Supplementary Information (ESI) for CrystEngComm
This journal is (c) The Royal Society of Chemistry 2008/10/21

Supporting Information

For

Infinite Chiral Single-helical Structures Formed by the Self-assembly of Cu(II)-*N*-(2-pyridylmethyl)-aspartate Complexes.

Contents

General Information

All solvents and reagents were obtained from commercial sources and used as received without further purification. IR data were obtained as KBr pellets on Bomem DA8.3 Fourier-Transform Infrared Spectrometer. Elemental analyses were performed on a HERAEUS CHN-O-RAPID elemental analyzer by Instrumental center, National Chiao Tung University. Ultraviolet-visible spectra were recorded on an Aligent UV-Visible 8453 Spectrophotometer. NMR spectra were obtained on a Bruker DRX-300 NMR spectrometer. Crystal data were collected on a Bruker AXS X8 APEX diffractometer by Instrumental center, National Tsing Hua University.

1. Synthesis of *N*-(2-Pyridylmethyl)-aspartic acid (pasp).

To an aqueous solution (10 mL) of aspartic acid (2.66 g, 20 mmol), NaOH (1.6 g, 40 mmol) and 2-pyridinecarboxyaldehyde (2.14 g, 20 mmol) were added. The solution was stirred for 30 min in the ice bath. $NaBH_4$ (0.74 g, 20 mmol, dissolved in 10 mL of water) was added slowly. The mixture was stirred for one hour and then the pH value was adjusted to the range of 3 – 5 by 1 M HCl. The solution was evaporated and the solid was extracted in hot and dry MeOH (150 mL) for three times. The filtrate was evaporated to obtain white powder.

N-(2-Pyridylmethyl)-L-aspartic acid, yield: 54 %; Formula: $C_{10}H_{12}N_2O_4$, 224.12; ESI-MS *m/z* (rel. intensity): 110(82), 225.2(65, M+H); ^{1}H NMR (300 MHz, D_2O): δ 8.43(d, 1H, J_{H-H} = 4.7 Hz, py), 7.77(t, 1H, J_{H-H} = 7.7 Hz, py), 7.40(d, 1H, J_{H-H} = 7.8 Hz, py), 7.33(t, 1H, J_{H-H} = 6.3 Hz, py), 4.30(q, 2H, CH_2), 3.78(q, 1H, CH), 2.72(m, 2H, CH_2), ^{13}C NMR (300 MHz, D_2O): δ 176.90, 173.17, 149.63, 146.26, 141.49, 139.07, 124.94, 59.30, 50.50, 35.45. Elemental Analysis: Calculated, C, 53.58 %; H, 5.35%; N, 12.50%; Found, C, 53.06 %; H, 5.74%; N, 12.16%.

N-(2-Pyridylmethyl)-D-aspartic acid, yield: 80 %; Formula: $C_{10}H_{12}N_2O_4$, 224.12; ESI-MS *m/z* (rel. intensity): 110(82), 225.2(66, M+H); ^{1}H NMR (300 MHz, D_2O): δ 8.40(d, 1H, J_{H-H} = 4.8 Hz, py), 7.72(t, 1H, J_{H-H} = 8.3 Hz, py), 7.36(d, 1H, J_{H-H} = 7.8 Hz, py), 7.28(t, 1H, J_{H-H} = 6.3 Hz, py), 4.27(q, 2H, CH_2), 3.74(q, 1H, CH), 2.67(m, 2H, CH_2), ^{13}C NMR (300 MHz, D_2O): δ 177.15, 173.19, 149.64, 145.74, 141.91, 138.97, 124.55, 59.36, 50.45, 35.51. Elemental Analysis: Calculated, C, 53.58 %; H, 5.35%; N, 12.50%; Found, C, 53.10 %; H, 5.86%; N, 12.05%.

2. Synthesis of [Cu(L-pasp)(H_2O)].4H_2O and [Cu(D-pasp)(H_2O)].4H_2O.

To an 10 mL aqueous solution, L-pasp or D-pasp (1 mmol, 0.224 g) and $Cu(CH_3COO)_2 \cdot xH_2O$ (0.182 g, 1 mmol) were mixed and left undisturbed to for several days. Blue crystals were collected for further study.

[Cu(L-pasp)(H_2O)].4H_2O, Formula: $C_{10}H_{20}CuN_2O_9$, Molecular weight: 375.82

Elemental Analysis : Calculated, C, 31.96 %; H, 5.36%; N, 7.45%; Found, C, 31.89 %; H, 5.41%; N, 7.61%; IR (KBr): ν_{max}/cm^{-1}, 3465 (br, OH), 3194 (w, NH), 1635 (s, C=O), 1393 (s, COO^-). UV-Vis (Nujol), 686 nm (d-d).

[Cu(D-pasp)(H_2O)].4H_2O, Formula: $C_{10}H_{20}CuN_2O_9$, Molecular weight: 375.82, Elemental Analysis: Calculated, C, 31.96 %; H, 5.36%; N, 7.45%; Found, C, 31.86 %; H, 5.21%; N, 7.86%; IR (KBr): ν_{max}/cm^{-1}, 3446 (br, OH), 3170 (w, NH), 1630 (s, C=O), 1400 (s, COO^-).UV-Vis (Nujol), 685 nm (d-d).

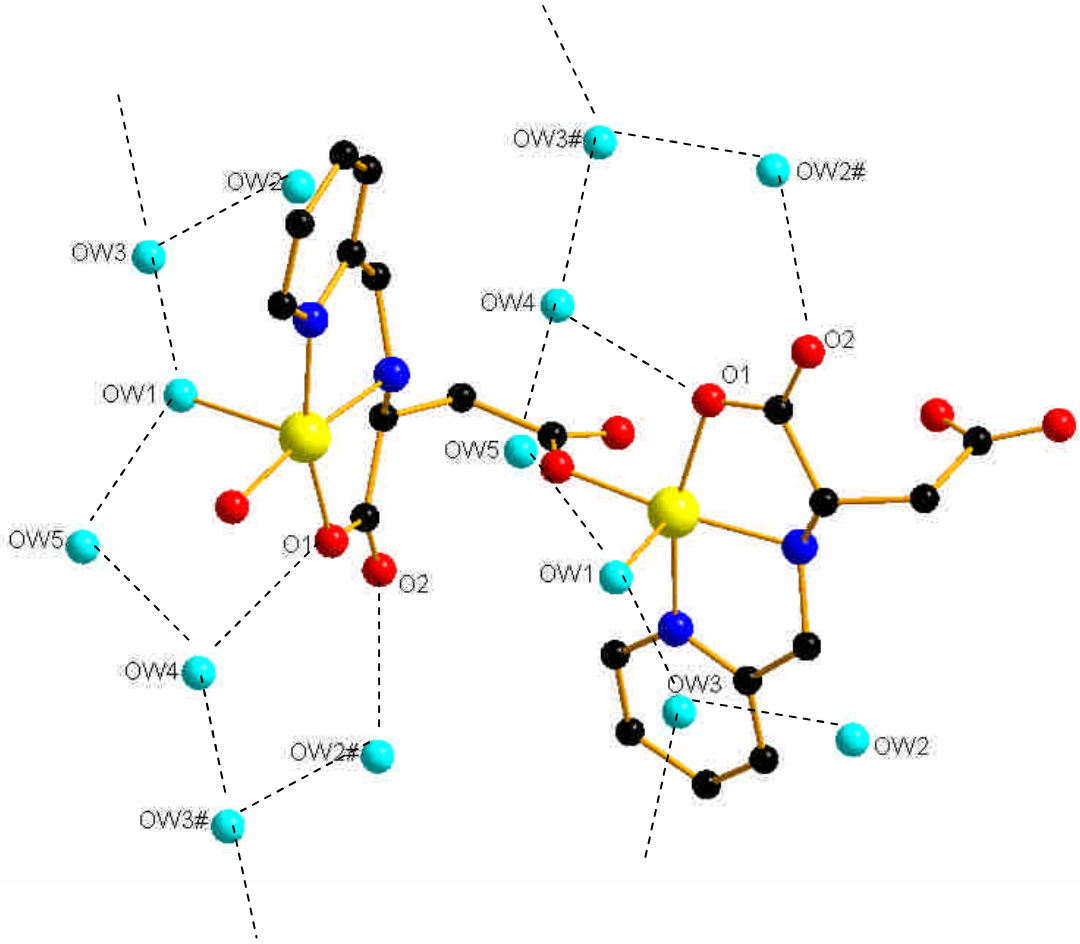

Figure S1. Water molecules in the crystal structure of [Cu(L-pasp) (H_2O)].4H_2O. C (black), N (dark blue), O (red), Cu (yellow), water (light blue). Symmetry transformations used to generate equivalent atoms: #1 -x+1, y+1/2, -z+1/2; #2 -x+1, y-1/2, -z+1/2.

Table S1. The distances between water molecules in the crystal structure of [Cu(L-pasp) (H_2O)].4H_2O.

O···O	Distance [Å]
OW5···OW4	2.733(1)
OW4···OW3	2.876(3)
OW3···OW1	2.776(4)
OW1···OW5	2.785(4)
OW2···OW5	2.779(4)
OW2···OW3	2.629(1)
OW4···O1	2.781(2)
OW2···O2	2.781(3)

Table S2. Selected Bond Lengths and Bond Angles for [Cu(L-pasp)(H_2O)].4H_2O

Bond Length [Å]		Bond Angle [°]	
Cu(1)-O(3)#1	1.930(3)	O(3)#1-Cu(1)-N(2)	172.89(16)
Cu(1)-O(1)	1.994(4)	O(3)#1-Cu(1)-O(1)	94.24(15)
Cu(1)-N(2)	1.977(4)	N(2)-Cu(1)-O(1)	82.60(15)
Cu(1)-N(1)	2.001(4)	O(3)#1-Cu(1)-N(1)	100.25(16)
Cu(1)-OW1	2.280(4)	N(2)-Cu(1)-N(1)	82.21(16)
O(1)-C(8)	1.272(6)	N(1)-Cu(1)-O(1)	163.92(17)
O(2)-C(8)	1.232(6)	O(3)#1-Cu(1)-OW1	91.19(16)
O(3)-C(10)	1.265(6)	N(2)-Cu(1)-OW1	95.33(16)
O(4)-C(10)	1.259(6)	O(1)-Cu(1)-OW1	93.01(17)
N(1)-C(1)	1.337(7)	N(1)-Cu(1)-OW1	93.61(16)
N(1)-C(5)	1.348(6)	C(8)-O(1)-Cu(1)	113.5(3)
N(2)-C(6)	1.476(6)	C(10)-O(3)-Cu(1)#2	114.6(3)
N(2)-C(7)	1.478(6)	C(1)-N(1)-Cu(1)	126.2(4)
C(1)-C(2)	1.378(9)	C(5)-N(1)-Cu(1)	113.5(3)
C(2)-C(3)	1.405(11)	C(6)-N(2)-Cu(1)	108.6(3)
C(3)-C(4)	1.373(10)	C(7)-N(2)-Cu(1)	106.3(3)
C(4)-C(5)	1.363(8)		
C(5)-C(6)	1.481(8)		
C(7)-C(8)	1.513(6)		
C(7)-C(9)	1.521(7)		
C(9)-C(10)	1.540(7)		

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, y+1/2, -z+1/2; #2 -x+1, y-1/2, -z+1/2

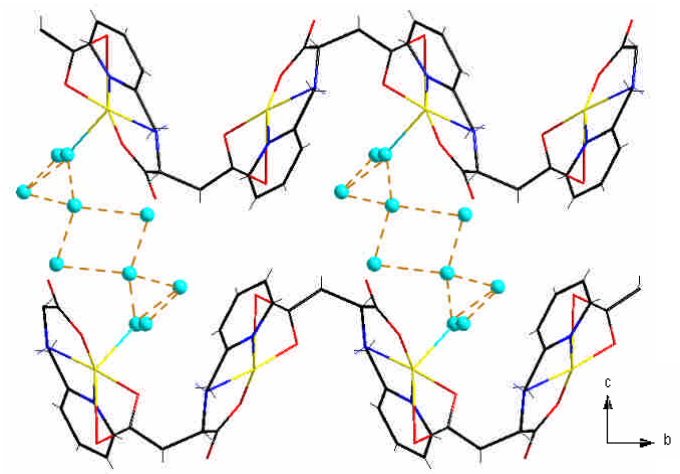

Figure S2. Water chains in the crystal structure of [Cu(L-pasp) (H_2O)].4H_2O. The top view of the *a* axis. C (black), N (dark blue), O (red), Cu (yellow), water (light blue).

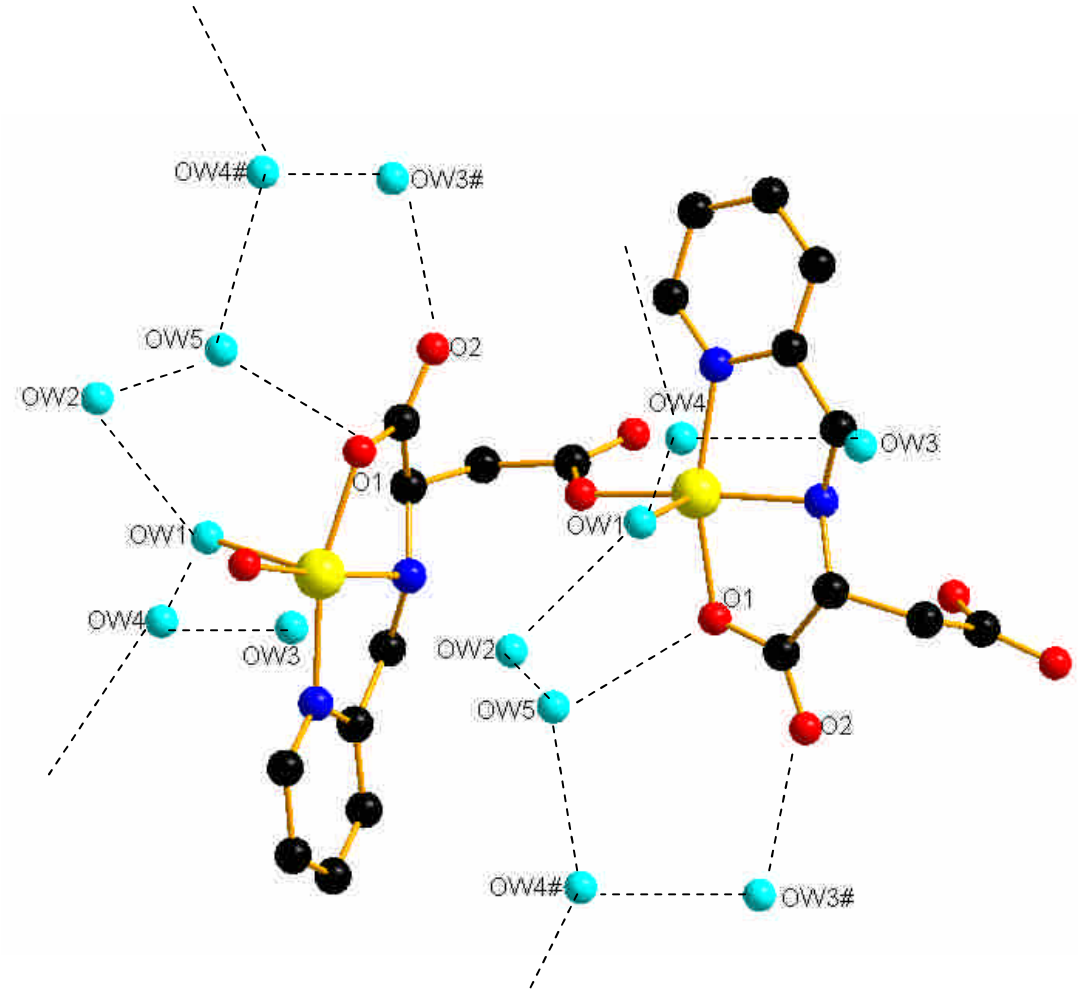

Figure S3. Water molecules in the crystal structure of [Cu(D-pasp) (H_2O)].4H_2O. C (black), N (dark blue), O (red), Cu (yellow), water (light blue). Symmetry transformations used to generate equivalent atoms: #1 x-1/2, -y+3/2, -z+1; #2 x+1/2, -y+3/2, -z+1.

Table S3. The distances between water molecules in the crystal structure of [Cu(D-pasp) (H_2O)].4H_2O.

O…O	Distance [Å]
OW5…OW4	2.788(1)
OW4…OW3	2.854(2)
OW4…OW1	2.723(1)
OW1…OW2	2.796(1)
OW2…OW5	2.787(1)
O2…OW3	2.672(4)
O1…OW5	2.820(1)

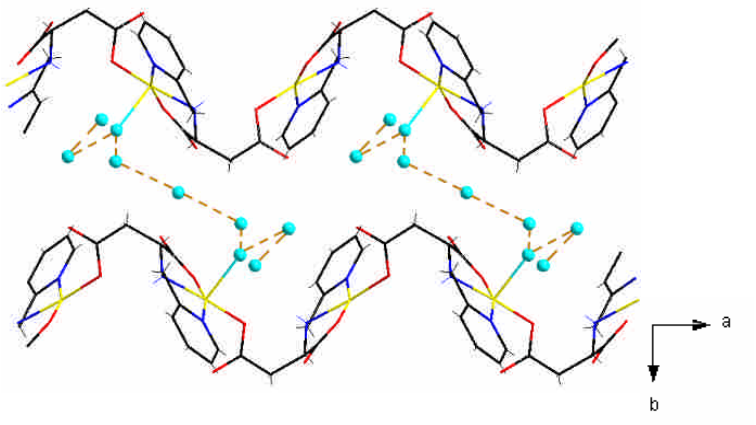

Figure S4. Water chains in the crystal structure of [Cu(D-pasp) (H_2O)].4H_2O. The top view of the *c* axis. C (black), N (dark blue), O (red), Cu (yellow), water (light blue).

Table S4. Selected Bond Lengths and Bond Angles for [Cu(D-pasp)(H_2O)].4H_2O

Bond Length [Å]		Bond Angle [°]	
Cu(1)-O(3)#1	1.940(3)	O(3)#1-Cu(1)-N(2)	172.54(15)
Cu(1)-O(1)	1.969(4)	O(3)#1-Cu(1)-O(1)	92.13(14)
Cu(1)-N(2)	1.998(4)	N(2)-Cu(1)-O(1)	82.72(15)
Cu(1)-N(1)	2.012(4)	O(3)#1-Cu(1)-N(1)	101.99(15)
Cu(1)-OW1	2.259(3)	N(2)-Cu(1)-N(1)	82.29(16)
O(1)-C(8)	1.278(6)	N(1)-Cu(1)-O(1)	163.01(15)
O(2)-C(8)	1.225(6)	O(3)#1-Cu(1)-OW1	90.75(13)
O(3)-C(10)	1.278(5)	N(2)-Cu(1)-OW1	94.93(14)
O(4)-C(10)	1.243(5)	O(1)-Cu(1)-OW1	93.50(15)
N(1)-C(1)	1.337(7)	N(1)-Cu(1)-OW1	95.71(15)
N(1)-C(5)	1.350(6)	C(8)-O(1)-Cu(1)	114.2(3)
N(2)-C(6)	1.470(6)	C(10)-O(3)-Cu(1)#2	117.6(3)
N(2)-C(7)	1.474(6)	C(1)-N(1)-Cu(1)	128.5(4)
C(1)-C(2)	1.379(9)	C(5)-N(1)-Cu(1)	112.8(3)
C(2)-C(3)	1.370(10)	C(6)-N(2)-Cu(1)	108.2(3)
C(3)-C(4)	1.372(10)	C(7)-N(2)-Cu(1)	104.9(3)
C(4)-C(5)	1.365(7)		
C(5)-C(6)	1.505(7)		
C(7)-C(8)	1.547(7)		
C(7)-C(9)	1.530(6)		
C(9)-C(10)	1.500(6)		

Symmetry transformations used to generate equivalent atoms:
#1 x-1/2, -y+3/2, -z+1; #2 x+1/2, -y+3/2, -z+1

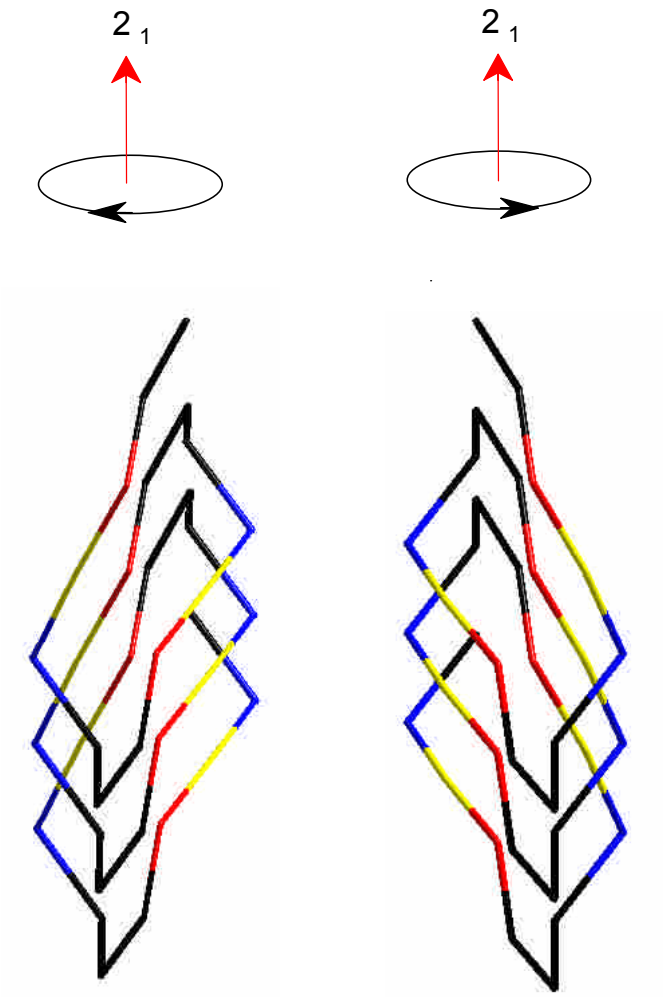

Figure S5. Helical structures of the two complexes [Cu(L-pasp)(H_2O)] (left) and [Cu(D-pasp)(H_2O)] (right). Only atoms in the helix are shown. C (black), N (dark blue), O (red), Cu (yellow).