Supporting Information

A 3D Chiral Nanoporous Coordination Framework Consisting of Homochiral Nanotubes Assembled from Octuple Helices

Xiang-Rong Hao, Xin-Long Wang, Chao Qin, Zhong-Min Su*, En-Bo Wang*,

Ya-Qian Lan, and Kui-Zhan Shao

Institute of Functional Materials, Department of Chemistry and Key Laboratory of Polyoxometalates Science of Ministry of Education, College of Chemistry, Northeast Normal University, Changchun 130024, China. Email: zmsu@nenu.edu.cn wangenbo@public.cc.jl.cn

X-ray structure analysis: $C_{31}H_{38}N_3O_8Cd$: M_r = 693.04, hexagonal, space group $P6_122$, $a = b$ = 26.712(4) Å, c = 15.919(3) Å, $\alpha = \beta$ = 90°, γ = 120°, V = 9837(3) Å^3, Z = 12, ρ_{calcd} = 1.404 mg m^{-3}, Flack parameter = –0.01(3), final $R1$ = 0.0384 for 9677 independent reflections [$I > 2\sigma(I)$]. The data were collected on a Bruker Apex CCD diffractometer at 133(2) K with graphite-monochromated $Mo_{K\alpha}$ radiation (λ = 0.71073Å).

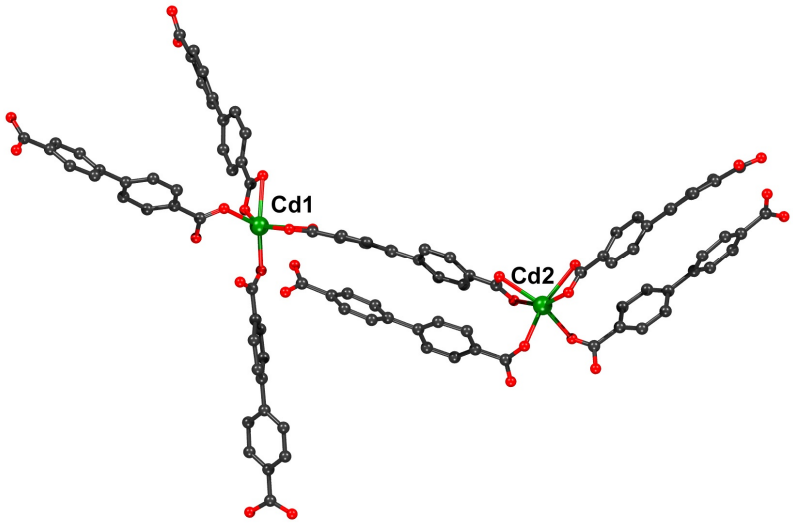
The data were collected on a Bruker Apex CCD diffractometer at 133(2) K with graphite-monochromated $Mo_{K\alpha}$ radiation (λ = 0.71073Å). The structures were solved by direct method and refined by full-matrix least-squares methods with SHELXL.[1] The anion and DMA molecules were highly disordered and could not be modeled properly, thus the SQUEEZE routine, a part of the PLATON package of crystallographic software, was applied to calculate the solvent disorder area and remove its contribution to the overall intensity data. The refinements of the guest-free structure on the squeezed data gave the final $R1$ = 0.0384 and w$R2$ = 0.0798 ($R1$ = 0.0463 and w$R2$ = 0.1322 without employing PLATON/SQUEEZE). The final formula was calculated from the SQUEEZE results combined with the following characterization techniques. Despite the disorder, identification of the guest molecules is readily accomplished by ^{1}H NMR spectroscopy. The ^{1}H NMR spectrum of **1** recorded in D_2O exhibits a set of well-resolved proton signals. The signals at 2.88, 2.73 and 1.91 ppm attributed to three CH_3 groups of DMA,[2] while the signal at 2.54 ppm ascribes to the CH_3 group of dimethylamine. Furthermore, NMR analysis reveals that the relative molar ratio of dimethylamine and DMA is approximately 1:2. The IR

spectrum of **1**, as expected, exhibits a sharp band at 1619 cm^{-1} corresponding to $\tilde{\upsilon}_{C=O}$ stretching frequency, which is indicative of DMA molecule. According to the previous literature, the peaks of 1540 and 1398 cm^{-1} are attributed to the asymmetric and symmetric stretching vibrations of the carboxylate group of bpdc ligand.[3] The peak at ca. 3419 cm^{-1} is attributed to the N-H absorption vibration of dimethylamine. Further information supporting the formula of **1** is obtained by thermogravimetric analysis, elemental analysis and the consideration of charge balance.

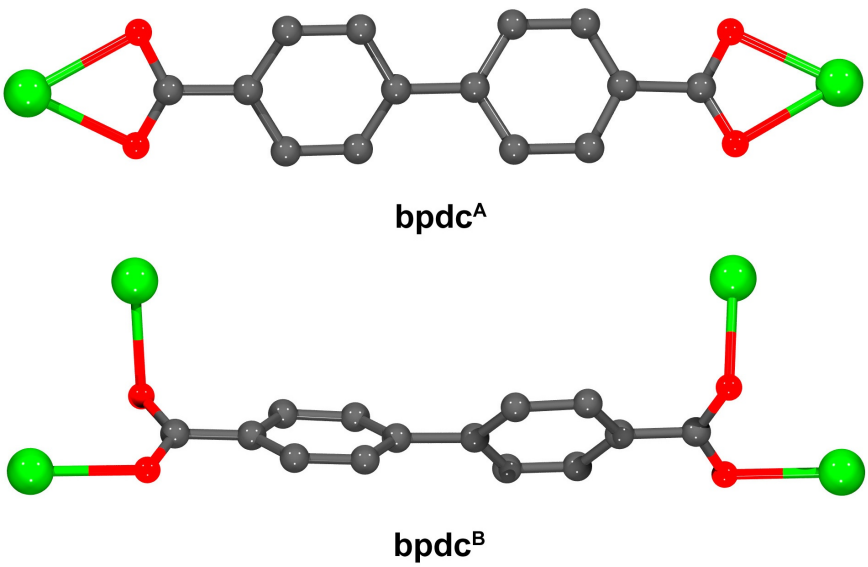
[1] G. M. Sheldrick, SHELXL, Version 5.1. Siemens Industrial Automation, Inc., Madison, WI, 1997.

[2] L. Carlucci, G. Ciani, D. M. Proserpio, F. Porta, *Angew. Chem. Int. Ed.* 2003, **42**, 317.

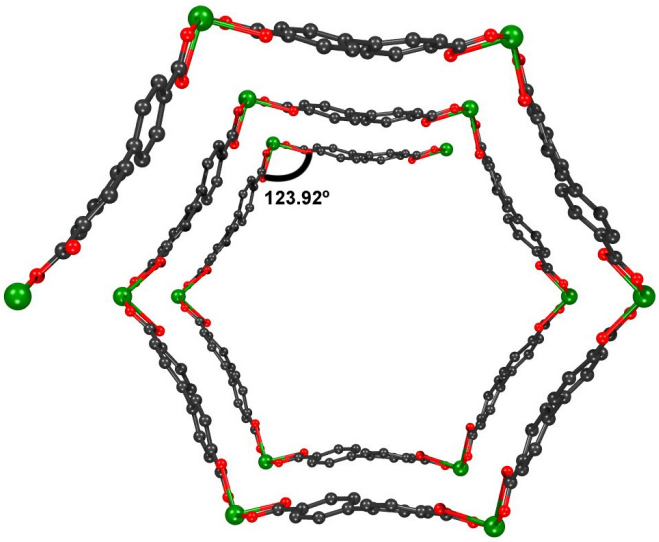
[3] a) F. A. Almeida Paz, Y. Z. Khimyak, A. D. Bond, J. Rocha, J. Klinowsk, *Eur. J. Inorg. Chem.* 2002, 2823; b) Q. R Fang, X. Shi, G. Wu, G. Tian, G. S. Zhu, R. W. Wang, S. L. Qiu, *J. Solid. State.* 2003, **176**, 1; c) Y. H. Wang, B. Bredenkötter, B. Riegera, D. Volkmer, *Dalton Trans.* 2007, 689.



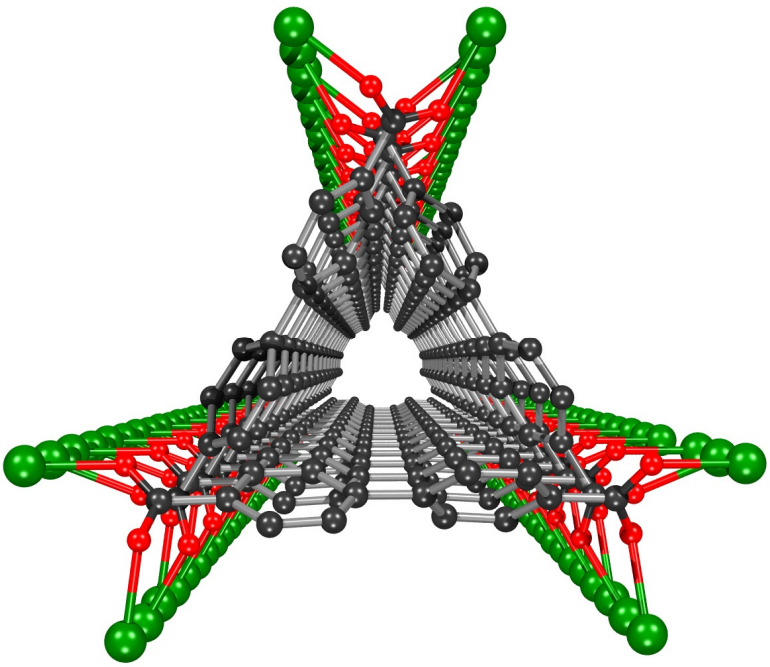
S1-1. Section of the X-ray crystal structure of **1**, showing the coordination environments of Cd^{2+} ions and their connectivity with bpdc anions. Color code: C black; O red; Cd green.



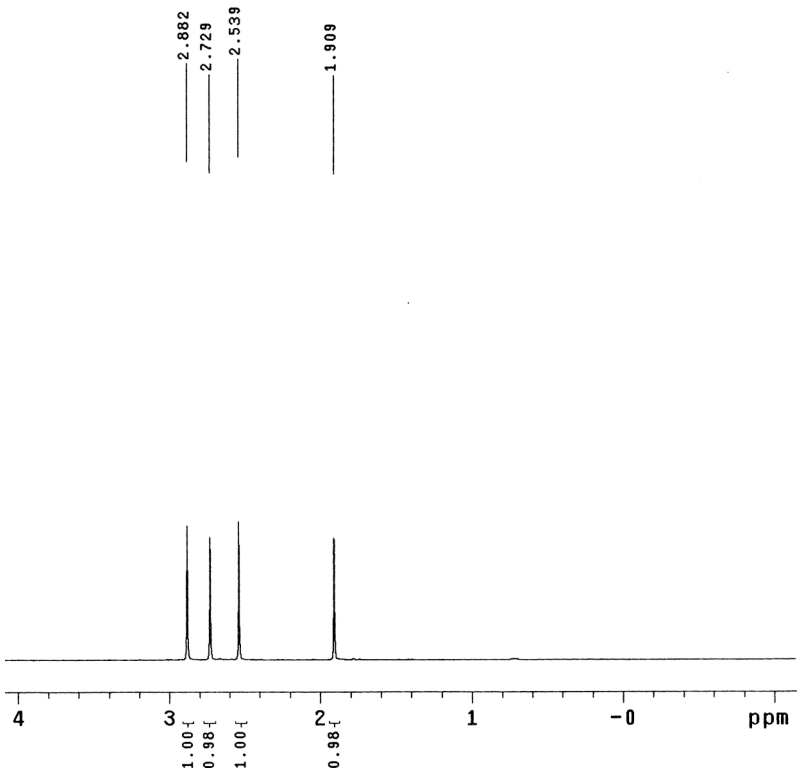
SI-2. Coordination modes of the bpdc ligands in the structure of **1**. $bpdc^A$: chelating bis(bidentate), $bpdc^B$: bridging bis(bidentate). Color code: C black; O red; Cd green.



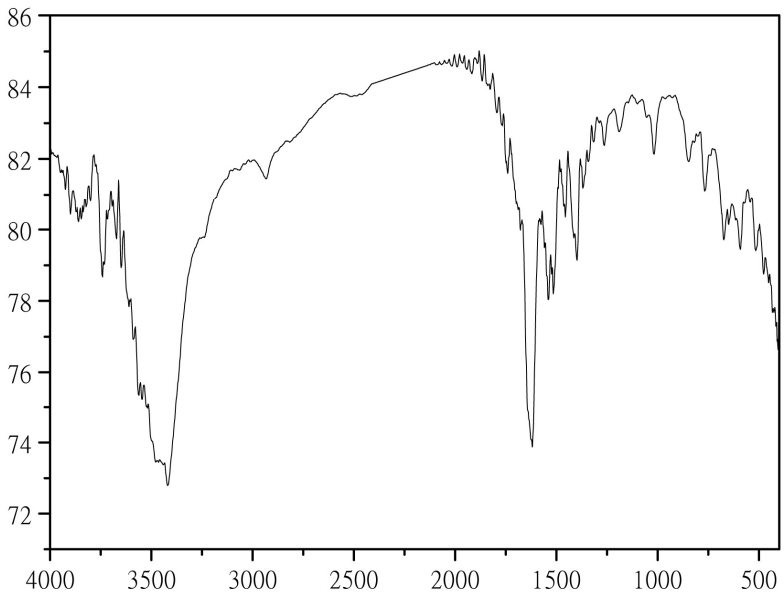
SI-3. An angle of 123.92º formed between the planes defined by the two C-O-Cd-O-C chelate rings at one metal center. Color code: C black; O red; Cd green.



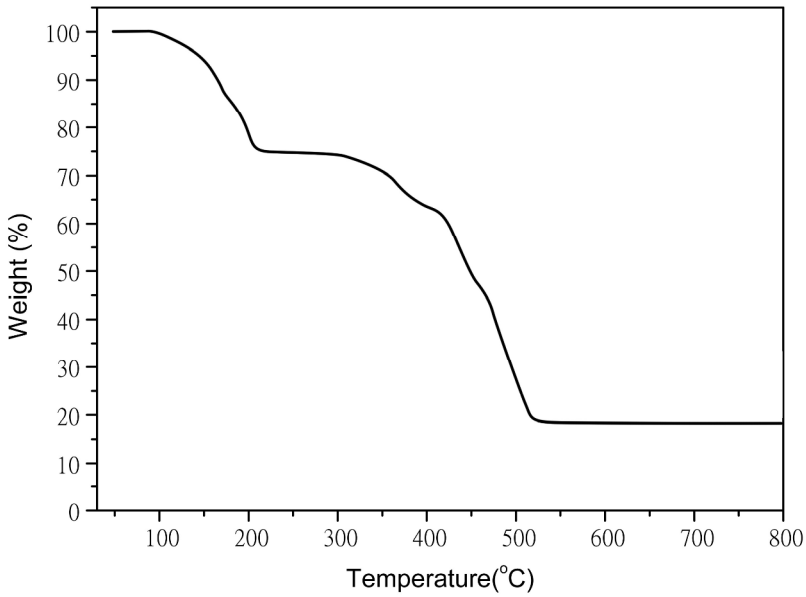
SI-4. Perspective view of the trigonal channel that is surrounded by bpdc^{B} ligands. Color code: C black; O red; Cd green.



SI-5. The ^{1}H NMR spectrum of **1** recorded in D_2O.



SI-6. FT-IR spectrum of as-synthesized **1**.



SI-7. TG curve of **1**.