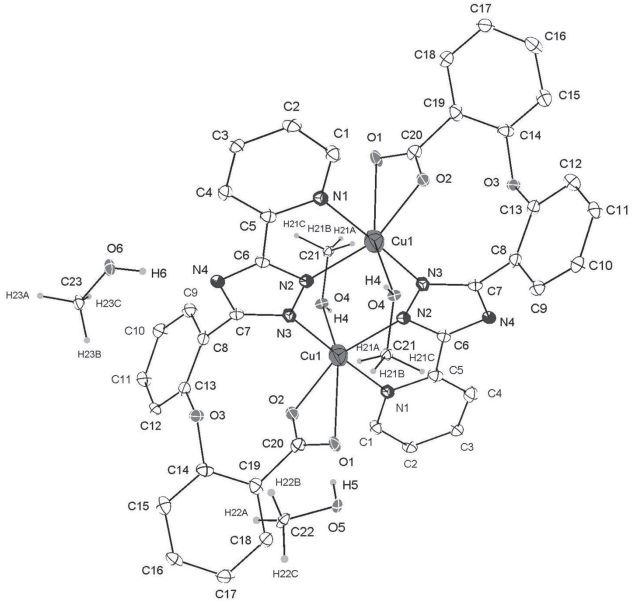
Open Access

Guo Qiang Li, Jia Hong He, Cai Yun Hui and Qiang Xu*

Crystal structure of dimethanolo-bis[μ-(2-(2-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)phenoxy) benzoato)-κ^5*O*,*O*′,*N*:*N*′,*N*″]dicopper(II) — methanol (1/2), $C_{46}H_{48}Cu_2N_8O_{12}$



DOI 10.1515/ncrs-2016-0087
Received March 21, 2016; accepted July 13, 2016; available online August 10, 2016

Abstract

$C_{46}H_{32}Cu_2N_8O_{12}$, monoclinic, $P2_1/c$, $a = 9.5638(9)$Å, $b = 20.2142(19)$ Å, $c = 12.0825(11)$ Å, $\beta = 94.696(1)°$, $V = 2328.0(4)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0273$, $wR_{ref}(F^2) = 0.0711$, $T = 100$ K.

CCDC no.: 1458701

***Corresponding author: Qiang Xu,** Chongqing Key Laboratory of Environmental Materials and Remediation Technologies, Chongqing University of Arts and Sciences, Chongqing 402160, P.R. China, e-mail: 1050573038@qq.com
Guo Qiang Li and Jia Hong He: Chongqing Key Laboratory of Environmental Materials and Remediation Technologies, Chongqing University of Arts and Sciences, Chongqing 402160, P.R. China
Cai Yun Hui: Chongqing University of Arts and Sciences, Chongqing 402160, P.R.China

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

Table 1: Data collection and handling.

Crystal:	Blue block Size 0.20 × 0.20 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	9.9 cm^{-1}
Diffractometer, scan mode:	Bruker, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16067, 4102, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3617
$N(param)_{refined}$:	313
Programs:	SHELX [7], OLEX2 [8], Bruker programs [9]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cu1	−0.09035(2)	0.568887(11)	0.087514(19)	0.01284(8)
O1	−0.01064(17)	0.31597(7)	0.01516(12)	0.0282(4)
O2	0.03287(13)	0.34682(6)	−0.15658(11)	0.0156(3)
O3	−0.25657(13)	0.32269(6)	−0.22656(11)	0.0152(3)
O4	−0.16599(15)	0.49913(7)	0.21776(13)	0.0243(3)
O5	−0.1727(2)	0.24296(11)	0.16117(16)	0.0591(6)
O6	−0.58400(14)	0.48165(8)	−0.29730(13)	0.0272(4)
H4	−0.2452(16)	0.5032(9)	0.2458(18)	0.036*
N1	−0.28869(16)	0.60247(8)	0.05744(13)	0.0138(3)
N2	−0.15575(16)	0.50953(7)	−0.04140(13)	0.0131(3)
N3	−0.10135(16)	0.46748(8)	−0.11568(13)	0.0132(3)
N4	−0.32291(16)	0.49055(7)	−0.17787(13)	0.0136(3)
H5	−0.1383	0.2691	0.1166	0.089*
H6	−0.5054	0.4851	−0.2613	0.041*
C1	−0.3474(2)	0.65068(10)	0.11391(17)	0.0180(4)
H1	−0.2919	0.6733	0.1708	0.022*
C2	−0.4865(2)	0.66853(10)	0.09182(18)	0.0217(5)
H2	−0.5257	0.7032	0.1324	0.026*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C3	−0.5675(2)	0.63534(10)	0.01008(19)	0.0238(5)
H3	−0.6638	0.6461	−0.0049	0.029*
C4	−0.5070(2)	0.58595(10)	−0.05028(18)	0.0200(4)
H4A	−0.5605	0.5631	−0.1079	0.024*
C5	−0.36740(19)	0.57088(9)	−0.02453(16)	0.0141(4)
C6	−0.28675(19)	0.52182(9)	−0.08154(16)	0.0131(4)
C7	−0.20441(19)	0.45778(9)	−0.19708(16)	0.0125(4)
C8	−0.18669(19)	0.42328(9)	−0.30354(16)	0.0144(4)
C9	−0.1497(2)	0.45979(10)	−0.39437(17)	0.0206(5)
H9	−0.1356	0.5062	−0.3870	0.025*
C10	−0.1331(2)	0.42968(11)	−0.49514(19)	0.0262(5)
H10	−0.1088	0.4552	−0.5567	0.031*
C11	−0.1521(2)	0.36178(11)	−0.50590(18)	0.0261(5)
H11	−0.1384	0.3408	−0.5745	0.031*
C12	−0.1910(2)	0.32445(10)	−0.41724(17)	0.0211(5)
H12	−0.2046	0.2781	−0.4249	0.025*
C13	−0.20966(19)	0.35547(9)	−0.31715(16)	0.0147(4)
C14	−0.2118(2)	0.25724(9)	−0.20824(16)	0.0147(4)
C15	−0.2923(2)	0.20516(10)	−0.25134(17)	0.0189(4)
H15	−0.3762	0.2130	−0.2971	0.023*
C16	−0.2479(2)	0.14117(10)	−0.22633(18)	0.0221(5)
H16	−0.3013	0.1047	−0.2561	0.027*
C17	−0.1262(2)	0.12984(10)	−0.15820(18)	0.0240(5)
H17	−0.0955	0.0858	−0.1430	0.029*
C18	−0.0496(2)	0.18280(10)	−0.11240(18)	0.0222(5)
H18	0.0318	0.1749	−0.0637	0.027*
C19	−0.0912(2)	0.24752(9)	−0.13724(16)	0.0161(4)
C20	−0.01664(19)	0.30736(10)	−0.08706(17)	0.0164(4)
C21	−0.1423(2)	0.42920(11)	0.2076(2)	0.0278(5)
H21A	−0.1311	0.4183	0.1298	0.042*
H21B	−0.2226	0.4049	0.2327	0.042*
H21C	−0.0571	0.4168	0.2535	0.042*
C22	−0.3001(3)	0.21613(15)	0.1110(2)	0.0468(7)
H22A	−0.2922	0.2101	0.0313	0.070*
H22B	−0.3776	0.2465	0.1223	0.070*
H22C	−0.3182	0.1733	0.1450	0.070*
C23	−0.5746(3)	0.43585(13)	−0.3852(2)	0.0356(6)
H23A	−0.6631	0.4357	−0.4323	0.053*
H23B	−0.5566	0.3915	−0.3544	0.053*
H23C	−0.4978	0.4487	−0.4296	0.053*

Source of material

A mixture of $Cu(ClO_4)_2{\cdot}6H_2O$ (19.9 mg, 0.1 mmol) and 2-(2-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)phenoxy)benzoic acid (*PTPBA*) (35.8 mg, 0.1 mmol) in 15 mL methanol was stirred for 2 h to afford a clear blue solution. The mixture was filtered and kept for slow evaporation to obtained a dark blue crystal in 62.4% yields (based on Cu).

Experimental details

The structure was solved by direct methods and refined using the SHELX software [7]. All hydrogen atoms were placed in calculated positions.

Discussion

During the past decade, a large amount of effort has been focused on the purposive design and controllable synthesis of various N-heterocyclic carboxylic acid coordination polymers because of their peculiar topology structure as well as potential properties [1–3]. It is well known that N-heterocyclic carboxylic acid ligands such as pyridine-, triazole-, tetrazole-, imidazole-, pyrazole-carboxylic acids containing N and O donors have a strong metal binding capability and can provide versatile coordination modes. Utilizing *PTPBA* and copper perchlorate hydrate a dinuclear copper(II) complex was synthesized (*cf.* the figure). The asymmetric unit of the title structure contains one deprotonated *PTPBA* one Cu(II) one, coordinated methanol ligand and one additional methanol molecule. In the structure, Cu(II) adopts a slightly distorted octahedral (CuN_3O_3) coordination with three nitrogen atoms (N1,N2 and N3) and two oxygen atoms (O1 and O2) from two deprotonated *PTPBA* and one oxygen atom (O4) from methanol. The bond angles around Cu(II) atom vary from 80.46(6)° to 177.81(6)°. The Cu—N bond distances range from 1.9787(15)Å to 2.2042(6)Å and the Cu—O bond distances range from 1.9562(13)Å to 2.2748(15)Å, which are in accordance with Cu(II) complexes previously reported [4–6].

Acknowledgements: I acknowledge support for the publication fee by the Chongqing Education Commission (KJ1401114), Chongqing Education Commission (KJ1501119), Chongqing science and technology commission funded projects (cstc2015jcyjA20031), Chongqing University of Arts and Sciences (Z2015CH02), Chongqing University of Arts and Sciences(XSKY2015023) and Chongqing University of Arts and Sciences (XSKY2014031).

References

1. Chen, D. M.; Zhang, X. P.; Wei, S.; Cheng, P.: Microporous metal-organic framework based on a bifunational linker for selective sorption of CO_2 over N_2 and CH_4. Inorg. Chem. **54** (2015) 5512–5518.
2. Rabten, W.; Karkas, M. D.; Akermark, T.; Chen, H.; Liao, R. Z.; Tinnis, F.; Sun, J. L.; Siegbahn, P. E. M.; Andersson, P. G.; Akermark, B.: Catalytic water oxidation by a molecular ruthenium complex: unexpected generation of a single-site water oxidation catalyst. Inorg. Chem. **54** (2015) 4611–4620.
3. Niu, C. Y.; Zheng, X. F.; Feng, C. L.; Kou, C. H.: A 2-dimensional cadmium(II) coordination polymer with the unique 4-fold interpenetrated (4, 4) layered structure from a long bridging ligand. J. Mol. Struct. **1028** (2012) 44–48.
4. Ozarowski, A.; Calzado, C. J.; Sharma, R. P.; Kumar, S.; Jezierska, J.; Angeli, C.; Spizzo, F.; Ferretti, V.: Metal-metal interactions in trinuclear copper(II) complexes

$[Cu_3(RCOO)_4(H_2TEA)_2]$ and binuclear $[Cu_2(RCOO)_2(H_2TEA)_2]$. Syntheses and combined structural, magnetic, high-field electron paramagnetic resonance, and theoretical studies. Inorg. Chem. **54** (2015) 11916–11934.
5. Tazaki, D.; Akitsu, T.; Silva, P. S. P.; Rodrigues, V. H. N.: Crystal structures and non-linear optics of several types of bimerallic assemblies comprising chiral Cu(II) complexes. Polyhedron **102** (2015) 297–307.
6. Ashurov, J. M.; Ibragimov, A. B.; Ibragimov, B. T.: Mixed-ligand complexes of Zn(II), Cd(II) and Cu(II) with triethanolamine and p-nitrobenzoic acid: Syntheses and crystal structures. Polyhedron **102** (2015) 441–446.
7. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
8. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Cryst. **42** (2009) 339–341.
9. Bruker. APEX2, SAINT and SADABS. Brucker AXS Inc., Madison, Wisconsin, USA, 2009.