Supplementary Material (ESI) for Chemical Communications

Electronic Supplementary Information

Spontaneous formation of chiral supramolecular superhelix in the crystalline state using single-stranded tetranuclear metallohelicate

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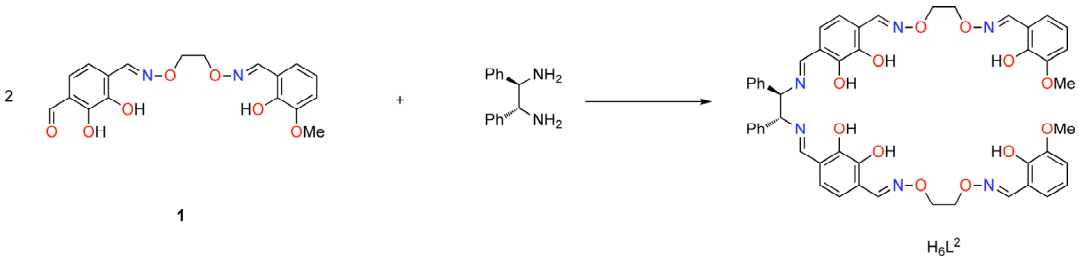
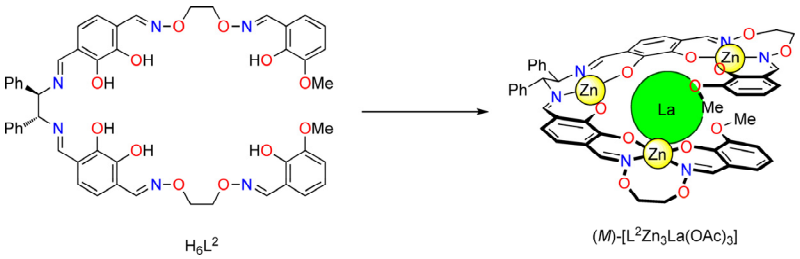
Synthesis of ligand H_6L^2.

A solution of (*R*,*R*)-1,2-diphenylethylenediamine (31.8 mg, 0.15 mmol) in chloroform/ethanol (1:1, 10 mL) was added dropwise to a solution of aldehyde **1**[1] (112.2 mg, 0.30 mmol) in chloroforom/ethanol (1:1, 10 mL) at 60 °C and the solution was heated for 2 h at the temperature. After the removal of the solvent, the crude product was purified by HPLC (GPC, $CHCl_3$) to afford yellow crystals of H_6L^2 (101 mg, 72%); mp 105-106 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.90 (s, 6H), 4.46-4.51 (m, 8H), 4.73 (s, 2H), 6.65 (d, J = 8.1 Hz, 2H), 6.73 (d, J = 8.1 Hz, 2H), 6.80-6.85 (m, 4H), 6.90 (dd, J = 6.9, 2.6 Hz, 2H), 7.14-7.21 (m, 10H), 8.19 (s, 2H), 8.25 (s, 2H), 8.33 (s, 2H), 9.45 (s, 2H), 9.71 (s, 2H), 13.58 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 56.22, 73.10, 73.21, 80.24, 113.60, 116.49, 118.29, 119.02, 119.47 (×2), 121.93, 122.46, 127.81, 127.89, 128.46, 138.77, 145.89, 147.15, 148.19, 150.16, 151.32, 152.01, 165.79. Anal. Calcd for $C_{50}H_{48}N_6O_{12}$: C, 64.93; H, 5.23; N, 9.09. Found: C, 64.81; H, 5.26; N, 9.09.



Synthesis of $[L^2Zn_3La(OAc)_3]$.

A solution of zinc(II) acetate dihydrate (3.29 mg, 0.015 mmol) and lanthanum(III) acetate sesquihydrate (1.72 mg, 0.005 mmol) in methanol/water (9:1) was added to a solution of H_6L^2 (4.63 mg, 0.005 mmol) in chloroform. After the removal of the solvent, the residue was recrystallized from chloroform/methanol to give yellow crystals of $[L^2Zn_3La(OAc)_3]$ (7.1 mg, 87%). Anal. Calcd for $C_{56}H_{51}LaN_6O_{18}Zn_3 \cdot H_2O \cdot 2MeOH \cdot CHCl_3$: C, 43.40; H, 3.83; N, 5.15. Found: C, 43.57; H, 3.88; N, 5.06.



References

[1] Akine, S.; Taniguchi, T.; Saiki, T.; Nabeshima, T. *J. Am. Chem. Soc.* **2005**, *127*, 540-541.