Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

for

Hexagon-prismatic dodecameric water cluster: a building unit of the five-fold interpenetrating six-connected supramolecular network

Xiaoju Li,*[ab] Xiahong Xu,[a] Daqiang Yuan,[b] Xiulan Weng [a]

[a] Fujian Key Laboratory of Polymer Materials, College of Materials Science and Engineering, Fujian Normal University, Fuzhou, Fujian, 350007, P.R. China. E-mail: xiaojuli@fjnu.edu.cn.

[b] State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian, 350002, P.R. China.

Experimental Section

General methods: IR spectra were recorded using KBr plates on a BIORAD model 3000 FTS spectrometer. ^{1}H and ^{13}C spectra were recorded on spectrometers at 300 and 75 MHz, respectively, by using D_2O as a locking solvent. Chemical shifts were reported in ppm relative TMS. Thermogravimetric analysis (TGA) measurements were carried out by heating samples at 10 °C/min from 25 to 800 °C in a dynamic nitrogen atmosphere (flow rate = 70 mL/min). Elemental analyses were performed on a CE-440 Elemental Analyzer.

X-ray Crystallography.

Data collection for **1** was performed on Saturn 724 CCD diffractometer using graphite monochromated Mo-*K*α radiation (λ = 0.71073 Å). Intensity data set was collected using *ω* scan technique and corrected for *Lp* effects. The primitive structures were solved with direct methods and refined on *F*2 with full matrix least-squares methods using the SHELXS-97 and SHELXL-97 programs, respectively. [1] The non-hydrogen atoms were refined anisotropically. The hydrogen atoms of water were located from the difference Fourier map and refined isotropically. The hydrogen atoms of Bmib were geometrically fixed. CCDC:827350.

Synthesis of $[(Bmib)_3(H_2O)_{12}]_n$ (1): Bmib was synthesized according to the modified literature method.[2] $[(Bmib)_3(H_2O)_{12}]_n$ was prepared by allowing a solution of Bmib in MeOH and water (1:2, volume ratio) to slowly evaporate at room temperature. Colorless crystals of **1** were formed after a few days. Anal. Calcd (%) for $C_{36}H_{78}N_{12}O_{12}$ (871.08): C, 49.64; N, 19.30; H, 9.03. Found: C, 49.71; N, 19.39; H, 8.79. ^{1}H NMR: δ 6.92 (s, 2H), 6.80 (s, 2H), 3.85 (s, 4H), 2.22 (2, 6H), 1.63 (s, 4H); ^{13}C NMR: δ 145.9, 125.5, 120.3, 45.2, 26.7, 11.6; IR (KBr,cm^{-1}) 3113(s), 3088(s), 2985(w), 2958(s), 2924(w), 1643(w), 1523(m), 1498(s), 1466(w), 1444(m), 1425(s), 1374(vw), 1359(m), 1314(w), 1280(s), 1240(vw), 1206(vw), 1190(vw), 1145(m), 1123(s), 1075(vw), 979(s), 937(vw), 913(w), 777(s), 679(m). After **1** was heated at 60 ^{o}C in vacuo for 6h, Bmib was obtained as white power. Anal. Calcd (%) for $C_{36}H_{54}N_{12}$ (654.90): C, 66.02; N, 25.67; H, 8.31. Found: 65.87; N, 25.29; H, 8.22.

References

(1) G. M. Sheldrick, SHELXTL-97, Program for the Solution of Crystal Structures, University of Göttingen: Germany 1997.

(2) X. J. Li, Y. Z. Cai, Z. L. Fang, L. J. Wu, B. Wei, S. Lin, *Cryst. Growth Des.*, 2011, **11**, 4517-4524.

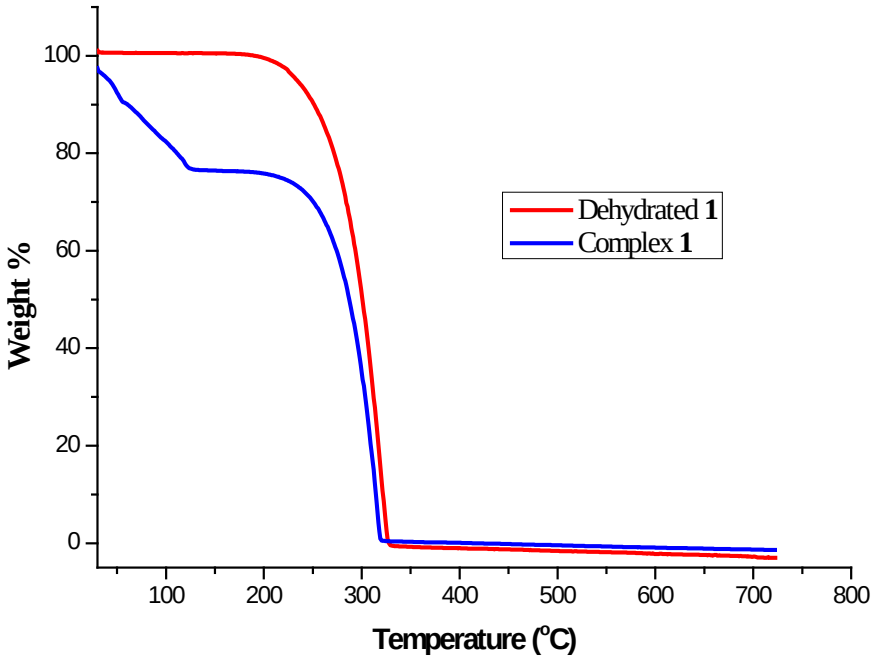

Figure S1 TGA curves of complex **1** and dehydrated **1**.

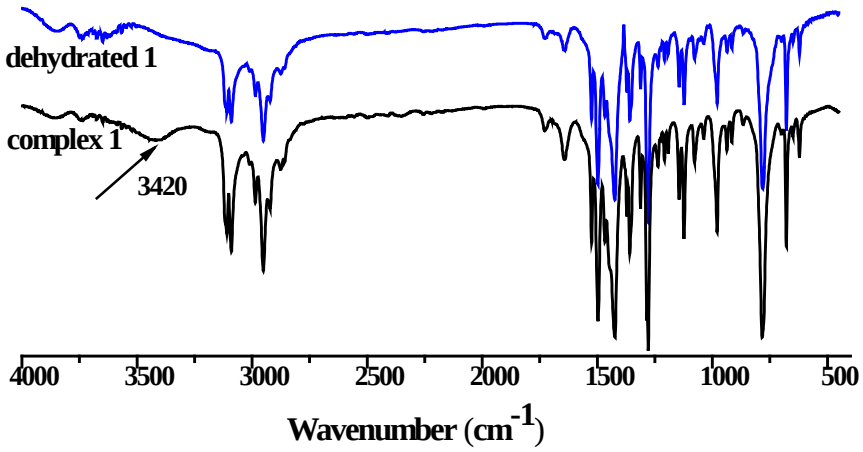

Figure S2 IR spectra of complex **1** and dehydrated **1**.