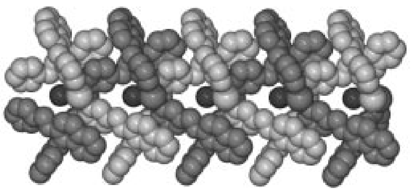

Figure 2. Space-filling diagram of the double-helical chains of **1** with counter anions, periphery water molecules and hydrogen atoms omitted for clarity

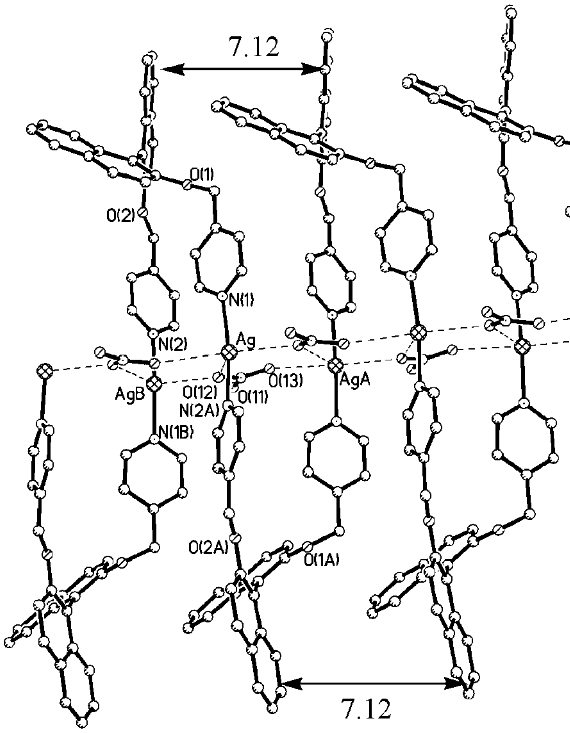

Figure 3. View of the helix-like chain of **2** with hydrogen atoms omitted for clarity

Ag−N 2.184(4) Å] in a distorted linear fashion with the N−Ag−N bond angle of 165.3(2)°. Three oxygen atoms from different $[NO_3]^-$ anions have weak interactions with the $Ag^I$ center [Ag−O(12) 2.625(8), Ag−O(12 from complex A) 2.644(10), Ag−O(13 from complex B) = 2.859(9) Å]. Thus, the coordination environment of two N and 3 O atoms around the $Ag^I$ centers can be best described as a slightly distorted triangular bipyramid. Two naphthyl rings with a twisting angle of 69.3° in $S$-$L_2$ have a smaller distortion than that in complex **1**, but the naphthyl rings and correspondingly linking pyridyl rings produce more twisting with dihedral angles between them of 42.3 and 85.4°, respectively, indicating that the metal coordination site at the 3-position of the pyridyl ring has more effect on the BINOL spacer than that at the 4-position. The interconnection of the *exo*-bidentate ($S$)-$L_2$ ligand and the $Ag^I$ centers forms an infinite left-handed helix-like framework. The separations between two $Ag^I$ centers bridged by the ($S$)-$L_2$ ligand and by the $[NO_3]^-$ anion (AgA−AgB) are 4.484 and 7.258 Å, respectively. It is worth noting that two adjacent ligands on the same side produce a cleft with a mean dimension of 7.12 Å (Figure 3). The ($S$)-$L_2$ moieties of adjacent helical chains are interimmersed into clefts of each other in an alternate fashion which effectively fills in the cleft void (Figure 4).[12]

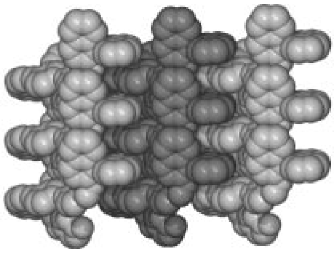

Figure 4. View of the pack diagram exhibiting interimmerse of clefts in **2**

It should be emphasized that the metal ions are not the center of chirality in complexes **1** and **2**. The chirality of the helical polymers is controlled by the inherently chiral twisting in the terminal BINOL spacer. Thus, the assembly of ($S$)-$L_1$ and ($S$)-$L_2$ with coordinatively flexible $Ag^I$ centers results in the formation of the left-handed double-helical coordination polymer and the helix-like complex, respectively.

In conclusion, assembly of optically active ($S$)-$L_1$ and ($S$)-$L_2$ units with $AgNO_3$ produces a double-stranded helical coordination polymer encapsulating guest water molecules and a helix-like complex with interimmersion clefts, respectively, in which helical chirality is due to axial chirality of the BINOL scaffold. This research demonstrates that new types of homochiral helical complexes can be constructed through monopyridine-donor groups linking the 2,2′-positions of the terminal BINOL chiral scaffold. Moreover, the different orientation of the coordination sites in the pyridyl rings of such ligands can result in different structural motifs. Further investigations are in progress.

## Experimental Section

**General:** ($S$)-1,1′-bi-2-naphthol, 3-picolyl chloride hydrochloride and 4-picolyl chloride hydrochloride were purchased from Acros. The other chemicals were commercially available and were used as purchased without further purification. The NMR spectra were recorded on a Varian Inova-500 spectrophotometer at room temperature and the chemical shifts were quoted in δ (ppm) relative to the deuterated solvent used. The optical rotation was measured with a Polarimeter 341. The mass spectra data were measured by the mass spectrometry laboratory at the Department of Applied Biology and Chemical Technology of The Hong Kong Polytechnic University and were recorded on a Fisons VG platform. The IR spectra were recorded as KBr disks on a Magna 750 FT-IR spectrophotometer. Elemental analyses were determined on an Elementar Vario ELIII elemental analyzer.

**Synthesis of ($S$)-$L_1$:** A mixture of ($S$)-1,1′-bi-2-naphthol (2.86 g, 10 mmol), 3-picolyl chloride hydrochloride (3.61 g, 22 mmol), and NaOH (1.76 g, 44 mmol) in acetone (50 mL) was stirred at reflux under nitrogen for 3 days. After cooling to room temperature, the inorganic salt was removed by filtration and washed several times with acetone. From the combined solution, the solvents were evaporated under reduced pressure, and the red oily residue was dissolved in ethyl acetate, washed twice with water, and once with brine. The organic layer was dried over anhydrous $Na_2SO_4$ and filtered. After the removal of the solvent under reduced pressure, the residue was purified by chromatography on silica gel to afford a light yellow oil-like substance. Yield: 4.12 g (88 % based on BINOL). $^1H$ NMR (500 MHz, $CDCl_3$): $\delta = 8.35$ (dd, $J = 1.5, 4.5$ Hz, 2 H, py), 8.20 (d, $J = 2.0$ Hz, 2 H, py), 8.07 (dd, $J = 9.0$ Hz, 2 H, bi), 7.96 (d, $J = 7.5$ Hz, 2 H, bi), 7.67 (d, $J = 9.0$ Hz, 2 H, bi), 7.36 (td, $J = 1.5, 6.5$ Hz, 15 Hz, 2 H, bi), 7.29−7.32 (m, 2 H, bi), 7.24−7.27 (m, 2 H, bi), 7.12 (dd, $J = 4.5, 7.5$ Hz, 2 H, py), 7.00 (d, $J = 8.0$ Hz, 2 H, py), 5.19 (s, 4 H, -$CH_2$-) ppm. $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta = 153.3$, 148.4, 147.9, 135.0, 134.8, 133.4, 132.9, 129.5, 129.0, 128.1, 126.5, 124.6, 123.6, 119.4, 115.5, 67.7 ppm. $[\alpha]_D^{20} = -99.2$ ($c = 1$, DMSO). Calcd. for $C_{32}H_{25}N_2O_2$ (469.19): calcd. C 81.91, H 5.33, N 5.97; found C 81.96, H 5.35, N 5.94. ES-MS: $m/z = 469$ ($M^+ + 1$). HRMS: calcd. $C_{32}H_{25}N_2O_2$ ($M^+ + 1$): $m/z = 469.1916$; found: 469.1938.

**Synthesis of (*S*)-L$_2$:** The procedure was similar to the synthesis of (*S*)-L$_1$ except that 4-picolyl chloride hydrochloride was used instead of 3-picolyl chloride hydrochloride. Yield: 3.75 g (80 % based on BINOL). $^1H$ NMR (500 MHz, $CDCl_3$): $\delta = 8.28$ (dd, $J = 1.5$, 4.5 Hz, 4 H, py), 8.07 (d, $J = 8.5$ Hz, 2 H, bi), 7.97 (d, $J = 8.5$ Hz, 2 H, bi), 7.59 (d, $J = 9.0$ Hz, 2 H, bi), 7.35−7.38 (m, 2 H, bi), 7.26−7.29 (m, 2 H, bi), 7.03 (d, $J = 8.5$ Hz, 2 H, bi), 6.94 (d, $J = 6.0$ Hz, 4 H, py), 5.24 (s, 4 H, -$CH_2$-) ppm. $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta = 153.7$, 149.6, 146.9, 134.3, 130.0, 129.8, 128.3, 127.0, 125.6, 124.5, 121.3, 120.7, 115.5, 69.6 ppm. $[\alpha]_D^{20} = -82.8$ ($c = 1$, DMSO). $C_{32}H_{25}N_2O_2$ (469.19): calcd. C 81.91, H 5.33, N 5.97; found C 81.97, H 5.38, N 5.92. ES-MS: $m/z = 469$ ($M^+ + 1$). HRMS: calcd. $C_{32}H_{25}N_2O_2$ ($M^+ + 1$): $m/z = 469.1916$; found 469.1925.

**Preparation of {Ag[(*S*)-L$_1$](NO$_3$)·H$_2$O}$_n$ (1):** A solution of $AgNO_3$ (0.017 g, 0.1 mmol) in $H_2O$ (5 mL) was added to a stirred solution of (*S*)-L$_1$ (0.047 g, 0.1 mmol) in MeCN (10 mL). The reaction mixture was stirred for 5 h at 60 °C, and the colorless solution was then filtered. Colorless crystals of complex **1** were obtained by allowing the filtrate to evaporate slowly in air for two weeks. Yield: 0.059 g (90 %). $C_{32}H_{26}AgN_3O_6$ (656.43): calcd. C 58.50, H 3.95, N 6.40; found C 58.39, H 3.98, N 6.37. IR (KBr pellet): $\tilde{\nu} = 3048$ (m, C−H), 2962 (m, $CH_2$), 1742 (w), 1619 (s, C−C), 1587 (s, C−C), 1506 (s, C−C), 1458 (m, C−C), 1434 (m, C−C), 1360 (w), 1385 (s, N−O), 1232 (m, C−O), 1151 (w, C−H), 1090 (m, C−H), 1016 (m, C−H), 965 (w), 916 (w), 864 (w), 808 (m), 751 (m), 700 (m), 654 (w), 573 (w), 430 (w), 405 (w).

**Preparation of [Ag{(*S*)-L$_2$}(NO$_3$)]$_n$ (2):** The procedure was similar to the synthesis of complex **1** except that (*S*)-L$_2$ was used instead of (*S*)-L$_1$. Yield: 0.060 g (94 %). $C_{32}H_{24}AgN_3O_5$ (638.4): calcd. C 60.15, H 3.76, N 6.58; found C 60.16, H 3.78, N 6.61. IR (KBr pellet): $\tilde{\nu} = 3058$ (m, C−H), 2925 (m, −$CH_2$−), 1619 (s, C−C), 1588 (s, C−C), 1561 (w, C−C), 1501 (s, C−C), 1505 (s, C−C), 1469 (w, C−C), 1446 (m, C−C), 1424 (m, C−C), 1385 (s), 1362 (w), 1266 (m, C−O), 1148 (m, C−H), 1095 (m, C−H), 1047 (m, C−H), 1019 (m, C−H), 959 (w), 911 (w), 858 (w), 804 (m), 743 (m), 670 (w), 619 (w), 527 (w), 485 (w).

**X-ray Crystallography:** The crystal data and structural determination summary for **1** and **2** are listed in Table 1. Intensity data for **1** and **2** were measured on a Siemens Smart CCD diffractometer with graphite-monochromated Mo-$K_\alpha$ radiation ($\lambda = 0.71073$ Å) at 298 K. Empirical absorption corrections were applied by using the SADABS program.[14] The structures were solved by direct methods and all calculations were performed using the SHELXL PC program.[15] The positions of the H atoms were generated geometrically (C−H bond fixed at 0.96 Å), assigned isotropic thermal parameters, and allowed to ride on their parent carbon atoms before the final cycle of refinement. The structure was refined by full-matrix least-squares minimization of $\Sigma(F_o - F_c)^2$ with anisotropic thermal parameters for all atoms except the H atoms. CCDC-205478 and -205479 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) + 44-1223-336-033; E-mail: deposit@ccdc.cam.ac.uk].

Table 1. Crystallographic data for compounds **1** and **2**

| | **1** | **2** |
|---|---|---|
| Empirical formula | $C_{32}H_{26}AgN_3O_6$ | $C_{32}H_{24}AgN_3O_5$ |
| $M_r$ [g·mol$^{-1}$] | 656.43 | 638.41 |
| Crystal system | orthorhombic | monoclinic |
| Space group | $P2_12_12$ | $C_2$ |
| $a$ [Å] | 11.371(1) | 25.225(5) |
| $b$ [Å] | 30.770(4) | 7.258(1) |
| $c$ [Å] | 9.061(1) | 16.881(3) |
| $\beta$ [°] | | 105.781(3) |
| $V$ [Å$^3$] | 3170.3(7) | 2973.9(9) |
| $Z$ | 4 | 4 |
| $T$ [K] | 294(2) | 294(2) |
| $D_c$ [g·cm$^{-3}$] | 1.375 | 1.426 |
| $\mu$ [mm$^{-1}$] | 0.681 | 0.722 |
| $\lambda$ [Mo-$K_\alpha$, Å] | 0.71073 | 0.71073 |
| Reflections collected | 21664 | 10102 |
| Unique reflections | 7280 | 6063 |
| $R_{int}$ | 0.0493 | 0.0383 |
| Parameters | 380 | 370 |
| $S$ on $F^2$ | 1.021 | 1.005 |
| $R_1$ [$I > 2\sigma(I)$][a] | 0.0774 | 0.0552 |
| $wR_2$ [all data][b] | 0.2296 | 0.1574 |
| Largest diff. peak and hole [e·Å$^{-3}$] | 0.920 and −0.428 | 0.696 and −0.334 |

[a] $R = R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

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