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A 3D chiral metal-organic framework based on left-handed helices containing 3-amino-1 H-1,2,4-triazole ligand



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ABSTRACT

A chiral metal-organic framework, $[Cu(atr)(OH)]^{0.5H_2O} \cdot 0.5en (1)$ (Hatr=3-amino-1 H-1,2,4-triazole, en=ethylenediamine), was constructed via diffusion reaction of the achiral Hatr ligand and CuSO₄ as starting materials. Compound **1** crystallizes in the chiral space group $P_{3_2}21$ and features a porous metal-organic framework with 44.1% solvent-accessible volume fabricated by left-handed helices with a pitch height of l_p =10.442 Å. Six helices gather around in a cycle forming a large honeycomb channel with a 6.58 Å inner diameter. Cu(II) center and atr⁻ ligand regarded as 3-connected nodes, compound **1** can be simplified to a 3-c uninodal {4.12²} (qtz-h) topological network. A gradual decreasing in the magnetic moment depending on temperature decreasing indicates an antiferromagnetic interaction in **1**. The powder XRD confirms the bulk sample is a single crystal pure phase, and the thermogravimetric analysis shows the thermal stability of **1** is up to ca. 240 °C.

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1. Introduction

Metal-organic frameworks (MOFs) materials are in a flourishing development in the last two decades, due to their applications in the fields of catalysis [1], ion-exchange [2], drug-delivery [3], gas sorption and separation [4], and so forth. MOFs based on the construction of inorganic metal center and organic ligands, are superior to other traditional inorganic porous materials in structural tunability, versatility and flexibility [5]. The effort on the MOF structure-property relationship promotes the purposeful design and controlled synthesis of the desired functional materials [6], though it is still a great challenge in predictable structures and properties [7]. One of the important strategies of the structural constructions is the selection of multifunctional organic ligands [8]. Among the numerous ligands employed in MOF constructions, the class of 1,2,4-triazole ligand has been demonstrated to be versatile by virtue of their potential bridging fashions of μ_3 -1 κ N: 2κN: 4κN, μ₂-1κN: 2κN, μ₂-2κN: 4κN [9]. 3-amino-1 H-1,2,4triazole (Hatr) is a two-connecting neutral or three-connecting anionic (deprotonated) ligand in the syntheses of coordination polymers, which vails to achieve open porous metal-organic frameworks [10] and "simple, high-symmetry" structures to study

new topological nets [11]. Its 1,2,4-triazole moiety is just in the region to give spin crossover compounds, and acts as a bridge between metal atoms to mediate magnetic exchange coupling [12]. As a part of our ongoing research dealing with the preparation of MOFs, we present a new 3D chiral MOF compound [Cu(atr)(OH)]·0.5H₂O·0.5en (1). The single-crystal structural analysis shows that the interconnection of the left-handed helices grows into large honeycomb-like open-ended hollow nanotubes with inner diameters of 6.58 Å. Compound 1 is also characterized by FT-IR, magnetism, PXRD and TGA.

2. Experimental

2.1. Materials and physical measurements

The reagents and solvents were used directly as supplied commercially without further purification. The FT-IR spectra were recorded on a Nicolet Magna 750 FT-IR spectrometer with KBr pellets (Aldrich, > 99%, FT-IR grade) in the range 4000-400 cm⁻¹. Elemental analysis of C, H and N was carried out on a Vario EL III elemental analyzer. Thermogravimetric analysis (TGA) was carried out in N₂ atmosphere on a SDT Q600 V8.3 Build 101 instrument with a heating rate of 10 °C min⁻¹ and a N₂ flow rate of 20 cm³ min⁻¹. Powder X-ray diffraction data for the title compound was collected at ambient temperature with a Rigaku D/

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Scheme 1. The synthesis route of 3-amino-1 H-1,2,4-triazole.

Max–3c (Japan) diffractometer (Cu-K $\alpha_{1,2}$ X-radiation, λ_1 = 1.540598 Å and λ_2 =1.544426 Å), equipped with an X'Celerator detector and a flatplate sample holder in a Bragg–Brentano parafocusing optics configuration (40 kV, 40 mA). Intensity data were collected by the step counting method (step being 0.02°), in continuous mode, in the range of $3 \le 2\theta \le 60^\circ$. Variable-temperature, solid-state direct current (DC) magnetic susceptibility data down to 2 K were collected on a Quantum Design PPMS60000 magnetometer. Diamagnetic corrections were applied to the observed paramagnetic susceptibilities using Pascal's constants.

2.2. Synthesis of 3-amino-1 H-1,2,4-triazole

3-Amino-1 H-1,2,4-triazole can be synthesized through condensation reaction of aminoguanidine bicarbonate and mathane acid [13] (Scheme 1) or decarboxylation [14,15]. M. P.=156–159 °C. C₂H₄N₄ elemental analysis (%), Found (calcd): C, 28.59 (28.47); H, 4.78 (4.55); N, 66.65 (66.81). FT-IR data (in KBr, cm⁻¹): 3412(w), 333(w), 3210(w), 3053(w), 2930(w), 2773(w), 2717(w), 1639(s), 1589(m), 1533(s), 1421(m), 1365(w), 1267(m), 1205(m), 1043(s), 965(w), 869(w), 828(w), 724(w), 637(w).

2.3. Synthesis of [Cu(atr)(OH)] • 0.5H₂O • 0.5en (1)

pH of Hatr ethanol solution (0.085 g, 1 mmol) was adjusted to 8 by ethylenediamine. Aqueous solution containing $CuSO_4 \cdot 5H_2O$ (0.25 g, 1 mmol) was covered with the Hatr solution in a tube. Over a period of approximate 20 d, the blue crystals were obtained in the yield of 29.7% (0.06 g). Elemental analysis (%), Found (calcd): C, 17.85 (17.73); H, 4.41 (4.58); N, 34.52 (34.65). FT-IR data (in KBr, cm⁻¹): 3399 (b, vs), 2975 (m), 2895 (m), 1631 (m), 1555 (m), 1518 (w), 1451 (w), 1382 (w), 1316 (w), 1271 (w), 1090 (s), 1050 (s), 881 (m), 804 (w), 736 (m), 627 (w), 495 (w).

2.4. Single crystal X-ray diffraction

Single crystals of compound 1 were manually harvested from crystallisation vials and mounted on Hampton Research CryoLoops using FOMBLIN Y perfluoropolyether vacuum oil (LVAC 25/6, purchased from Aldrich) [16] with the help of a Stemi 2000 stereomicroscope equipped with Carl Zeiss lenses. Data were collected on a Rigaku Mercury CCD diffractometer equipped with a graphite-monochromated Mo- $K\alpha$ radiation (λ =0.71073 Å) at 293 (2) K. The intensity data were collected by the ω scan technique and were reduced using CrystalClear program [17]. The crystal structure of compound 1 was solved by direct method using SHELXTL[™] package of crystallographic software [18] and refined by full-matrix least-squares technique on F^2 . All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located at geometrically calculated positions to their carrier atoms and refined with isotropic thermal parameters included in the final stage of the refinement. A summary of the structural determination and refinement for compound 1 is listed in Table 1. The selected bond distances and angles are listed in Table 2.

2.5. Topology

According to A. F. Wells' topology definition [19] and the MOF structural features, Cu(II) atom and atr[–] ligand in compound

Table	1	

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

Empirical formula	$C_3H_9CuN_5O_{1.5}$	
Color and Habit	Blue prism	
Crystal Size (mm ³)	$0.24 \times 0.20 \times 0.20$	
Crystal system	hexagonal	
Space group	P3221	
a (Á)	11.9286(6)	
b (Å)	11.9286(6)	
c (Å)	10.4690(11)	
V (Å ³)	1290.07(16)	
Ζ	6	
Fw	202.69	
$D_{\rm calcd} ({\rm Mgm^{-3}})$	1.565	
$\mu ({\rm mm^{-1}})$	2.496	
F (000)	618	
θ (°)	3.42 to 25.01	
Reflections measured	8415	
Independent reflections	$1503 (R_{int}=0.0229)$	
Observed Reflection $[I > 2\sigma(I)]$	1473	
Final R_{1} , wR_{2} indices (obs.)	0.0577,0.1529	
R_{1} , wR_{2} indices (all)	0.0583, 0.1540	
S	1.132	
$(\Delta/\sigma)_{\rm max/min}$	0.017,0.000	
$(\Delta \rho)_{\text{max/min}} (e \text{\AA}^{-3})$	1.024, -0.524	

 $R_1 \!=\! (\varSigma ||F_o|\!-\!|F_c|\!\mid / \varSigma |F_o|). wR_2 \!=\! [\varSigma (w(F_o^2\!-\!F_c^2)^2) / \varSigma (w|F_o^2|^2)]^{1/2}$

 Table 2
 Selected bond distances (Å) and bond angles (°) of compound 1.

Cu1-N11A	1.9782(19)	Cu1-N12B	1.989(2)
Cu1-N14	1.982(2)	Cu1-01	2.009(2)
N11A-Cu1-N14	176.50(8)	N11A-Cu1-O1	88.75(8)
N11A-Cu1-N12B	90.90(6)	N14-Cu1-O1	89.08(9)
N14-Cu1-N12B	91.42(8)	N12B-Cu1-O1	176.93(9)

Symmetry codes: A=*x*-*y*+1, -*y*+1, -*z*+1/3; B=-*y*+1, *x*-*y*+1, *z*-1/3.

1 were both considered as 3-connected nodes and the topological network of compound **1** was calculated using the ADS program of the TOPOS **4.0** Professional structure-topological program packages.

3. Results and discussion

3.1. Structural description of compound 1

Much interest has been focused on helical supramolecular architectures with potential important applications in advanced materials such as optical devices [20]. Though the achiral approach almost always leads to a racemic mixture [21], chiral crystals can still occur through self-assembly, which is not surprising even if simple salts can crystallize in chiral space group [22]. The inherent chirality of this architecture comes from spatial disposition rather than the presence of chiral centers, which can have important applications in a spontaneous splitting of the racemate on crystallization. In this paper, compound **1** features a chiral metal-organic framework fabricated by left-handed helices based on foundational repeating neutral [Cu(atr)(OH)] units. Compound **1**



Fig. 1. The coordination sphere of Cu(II) center (a). *Symmetry codes*: A=x-y+1, -y+1, -z+1/3; B=-y+1, x-y+1, z-1/3; the left-handed helix viewed along the *c* axis (b); Every six helices connects through N11–Cu–N14 bridges to generate a channel along the *c* axis (c); viewed along the *a* axis (d); Honeycomb tubular channels formed in the [Cu(atr)(OH)] framework along the *c* axis (e). Hydrogen atoms and guest molecules are omitted for clarity.

crystallizes in a chiral space group of P3₂21, which presents the chiral nature of the complex. In its asymmetric unit, the copper(II) center has a square-like planar arrangement with maximum deviation of copper(II) from the best least-squares plane formed by O1, N11 A, N12B and N14 (A = x - y + 1, -y + 1, -z + 1/3; B = -y + 1, x-y+1, z-1/3) being 0.6342 Å (Fig. 1a). The atr⁻ ligand acts as deprotonated μ_3 -bridging ligand coordinated to three separated copper(II) centers. The ligands bridge copper(II) centers with 2and 4-positioned nitrogen in alternation around a central 3₂ helical axis to form a left-handed helix with a pitch height of $l_p = 10.442$ Å (Fig. 1b). The chirality arises from a twist of the [Cu (atr)(OH)] units spontaneously spatially depositing according to the requirement of the chiral space group P3₂21. Six helices connect each other by the bridges of N11-Cu-N14 bonds into a cycle to generate an open-ended channel along the *c* axis (Fig. 1c and d). The growth of the helices gives a 3D framework containing honeycomb channels as shown in Fig. 1e. The channels can be viewed as consisting of hollow tubes extending along the *c* axis (Fig. 1c and d). The honeycomb channels possess hydrophilic internal cavities, in which the guest ethylenediamine molecules "anchor" through H-bonds such as $N1-H \cdot \cdot \cdot N2C = 2.954(5)$ Å, 134.6° , C = -x + y, 1 - x, 1/3 + z, resulting in hydrophilic internal cavities with 24.1% solvent-accessible volume and a 5.8 Å inner diameter. Omitting the guest molecules, the channels have a 9.18 Å diameter. minusing the van der Waals radii of O and H (1.4 and 1.2 Å, respectively), the effective aperture of the channels is 6.58 Å. The total solvent-accessible volume of the channels in the unit is 569.4 Å³ accounting for 44.1% solvent-accessible volume calculated by PLATON [23].

We will have a better insight into the nature of this intricate architecture by topological analysis. Regarding on the structural features of compound **1**, apparently, Cu(II) center and atr⁻ ligand could be both regarded as 3-connected nodes. The vertex point symbols are both $\{4.12^2\}$ for the two 3-coonected nodes. Therefore, the overall structure of compound **1** can be simplified to a 3-c uninodal $\{4.12^2\}$ (or qtz-h) topological network (Fig. 2) [24].



Fig. 2. The 3-c uninodal $\{4.12^2\}$ or qtz-h topological network of compound **1** with Cu(II) center and atr⁻ ligand regarded as 3-connected nodes.



Fig. 3. Plot of χ_m vs. T over 2–300 K at a field of 1 T showing a Curie-Weiss paramagnetic behavior of the title complex.



Fig. 4. Comparison of powder XRD pattern of compound ${\bf 1}$ to the one simulated from single crystal structure data.



3.2. Magnetic property of compound 1

The magnetic behavior of the present compound is shown in Fig. 3 in the form of χ_m vs. *T* and μ_{eff} vs. *T* plots. A nonlinear fit via $\chi_m = C/(T-\theta) + \chi_0$ to the data above 40 K reveals a Curie-Weiss behavior with the Curie constant $C=0.056(1) \text{ cm}^3 \text{ mol}^{-1}$ K, the Weiss constant $\theta = -24.8(10)$ K, and the background susceptibility $\chi_0 = 8.0(4) \times 10^{-5} \text{ cm}^3/\text{mol}$. An effective magnetic moment of 0.67 µB can thus be obtained for each Cu center via $\mu_{eff} = 2.828 \text{ (C)}^{1/2} \text{ µB}$, which is less than the expected 1.73 µB for the free-ion ground state ($^{2}D_{2/5}$) of Cu(II) with one localized unpaired D-electron. A gradual decreasing in the magnetic moment is observed as the temperature is decreasing, indicating the presence of an antiferromagnetic interaction.

3.3. Powder XRD and thermogravimetric analysis of compound 1

The experimental power XRD pattern for compound **1** matches well with the simulated one from single crystal structure data (Fig. 4), indicating that bulk sample of compound **1** was isolated as a single crystal pure phase. The thermogravimetric analysis of compound **1** shows a thermal stability up to ca. 240 °C, and the overall weight loss appears as a three-step process (Fig. 5). In the

temperature range of 72–95 °C, a small weight loss of c.a. 4.91% occurs, attributed to the lattice water loss with the calculated value of 4.44%. The second loss of 14.13% starting at c.a. 176 °C concerns with the release of the confined en molecules in the cavities of the skeleton framework, close to calculated weight loss of 14.83%. The higher releasing temperature of en molecules is related with the strong H-bond of amino groups with triazole moieties, which is supported by the single crystal structure analysis. The last considerable weight loss (ca. 42.14%) at about 240 °C attributes to the destruction of the [Cu(atr)(OH)] skeleton due to the oxidation of the organic component. The remnant 38.82% product is CuO, according with the calculated value of 39.25%.

4. Conclusion

The diffusion reaction of achiral 3-amino-1 H-1,2,4-triazole ligand with CuSO₄ produced a chiral metal-organic framework, [Cu(atr)(OH)][•]0.5H₂O[•]0.5en (1). The chirality of the compound comes from the spatial deposition required by the chiral space group P3₂21. The atr⁻ ligand connects copper(II) centers alternately with 2- and 4-positioned N atom to shape a left-handed helix along the *c* axis. Through the N11–Cu–N14 bridges, six lefthanded helices gather around in a cycle forming a honeycomb channel with a 6.58 Å inner diameter, and further grow into a porous 3D architecture with 44.1% solvent-accessible volume. The overall structure of compound **1** is simplified as a 3-c uninodal {4.12²} (or qtz-h) topological network with Cu(II) center and atr⁻ ligand regarded as 3-connected nodes. The negative Weiss constant θ and the decreasing magnetic moment with decreasing temperature indicate an antiferromagnetic interaction in compound 1. The purity of the bulk sample of compound 1 is demonstrated by the similarity of experimental and simulated power XRD patterns, and compound **1** shows a thermal stability up to 240 °Cs.

Supplementary material

CCDC-277659 contains the supplementary crystallographic data of compound 1 in this paper. The crystallographic data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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