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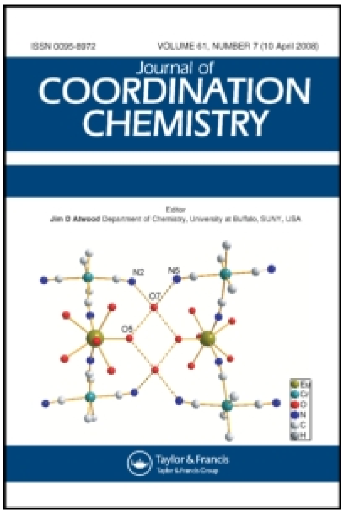

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
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Microporous 3-D chiral metal-organic framework with a quartzlike topology based on an achiral building unit

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First published on: 24 May 2010

To cite this Article Wu, Yapan , Li, Dongsheng , Fu, Feng , Tang, Long , Wang, JIJIANG and Yang, Xiao Gang(2009) 'Microporous 3-D chiral metal-organic framework with a quartzlike topology based on an achiral building unit', Journal of Coordination Chemistry, 62: 16, 2665 — 2674, First published on: 24 May 2010 (iFirst)

To link to this Article: DOI: 10.1080/00958970902902713

URL: http://dx.doi.org/10.1080/00958970902902713

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Journal of Coordination Chemistry
Vol. 62, No. 16, 20 August 2009, 2665–2674

Microporous 3-D chiral metal-organic framework with a quartzlike topology based on an achiral building unit

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(*Received 9 October 2008; in final form 7 January 2009*)

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A zinc coordination polymer, $Zn(IN)_2 \cdot 2H_2O$ (**1**) (HIN = isonicotinic acid), has been synthesized and characterized by IR, elemental analysis, thermogravimetric analysis, and single crystal X-ray diffraction. This compound crystallizes in the chiral space group $P6_2$ with $a = 15.512(3)$, $b = 15.512(3)$, $c = 6.262(2)$ Å, $V = 1304.9(5)$ Å^3, and $Z = 3$. The O and N atoms of the IN ligand are coordinated to a Zn^{II} ion, locking the asymmetry of the IN ligand and transferring this asymmetry throughout the crystal structure to "direct" the formation of a chiral network. The structure of **1** is characteristic of a 3-D quartz-like framework, which contains hetero-chiral helical channels (pseudo-trigonal and hexagon channels) alternately along the *c*-axis. Compound **1** exhibits high stability and intense fluorescent emission at room temperature.

Keywords: Zinc(II) compound; Crystal structures; Interpenetration; Topology; Chirality

1. Introduction

On the basis of the pioneering work of Zaworotko, Kitagawa, Robson, and Yaghi [1–4], a very large number of metal-organic frameworks (MOFs) have been synthesized, many exhibiting intriguing structural diversity and potential applications in gas adsorption, molecular storage, and heterogeneous catalysis [5]. As a result, porous MOFs, especially with chiral topology, are among the most explored and best investigated supramolecular architectures [1b, 3, 6–8]. However, even with the most powerful computational approaches, as yet, it is challenging to prepare chiral MOF materials by using chiral organic linkers, or by using achiral ligands under a spontaneous resolution without a chiral auxiliary [9]. Recently, excellent work has been performed by Lin *et al.*, demonstrating that combining achiral unsymmetrical bridging ligands and metal centers with well-defined coordination geometries or employing *in situ* generation of unsymmetrical organic ligands under hydrothermal conditions are effective strategies [7b, 8d, 10]. The asymmetry of the organic ligands can be transferred to building blocks of

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Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online © 2009 Taylor & Francis
DOI: 10.1080/00958970902902713

these compounds, further connecting through organic ligands to produce chiral networks and keep the initial asymmetry [11]. Nevertheless, to the best of our knowledge, few 3-D chiral coordination polymers based on achiral unsymmetrical ligands have been reported except for some carboxylic and pyridyl bridged ligands, etc. [8d, 12]. Considerable attention has been devoted to the networking ability of an appropriate multifunctional ligand and its derivatives, able to link metal ions to build a chiral coordination polymer. As a multifunctional ligand, isonicotinic acid has been widely used in the assembly of such compounds because it can lead to varieties of coordination polymers with different topologies [10a, 13]. To develop new chiral coordination polymer supramolecules with achiral unsymmetrical bridging ligands, we focused our attention on metal-isonicotinic acid coordination polymers. Herein, we describe a microporous chiral 3-D quartz-like framework with two distinct helical channels (trigonal and hexagon channel), $Zn(IN)_2 \cdot 2H_2O$ (**1**); hexagonal helical channels of **1** host right-handed double helical chains of lattice water molecules. Compound **1** exhibits intense fluorescent emission upon photoexcitation in the solid state at room temperature, and high thermal stability.

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2. Experimental

2.1. *Materials and methods*

All chemicals are of analytical reagent grade and used as received. FT-IR spectra were recorded from KBr pellets in the range 4000–400 cm^{-1} on a Bruker EQUINOX-55 spectrometer. Fluorescence spectra were performed on a Hitachi F-4500 fluorescence spectrophotometer at room temperature. Elemental analysis was determined with an Elementar Vario EL III elemental analyzer. Thermogravimetric analyses (TGA) were performed under nitrogen with a heating rate of 10°C min^{-1} using a NETZSCH STA 449C thermogravimetric analyzer. Variable-temperature X-ray powder diffraction (XRPD) was carried out on a Shimadzu XRD-7000 analyzer.

2.2. *Synthesis*

A dimethylsulfoxide (DMSO) solution (5 mL) of isonicotinic anhydride (0.1 mmol, 0.0228 g) was placed at the bottom of a straight glass tube, over which a solution of $Zn(OAC)_2 \cdot 2H_2O$ (0.2 mmol, 0.0439 g) in methanol (5 mL) was carefully layered. The tube was sealed under vacuum and put into a refrigerator. Over 2 weeks, large colorless needle-like single crystals with dimensions up to $0.46 \times 0.33 \times 0.30\,mm^3$ were obtained. Anal. Calcd for $C_{12}H_{12}N_2O_6Zn$ (%): C, 41.7; H, 3.5; N, 8.6. Found: C, 41.6; H, 3.7; N, 8.9. IR (KBr pellet, cm^{-1}): $\nu = 3473\,cm^{-1}$ (br, OH of water), ν_{as} and ν_s are 1635 and 1361 cm^{-1}, respectively (C=O, mondentate carboxylate group) (see Supplementary material: figure S1).

2.3. *X-ray crystallography*

Crystal structure determination of **1** with dimensions $0.46 \times 0.33 \times 0.30\,mm^3$ was performed on a Bruker Smart APEX II CCD diffractometer with graphite-monochromated

Mo-Kα radiation ($\lambda = 0.71073$ Å) at 298(2) K. The structure was solved by direct methods and successive Fourier difference synthesis (SHELXS-97) and refined by full-matrix least-squares on F^2 with anisotropic thermal parameters for all non-hydrogen atoms (SHELXL-97) [14]. For **1**, the disorder was treated by performing half-occupancies with O atoms of the guest water molecules. Because of the disorder of the guest water molecules in the channels, the distribution of these peaks was chemically featureless. Elemental analysis and TGA showed that each formula unit of **1** includes two guest water molecules. All non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atoms were located by geometric calculations. Crystal data and details on refinements for **1** are summarized in table 1. Selected bond distances and angles are listed in table 2.

3. Results and discussion

3.1. *Crystal structure*

X-ray crystallographic analysis reveals that each Zn^{II} coordinates to two nitrogens of two IN ligands (Zn–N, 2.025(3) Å; N–Zn–N, 112.36(17)°), and to two oxygens of two

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Table 1. Crystal data and structure refinement for **1**.

Empirical formula	$C_{12}H_{12}N_2O_6Zn$
Formula weight	345.6
Crystal system	Hexagonal
Space group	$P6_2$
Unit cell dimensions (Å)	
a	15.512(3)
b	15.512(3)
c	6.262(2)
V (Å^3)	1304.9(5)
Z	3
D_{Calcd} (g cm^{-3})	1.251
μ (Mo-Kα) (Å)	0.71073
$F(000)$	498
θ range (°)	3.03–24.97
Goodness-of-fit on F^2	1.089
Final R_1, wR_2 [$I > 2\sigma(I)$]^a	0.0309, 0.0742
R_1, wR_2 (all data)	0.0371, 0.0783

$^aR = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $^bwR_2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[(F_o^2)^2]]^{1/2}$.

Table 2. Selected bond lengths (Å) and angles (°) for **1**.[a]

Zn1–O1	1.952(2)	Zn1–N1	2.026(3)
Zn1–O1#1	1.952(2)	Zn1–N1#1	2.026(3)
O1–Zn1–O1#1	100.30(15)	O1–Zn1–N1#1	104.74(10)
O1–Zn1–N1	117.31(10)	O1#1–Zn1–N1#1	117.31(10)
O1#1–Zn1–N1	104.74(10)	N1–Zn1–N1#1	112.37(17)

[a] Symmetry transformations used to generate equivalent atoms: #1: $-x+1, -y+2, z$.

carboxylates from two other IN ligands (Zn–O, 1.952(2) Å; O–Zn–O, 100.29(15)°; N–Zn–O, 117.32(10), and 104.74(10)°, respectively) in a monodentate fashion (figure 1). The Zn^{II} centers have a slightly distorted tetrahedral geometry and extend three-dimensionally to form a α-quartz $(6^4 \cdot 8^2)$ network (figure 2). In **1**, large Zn cations replace the Si atoms in a quartz structure, and the long IN anions replace the O atoms, so that the separation between two T units can be expanded to 8.8 Å, therefore, a large void is generated within a single quartz-like net. The void space in **1** is filled by the formation of a two-folded quartz-like structure, in which two independent quartz-like nets interpenetrate (figure 2b).

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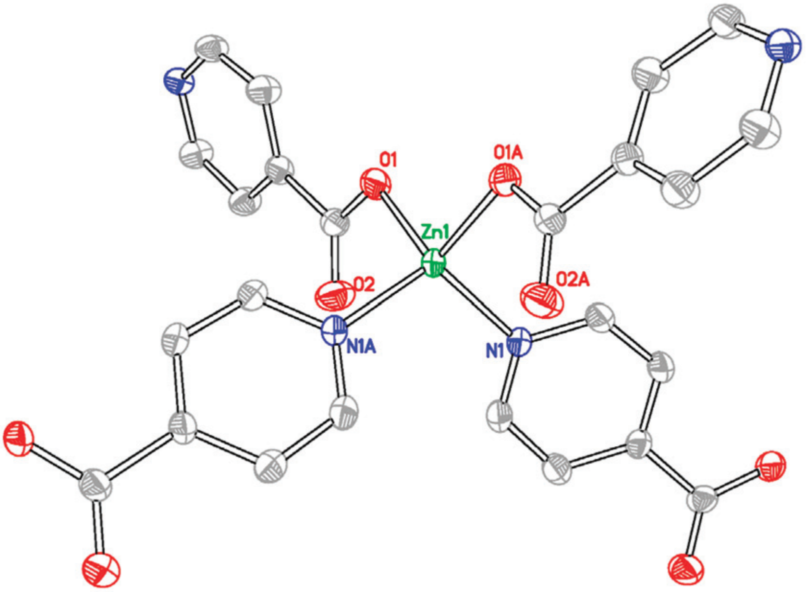

Figure 1. Coordination environment of Zn in **1**. Thermal ellipsoids are drawn at 30% probability level. Hydrogen atoms and water molecules are omitted for clarity.

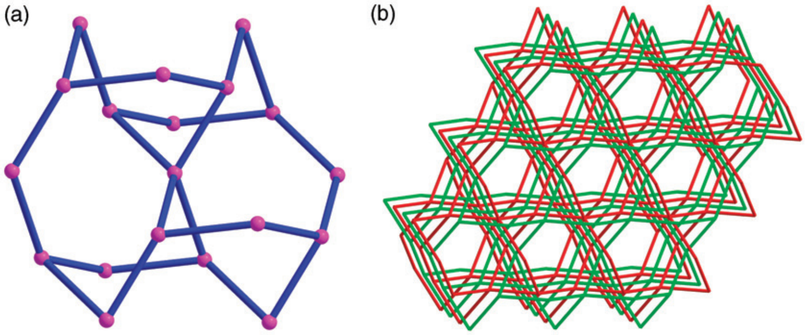

Figure 2. (a) Schematic diagram (OLEX) showing the 4-connected $(6^4 \cdot 8^2)$ network; (b) schematic illustration of the 2-fold interpenetrating $(6^4 \cdot 8^2)$ frame isolated by removing isonicotinic acid groups from **1**.

Compound **1** possesses infinite right-handed and left-handed helical $[ZnL_2]_\infty$ chains (figure S2), which are assembled alternately into the 3-D open framework (figure 3). The right-handed helix is generated around the crystallographic 3_1 axis with a helical pitch of 12.524 Å, which is double the *c*-axis length (figure 4). On turning the helix, the angle between the pyridyl planes at one metal center is 75.738°, leading to helix coiling along the winding axis in a trigonal cross-section. Most exciting, however, is that

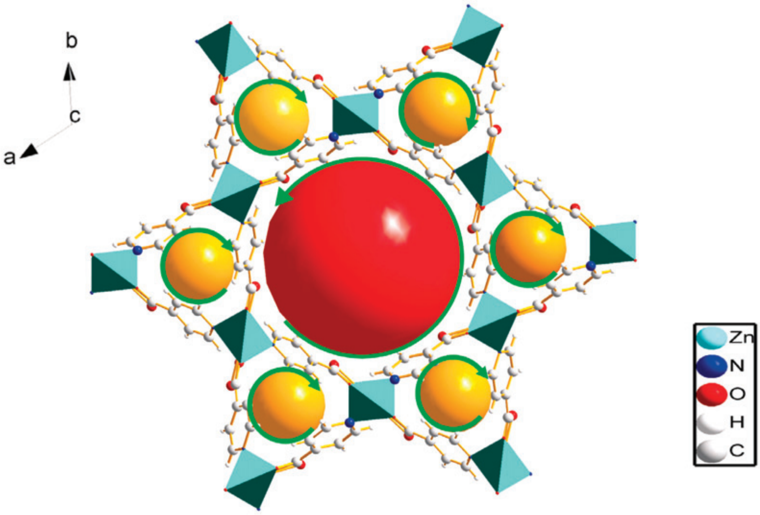

Figure 3. The framework of **1** viewed down the *c*-axis, showing two distinct types of channels in the structure of **1**. Zn^{2+} ions are represented by polyhedra. The small trigonal channels are colored light orange while the large hexagonal channels are colored red with surrounding arrows representing their chirality.

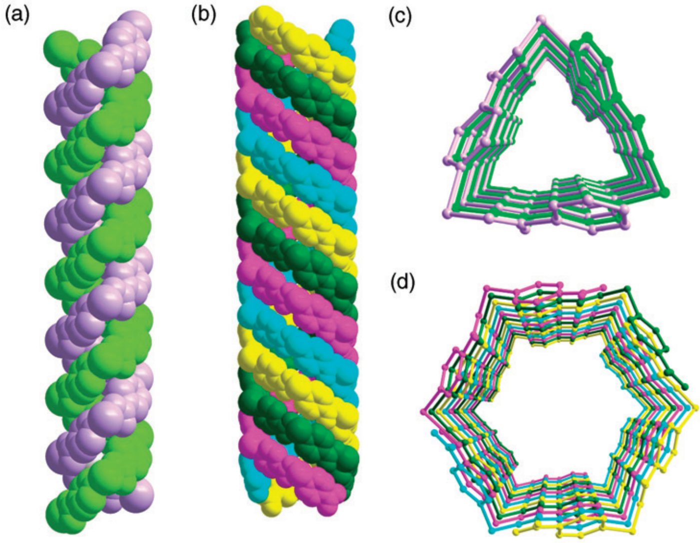

Figure 4. Perspective view of (a) the double right-handed helices in **1**; (b) the quadruple left-handed helices in **1**; (c) side view of the double helices along the *c* axis; (d) side view of the quadruple helices along the *c*-axis.

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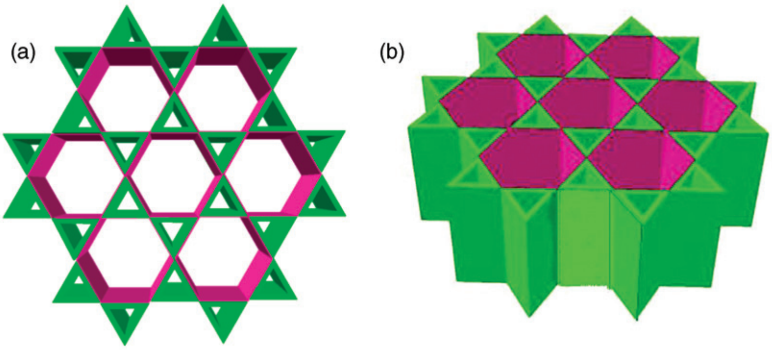

Figure 5. (a) Perspective view of **1**, showing the trigonal and hexagon pores; (b) perspective view of the 3-D structure of **1**.

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quadruple homochiral left-handed helical chains associate in parallel to form the wall of a hexagonal channel (figure 4).

The structure of **1** is built up by two kinds of building blocks, one can be viewed as a hexagonal tube with pores of approximately 13.595–15.512 Å (block **I**) and the other as a trigonal prism (block **II**) (figure 5). Blocks **I** and **II** connect to each other using Zn^{II} as their common vertices. Viewed down the crystallographic *c*-axis, each block **I** shares six vertexes with six adjacent blocks **II**, while each block **II** shares all vertexes with three blocks **I** and three blocks **II**. Two blocks are connected by Zn^{II} and bridging IN ligands to form the 3-D micro-porous framework with two disordered water molecules in the channels (figure S3). This type of network is composed of hexagonal channel **I** and trigonal prism **II**, and both share common borders resulting in a unique helix. The presence of water in the open channels of **1** is also supported by the IR spectrum, which exhibits a broad peak at $3473\,\mathrm{cm}^{-1}$ for O–H stretches. The potential free volume accessible for water, determined by PLATON [15] calculation, is $473\,\mathrm{Å}^3$ per unit cell volume ($1305\,\mathrm{Å}^3$), which represents 36% void per unit volume for **1**.

The peculiar structural feature of **1** is that the hexagonal left-handed channel hosts right-handed double helical water chains through hydrogen bonding of two disordered water molecules. Although the position of the water hydrogens could not be located, the $O\cdots O$ average separation suggests the presence of strong intermolecular interactions between neighboring water molecules. In the direction of the helical channel, each water molecule is hydrogen-bonded to produce 1-D double right-handed helices (figure 6). There appears to be no hydrogen bonding contacts between the guest water molecules and the host framework, providing an explanation for the ease of the removal of water from the lattice, resulting in a compound possibly accessible by other small molecules. One dimensional water morphologies containing chains and tapes play a significant role in many fundamental biological processes as "proton wires" [16–19], however, helical water chains are extremely rare in synthetic crystal hosts. Recently, a hydrogen-bonded helical supramolecular host was found to be anchored by hydrogen bonding to alternate water molecules in a single-stranded, both right- and left-handed, helical chain of water molecules [20, 21]. A hydrogen-bonded helix inside a helical 1-D coordination polymer has also been observed [21]. Such right-handed double helical water chains inside a left-handed helical channel in **1** are scarce in synthetic chiral hosts. This graceful

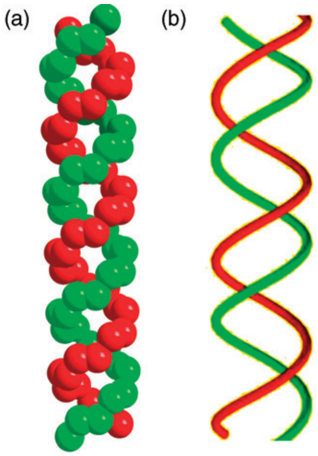

Figure 6. (a) View of the right-handed double helical chains of lattice water molecules; (b) schematic illustration of the homochiral double helical chain.

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double helical stream of water molecules inside helical channel is striking, which is a unique structural feature among existing porous chiral structures.

3.2. *Spectroscopic properties*

Intense fluorescent emissions occur at 391 nm (figure S4, $\lambda_{ex} = 240$ nm) for **1**. In contrast, free IN ligand shows no photoemission at the corresponding excitation [22]. Therefore, the emission of **1** may be attributed to ligand–to–metal charge transfer bands (LMCT). The enhancement of luminescence may be attributed to increase of the rigidity of the ligand, and reduce loss of energy by radiation less decay upon coordination. To examine the chiroptics, the CD spectrum of **1** in solid state was investigated (figure S5). The CD spectrum exhibits several Cotton effects at 203, 209, 213, 216, 223, 228 and 232 nm, indicating that **1** is chiral.

3.3. *Thermogravimetric analysis and dehydration behavior*

Encouraged by the single-crystal X-ray diffraction result, which reveals large free spaces within the framework, we carried out the thermogravimetric analysis (TGA) and powder X-ray diffraction (PXRD) experiments to investigate the stability of **1** (see "Supplementary material" – figure S6 and figure 7). The first weight loss of 11.2% (Calcd 10.4%) corresponding to the loss of crystallization water occurred in the range 50–100°C. At 100–350°C, compound **1** is stable without weight loss. On further heating, the sample decomposed at 350°C, ending above 400°C. The final product is assumed to be ZnO (23.8%), supported by the expected value of 25.7% and also confirmed by the powder X-ray diffraction (PXRD) patterns (see figure 7, beyond 350°C, the diffraction peaks of ZnO appear). Notably, temperature-variable PXRD patterns of **1** also reveal that **1** has quite high thermal stability and retains crystalline form up to at least 350°C. The high thermal stability is attributed to mutual interlocking among the 3-D MOFs by the two independent quartz-like nets.

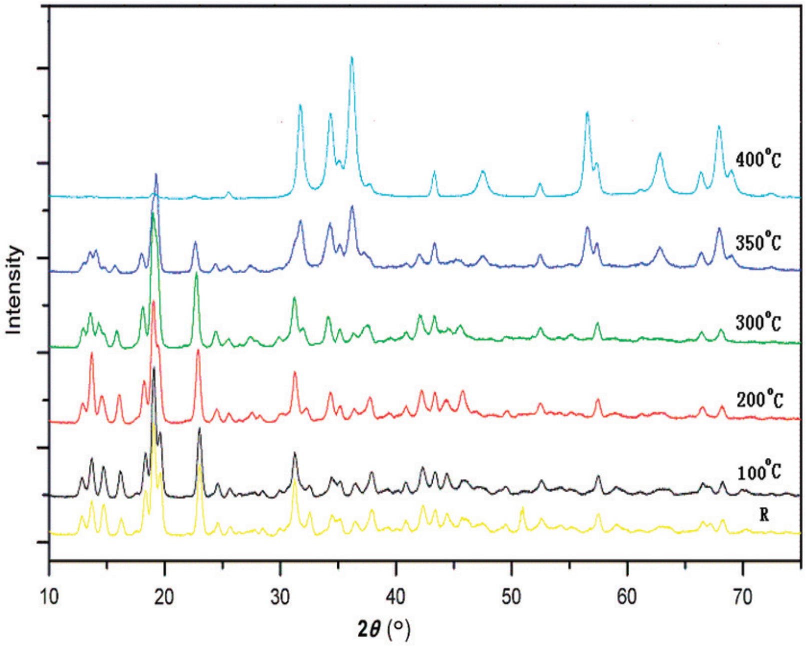

Figure 7. Variable temperature powder diffraction patterns of $Zn(IN)_2 \cdot 2H_2O$. The temperature ramps from room temperature to 400°C. The baselines for each temperature were shifted for clarity.

4. Conclusion

We have obtained a microporous chiral 3-D quartz-like framework, based on the covalent linkage of 1-D hetero-chiral channels. The unusual features observed in **1** (unique chiral channels, network chirality) makes it a rare case of chiral porous compounds and a useful model for exploring the sorption, inclusion, and catalytic properties of porous metal-organic frameworks. Our results together with Lin *et al.*'s shed light on the significance of the structures of achiral unsymmetrical bridging ligands in the process of recognition and crystallization of metal centers into chiral networks. We will continue by replacing other metals and achiral unsymmetrical ligands to synthesize novel chiral and noncentrosymmetric coordination polymers and to study their fascinating properties.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 625471 for **1**. Copies of this information may be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336-033; Email: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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Acknowledgments

This project is supported by the National Natural Science Foundation of China (20773104), the Program for New Century Excellent Talents in University (NCET-06-0891), the Key Project of Chinese Ministry of Education (810243), the Key Project of Key Laboratory of Shaanxi Province (08JZ81), the Natural Science Foundation of Hubei Provinces of China (2009H09), the Natural Scientific Research Foundation of Shaanxi Provincial Education Office of China (08JK311), and the Scientific Research Foundation of Shaanxi Provincial Education Office of China (08JK490).

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