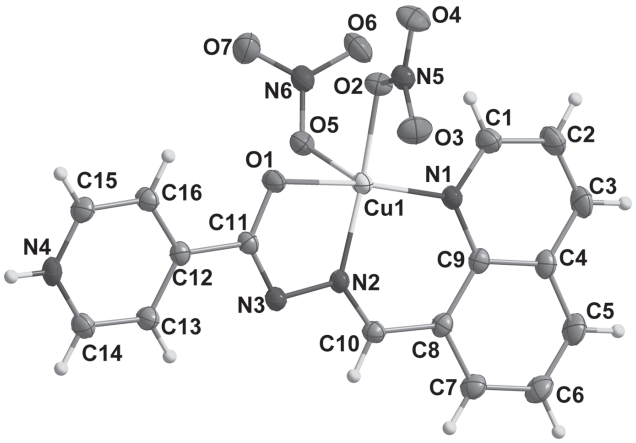
Zhi-Hong Xu, Ye Shen* and Wei-Na Wu

Crystal structure of dinitrato-(*N′*-((quinolin-8-yl)methylene)isonicotinohydrazide κ³-*N*,*N′*,*O*) copper(II), $C_{16}H_{12}N_6O_7Cu$



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Abstract

$C_{16}H_{12}N_6O_7Cu$, monoclinic $P2_1/n$, $a = 7.832(4)$ Å, $b = 15.641(9)$ Å, $c = 14.747(8)$ Å, $\beta = 101.365(9)°$, $V = 1770.9(17)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0338$, $wR_{ref}(F^2) = 0.0885$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title complex was obtained by reaction of *N′*-((quinolin-8-yl)methylene)isonicotinohydrazide (5 mmol) [5] with equimolar of $Cu(NO_3)_2$ in methanol/DMF (5 mL, v:v = 1:1)

***Corresponding author: Ye Shen,** Institute of Resources & Environment, Henan Polytechnic University, Jiaozuo, 454000, P.R. China, e-mail: wllishy@126.com
Zhi-Hong Xu: Key Laboratory of Chemo/Biosensing and Detection, School of Chemistry and Chemical Engineering, Xuchang University, 461000, P.R. China
Wei-Na Wu: College of Chemistry and Chemical Engineering, Henan Polytechnic University, Jiaozuo, 454000, P.R. China

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.20 × 0.15 × 0.12 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.29 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8831, 3121, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2538
$N(param)_{refined}$:	271
Programs:	Bruker programs [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.44543(4)	0.22709(2)	0.10763(2)	0.03275(13)
O1	0.3401(3)	0.11245(11)	0.11246(12)	0.0415(5)
O2	0.4612(3)	0.24076(12)	0.24308(13)	0.0384(5)
O3	0.6932(3)	0.16298(15)	0.24626(15)	0.0590(6)
O4	0.6172(3)	0.20022(14)	0.37422(14)	0.0560(6)
O5	0.1556(3)	0.29468(13)	0.08581(13)	0.0443(5)
O6	0.1927(3)	0.37797(15)	0.20608(15)	0.0628(7)
O7	−0.0382(3)	0.29989(16)	0.17268(18)	0.0672(7)
N1	0.5622(3)	0.33908(14)	0.08920(15)	0.0343(5)
N2	0.4449(3)	0.18990(13)	−0.01778(14)	0.0306(5)
N3	0.3683(3)	0.10978(13)	−0.04119(15)	0.0348(5)
N4	0.0729(3)	−0.16474(14)	−0.00831(16)	0.0394(6)
H4A	0.0161	−0.2120	−0.0173	0.047*
N5	0.5956(3)	0.19978(14)	0.28940(16)	0.0379(6)
N6	0.1029(3)	0.32529(14)	0.15593(16)	0.0381(6)
C1	0.5869(4)	0.39371(19)	0.1602(2)	0.0501(8)
H1A	0.5462	0.3779	0.2129	0.060*
C2	0.6696(5)	0.4728(2)	0.1608(2)	0.0570(9)
H2A	0.6802	0.5087	0.2119	0.068*
C3	0.7338(4)	0.49651(19)	0.0861(2)	0.0506(8)
H3B	0.7922	0.5483	0.0859	0.061*
C4	0.7116(4)	0.44219(17)	0.0084(2)	0.0397(7)
C5	0.7755(4)	0.46603(19)	−0.0718(2)	0.0507(8)
H5A	0.8351	0.5174	−0.0722	0.061*
C6	0.7508(4)	0.4150(2)	−0.1477(2)	0.0510(8)
H6A	0.7946	0.4308	−0.1995	0.061*
C7	0.6589(4)	0.33846(18)	−0.1474(2)	0.0422(7)
H7A	0.6407	0.3047	−0.2004	0.051*
C8	0.5936(4)	0.31061(17)	−0.07152(18)	0.0323(6)
C9	0.6213(3)	0.36275(16)	0.01047(18)	0.0323(6)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C10	0.5027(4)	0.22863(16)	−0.08284(18)	0.0331(6)
H10A	0.4855	0.2027	−0.1407	0.040*
C11	0.3229(3)	0.07642(16)	0.03234(17)	0.0308(6)
C12	0.2411(3)	−0.01046(16)	0.01917(18)	0.0310(6)
C13	0.2069(4)	−0.04887(18)	−0.06820(19)	0.0409(7)
H13A	0.2419	−0.0224	−0.1180	0.049*
C14	0.1211(4)	−0.12598(18)	−0.0801(2)	0.0437(7)
H14A	0.0964	−0.1513	−0.1383	0.052*
C15	0.1104(4)	−0.13215(18)	0.0767(2)	0.0419(7)
H15A	0.0797	−0.1619	0.1257	0.050*
C16	0.1945(4)	−0.05431(17)	0.09239(18)	0.0372(7)
H16A	0.2198	−0.0315	0.1517	0.045*

solution. Crystals were grown by evaporating the reaction solution at room temperature.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [2]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Discussion

Schiff base derived from 8-formylquinoline and their metal complexes have been widely investigated due to their excellent biological activities [3, 4]. Our previous work has shown that *N′*-((quinolin-8-yl)methylene) isonicotinohydrazide can form stable complexes with $Cu(OAc)_2$ and $Zn(OAc)_2$ [5]. In this work, the title complex from $Cu(NO_3)_2$ was synthesized and characterized by X-ray diffraction.

In the title crystal structure, the central Cu(II) ion is surrounded by two monodentate coordinated nitrate anions and one NO_2 donor set of the hydrazone ligand L, thus giving a distorted tetragonal pyramid coordination geometry (the geometric index τ being 0.083 according to the Addison rule [5]). The equatorial plane is made up of O1, O2, N1 and N2 atoms, while O5 atom occupies the axial position. It should be noted that the carbonyl bond distance of C11—O1 is 1.292(3) Å, indicating that carbonyl bond of the ligand HL is deprotonated and in enolic form. Consequently, the pyridinyl nitrogen atom is protonated. A fact which has been verified by difference Fourier synthesis. In the crystal structure, two molecules are linked with each other into a centrosymmetric dimer by a pair of intermolecular N—H···O hydrogen bonds.

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