Shi-Jun Chen, Shi-Hui Li*, Yao Huang, Bin Liu, Ting-Song Xiong, Jun Cui, Xiao-Shuang Shen and Deng-Qian Chen

Crystal structure of (5*Z*,10*Z*)-3,13-dichloro-17,18-dioxo-5,11-diphenyl-8,9,17,18-tetrahydro-7*H*-dibenzo[*e,n*][1,4,8,12]tetraazacyclopentadecine-16,19-diido- $\kappa^4 N,N',N'',N'''$)copper(II), $C_{31}H_{22}N_4O_2Cl_2Cu$

Table 1: Data collection and handling.

Crystal:	Block, blue
Size:	0.22 × 0.18 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.05 mm ^{−1}
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6230, 2511, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2098
$N(\text{param})_{\text{refined}}$:	183
Programs:	Bruker programs [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.0000	0.47197(3)	0.2500	0.0335(2)
Cl1	0.8874(2)	0.43393(8)	0.06693(7)	0.0680(4)
O1	0.1536(5)	0.27186(15)	0.21312(17)	0.0557(9)
N1	0.1601(5)	0.39735(17)	0.21461(15)	0.0316(8)
N2	0.1027(5)	0.54528(17)	0.19162(16)	0.0348(8)
C1	0.0911(6)	0.3305(2)	0.22914(17)	0.0346(10)
C2	0.3307(6)	0.4070(2)	0.18016(18)	0.0316(10)
C3	0.4667(8)	0.3507(2)	0.17178(19)	0.0431(12)
H3	0.4401	0.3059	0.1896	0.052*
C4	0.6354(7)	0.3592(3)	0.1387(2)	0.0443(11)
H4	0.7224	0.3210	0.1348	0.053*
C5	0.6760(6)	0.4246(3)	0.1112(2)	0.0435(11)
C6	0.5510(6)	0.4814(2)	0.11840(19)	0.0396(11)
H6	0.5823	0.5255	0.1003	0.047*
C7	0.3767(6)	0.4748(2)	0.15235(18)	0.0325(9)
C8	0.2508(6)	0.5399(2)	0.15459(19)	0.0317(10)
C9	0.3039(6)	0.5992(2)	0.1096(2)	0.0338(10)
C10	0.2529(8)	0.5944(3)	0.0467(2)	0.0456(12)
H10	0.1776	0.5558	0.0330	0.055*
C11	0.3110(7)	0.6452(3)	0.0045(2)	0.0529(13)
H11	0.2738	0.6412	−0.0375	0.063*
C12	0.4240(7)	0.7024(3)	0.0233(2)	0.0524(14)
H12	0.4659	0.7364	−0.0060	0.063*
C13	0.4747(8)	0.7090(2)	0.0857(2)	0.0507(12)
H13	0.5497	0.7480	0.0988	0.061*
C14	0.4147(7)	0.6580(2)	0.1290(2)	0.0420(11)
H14	0.4485	0.6630	0.1713	0.050*
C15	−0.0226(8)	0.6092(2)	0.1904(2)	0.0443(11)
H15A	−0.1573	0.5943	0.1861	0.053*
H15B	0.0104	0.6386	0.1541	0.053*
C16	0.0000	0.6538(3)	0.2500	0.0474(15)
H16A ^a	−0.1131	0.6847	0.2545	0.057*
H16B ^a	0.1131	0.6847	0.2455	0.057*

^aOccupancy: 0.50.

<https://doi.org/10.1515/ncrs-2017-0182>

Received June 24, 2017; accepted January 16, 2018; available online March 16, 2018

Abstract

$C_{31}H_{22}N_4O_2Cl_2Cu$, orthorhombic, $C222_1$ (no. 20), $a = 6.869(2)$ Å, $b = 18.535(7)$ Å, $c = 21.199(7)$ Å, $V = 2698.9(16)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0385$, $wR_{\text{ref}}(F^2) = 0.1036$, $T = 293$ K.

CCDC no.: 1817199

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

A mixture of $Cu(OAc)_2 \cdot H_2O$ (0.1 mmol, 0.020 g), N^1 -(2-benzoyl-4-chlorophenyl)- N^2 -(3-benzoyl-4-chlorophenyl)oxalamide (0.1 mmol, 0.033 g), propane-1,3-diamine (0.1 mmol, 0.015 g) and distilled water (10 mL) was heated in a 25 mL

***Corresponding author: Shi-Hui Li**, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang 471022, Henan Province, P.R. China, e-mail: shihui471022@126.com

Shi-Jun Chen: College of Chemistry and Chemical Engineering, Xi'an Shiyou University, Xi'an 710065, Shaanxi Province, P.R. China

Yao Huang, Bin Liu, Ting-Song Xiong, Jun Cui, Xiao-Shuang Shen

and Deng-Qian Chen: Research institute of Exploration and Development, Qinghai Oilfield Company, Dunhuang 736202, Gansu Province, P.R. China

stainless steel reactor with a teflon liner at 413 K for 45 h, and then slowly cooled to room temperature. Colorless block crystals of the title compound were obtained.

Experimental details

C-bound hydrogen atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. The hydrogen atom of the hydroxyl group was allowed to rotate with a fixed angle around the C—O bond to best fit the experimental electron density, with $U_{\text{iso}}(\text{H})$ set to $1.5U_{\text{eq}}(\text{O})$.

Discussion

Over the past few decades, supramolecular chemistry depending upon hydrogen bonding and intermolecular weak interactions have been a field of rapid growth due to potential application such as catalysis, magnetism, semiconductor, nonlinear optical materials, biological activity [4–7]. N-Heterocycles are important building modules which provide supramolecular recognition sites for N—H···O, C—H···O, C—H··· π and π – π interactions construct supramolecular architectures.

The asymmetric unit of the title structure contains one half of a Cu(II) and one half of the organic ligand (*cf.* the figure). The Cu(II) is located on a twofold axis and is four-coordinated by four nitrogen atoms from the N-heterocyclic ligand. The Cu—N bond lengths are 1.920(3) Å and 1.969(3) Å. The bond angles of N—Cu—N are in the range of 87.8(2)°–161.48(14)°.

Acknowledgements: This work was supported by the Institutions of higher learning key scientific research project of Henan Province (18B150015) and the Natural Science Foundation of Henan Province (182102310863).

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