Electronic supplementary information

Synthesis, structure and magnetic properties of copper(II) azide

Taqing Shi,[a] Ye Xu,[b] Ya-Jing Zou [b] and Zhao-Xi Wang[b]*

[a] School of Pharmacy, Guangdong Medical University, Dongguan 523800, People's Republic of China.
[b] Department of Chemistry, Center for Supramolecular Chemistry and Catalysis, Innovative Drug Research Center, Shanghai University, Shanghai 200444, People's Republic of China.

Experimental Section

Materials and Methods

All chemicals were of reagent grade and used as received from commercial sources without further purification. Infrared spectra were recorded with a Nicolet A370 FT-IR spectrometer using KBr pellets in the 4000–400 cm^{-1} region. Variable-temperature magnetic susceptibility measurements were taken at an applied field of 2 KOe on a Quantum Design MPMS-XL7 SQUID magnetometer working in the temperature range of 300–1.8 K. The molar magnetic susceptibilities were corrected for the diamagnetism estimated from Pascal's tables and for the sample holder by previous calibration.

Caution! Azide derivatives are potentially explosive, only a small amount of material should be prepared, and it should be handled with proper care.

Preparation of 1

A mixture of $CuCl_2 \cdot 2H_2O$ (51.2 mg, 0.3 mmol), NaN_3 (65.7mg, 1 mmol), hydroxylamine hydrochloride (69.8 mg, 1 mmol) and water 10 ml was sealed in a 15 ml Teflon-lined stainless-steel reactor, which was heated at 110 ºC for 72 hours. Upon cooling to room temperature at a rate of 10 °C h^{-1}, brown prism crystals suitable for X-ray structure study were obtained with 47% yield based on Cu(II). Elemental analysis calcd (%) for CuN_6 (147.60): N, 56.94. Found: N, 56.72. Selected IR data (KBr, cm^{-1}): ν = 2037, and 2052 for the azide groups.

Tables S1. Crystallographic data and details of refinements for **1**.

Temperature (K)	295(2)	153(2)
Empirical formula	CuN_6	CuN_6
Formula weight	147.60	147.60
Crystal system	Orthorhombic	Orthorhombic
Space group	*Pnma*	*Pnma*
a (Å)	13.486(3)	14.927(10)
b (Å)	3.0841(6)	3.087(2)
c (Å)	9.0857(18)	7.812(5)
V (Å^3)	377.89(13)	360.0(4)
Z	4	4
ρ_{calcd} (g/cm^3)	2.594	2.723
μ (mm^{-1})	5.608	5.887
F(000)	284	284
GOF (F^2)	1.072	1.111
$R1^a$ [$I>2\sigma(I)$]	0.0193	0.0975
$wR2^b$ [$I>2\sigma(I)$]	0.0457	0.2245

^{a} $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, ^{b} $wR_2 = [\Sigma w(|F_o^2| - |F_c^2|)^2/\Sigma w(|F_o^2|)^2]^{1/2}$

Tables S2. Selected bond distances (Å) and bond angles (º) for **1**.

	295(2)	153(2)
Cu(1)-N(1)	2.003 (2)	2.000(7)
Cu(1)-N(4)	1.992 (7)	1.981(6)
Cu(1)-N(1A)	2.003(2)	2.000(7)
Cu(1)-N(4C)	1.992(7)	1.981(6)
Cu(1)-N(1B)	2.696(8)	2.819(9)
Cu(1)-N(6D)	2.565(6)	2.475(0)
Cu(1)-N(1)-Cu(1A)	100.67(10)	101.0(5)
Cu(1)-N(4)-Cu(1B)	101.41(11)	102.4(4)

Symmetry codes: A: x, -1+y, z; B: -x, -1/2+y, 2-z; C: x, 1+y, z; D: 1/2-x, -y, -1/2+z.

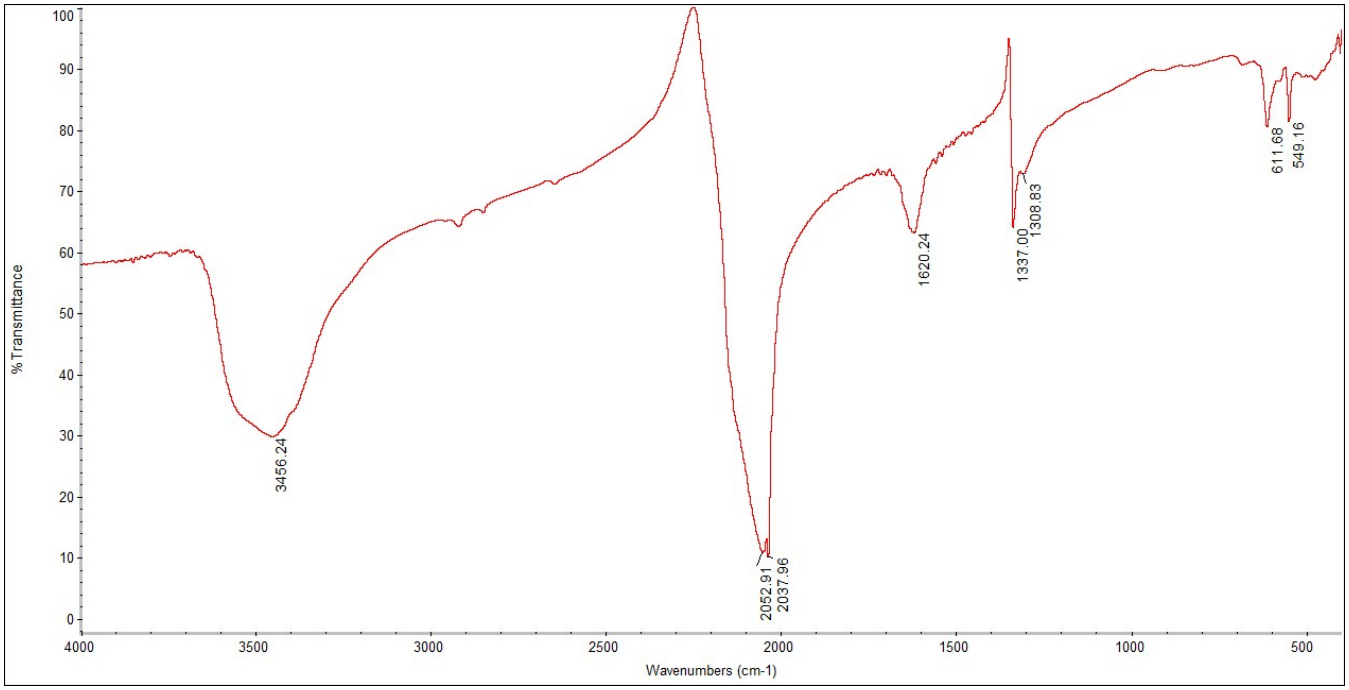

Fig. S1. FT-IR spectra of **1**

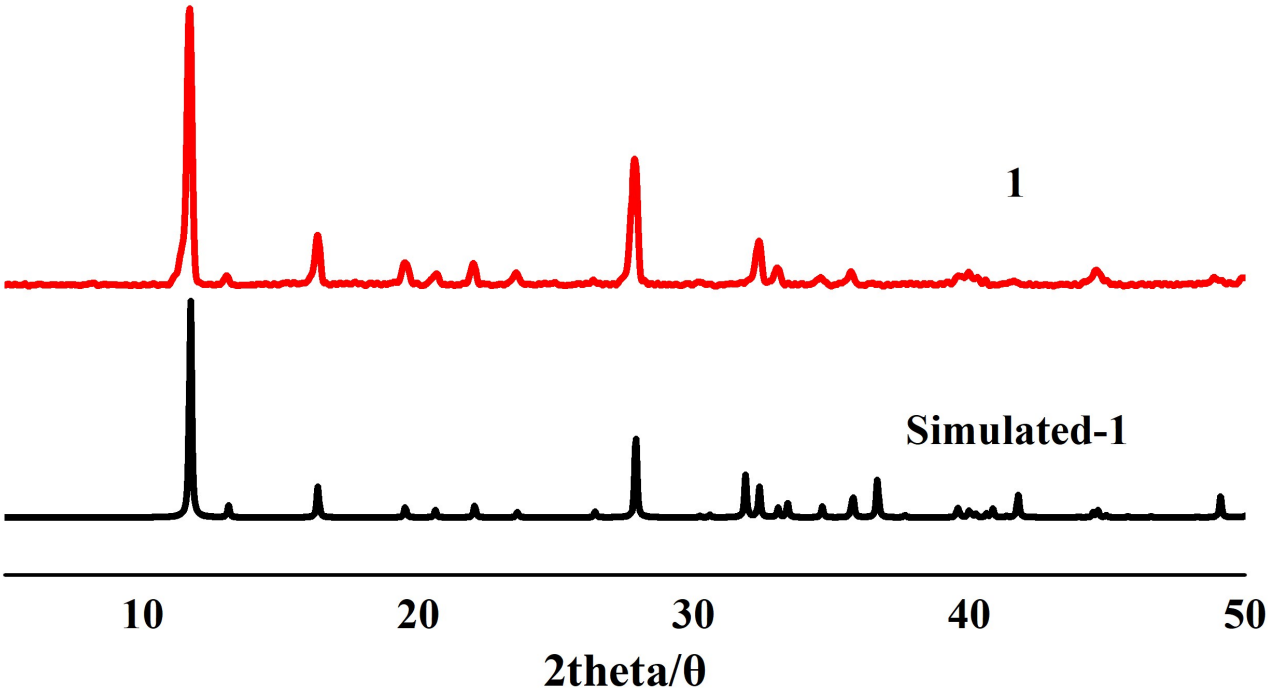

Fig. S2. PXRD patterns of **1**

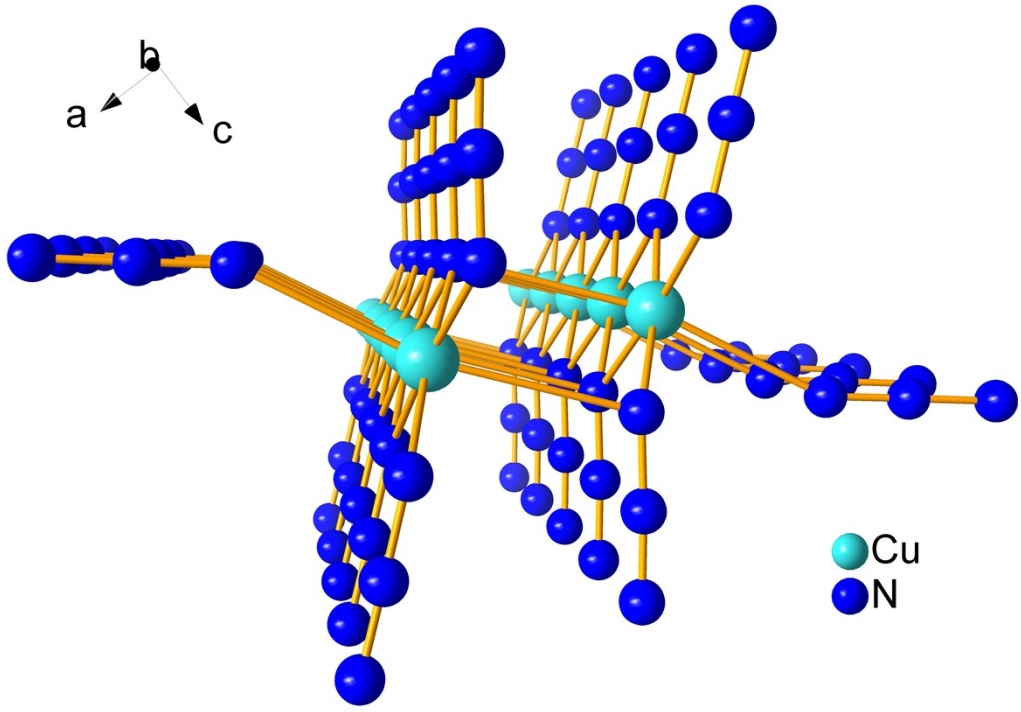

Fig. S3. Double chain structure of **1**.

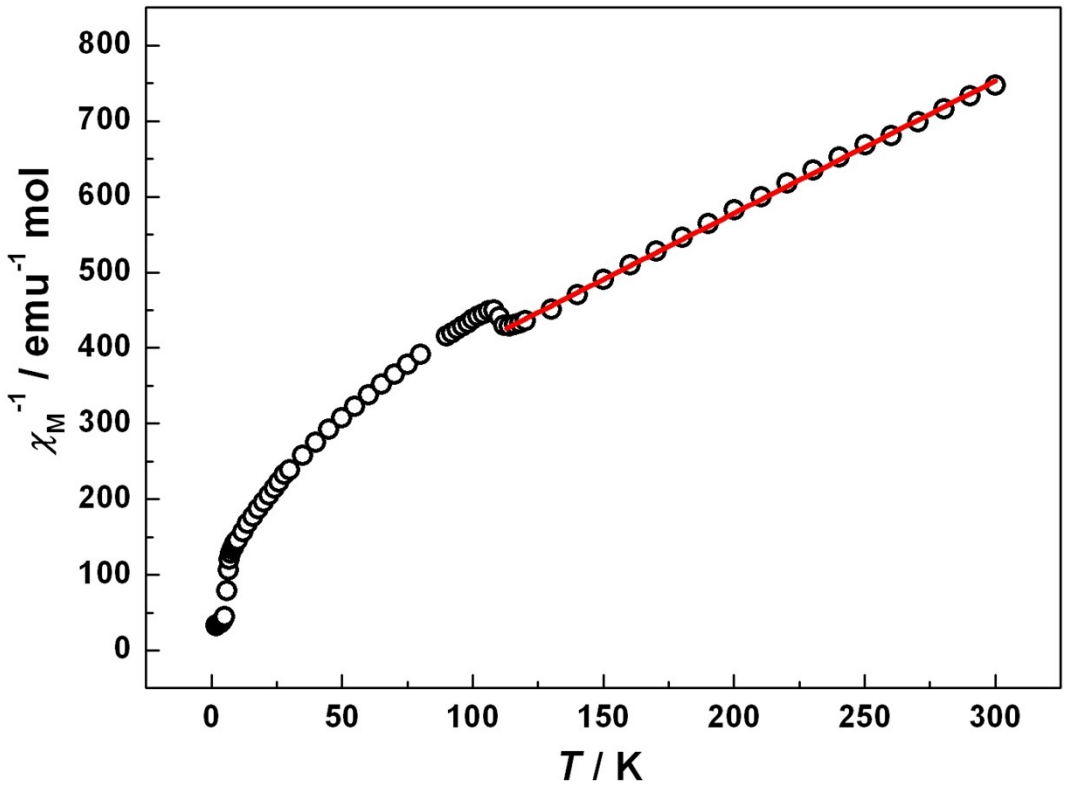

Fig. S4. Plot of χ_M-T for **1**, the solid line shows the best fit curve by Curie-Weiss law.

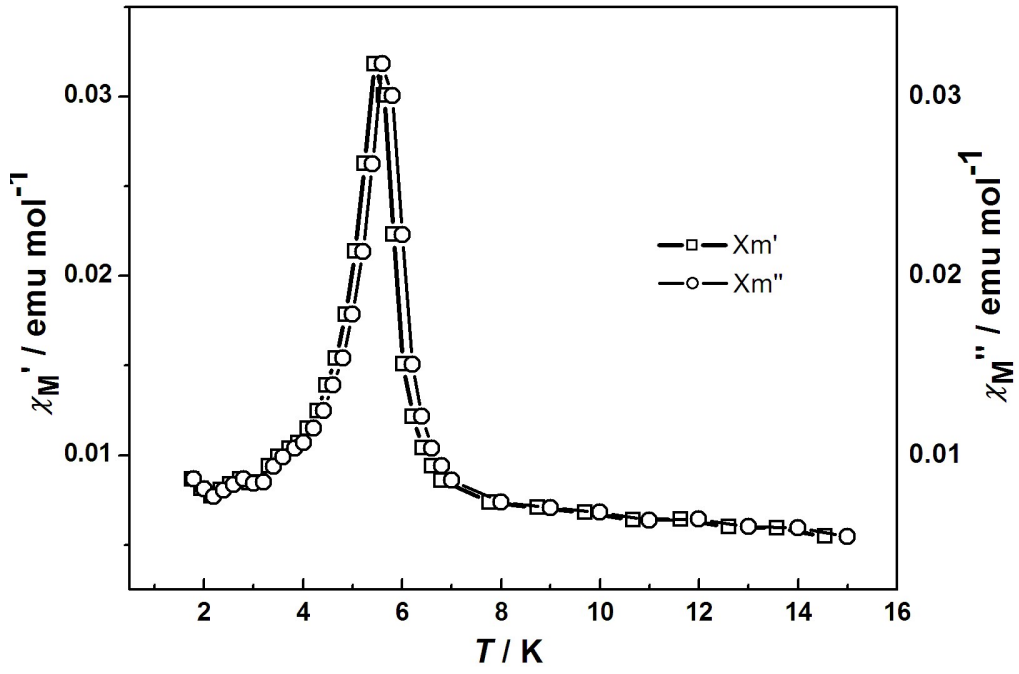

Fig. S5. Temperature dependence of real (χ′) and imaginary (χ′′) ac magnetic susceptibilities in a zero applied dc field with an oscillating field of 3 Oe at frequency of 10 Hz for **1**.