Qi Yang, Meng-Qi Hou, Pei-Rou Chen, Shuo Li*, Guang-Peng Zhou*, Gan Li and Dong-Dong Chen

Crystal structure of the Cu(II) complex chlorido-(6-oxo-2-phenyl-1,6-dihydropyrimidine-4-carboxylato- k^2N,O)-(phenanthroline- k^2N,N') copper(II), $C_{23}H_{15}ClCuN_4O_3$

The molecular structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Blue plate
Size:	0.20 × 0.20 × 0.05 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	3.09 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	71.7°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15,216, 3863, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3568
$N(\text{param})_{\text{refined}}$:	293
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

Source of material

The title complex was synthesized by hydrothermal synthesis. CuCl_2 (0.017 g, 0.1 mmol), 1,10-phenanthroline (0.02 g, 0.1 mmol), 6-hydroxy-2-phenylpyrimidine-4-carboxylic acid $\text{C}_{11}\text{H}_8\text{N}_2\text{O}_3$ (0.0432 g, 0.2 mmol), and H_2O (6 mL) were stirred at room temperature for 1 h and then put in a 20 mL high pressure reaction vessel. The autoclave was heated in an oven at 140° for 3 days and then cooled to room temperature to obtain blue flakes of the complex based on copper chloride. Then the complex was washed several times with deionized water and filtered to give crystals. Yields: 47.65%. 6-hydroxy-2-phenyl-pyrimidine-4-carboxylic acid was prepared by reference [5]. ^1H NMR (600 MHz, DMSO) δ : 6.87 (s, DiazineH, 1H), 7.54–7.56 (m, PhH, 2H), 7.60 (d, $J = 6$ Hz, PhH, 1H), 8.17–8.18 (d, $J = 6$ Hz, PhH, 1H) ppm.

Experimental details

The structure was determined by the intrinsic phasing routines in the SHELXT program [2] and refined in SHELXL

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Abstract

$\text{C}_{23}\text{H}_{15}\text{ClCuN}_4\text{O}_3$, monoclinic, $P2_1/c$ (no. 14), $a = 10.63346(14)$ Å, $b = 17.99387(15)$ Å, $c = 11.60320(16)$ Å, $\beta = 116.2165(17)^\circ$, $V = 1991.74(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0315$, $wR_{\text{ref}}(F^2) = 0.0898$, $T = 293(2)$ K.

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***Corresponding authors:** Shuo Li and Guang-Peng Zhou, School of Chemical Engineering, Chongqing University of Technology, Chongqing 400054, PR China, E-mail: lishuo@cqut.edu.cn (S. Li), E-mail: gpzhou@cqut.edu.cn (G.-P. Zhou). <https://orcid.org/0000-0002-1459-4132> (S. Li)

Qi Yang, Meng-Qi Hou and Pei-Rou Chen, School of Chemical Engineering, Chongqing University of Technology, Chongqing 400054, PR China

Gan Li, Shengli Oil Field Petroleum Engineering Technology, Research Institute Sino Petroleum Corp, Dongying 257000, PR China

Dong-Dong Chen, Synthesis Department, Shanghai Synthfine Chemical Co., LTD, Shanghai 200000, PR China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2768 (2)	0.59654 (10)	0.42890 (19)	0.0337 (4)
H1	0.266054	0.645590	0.448167	0.040*
C2	0.2385 (2)	0.57708 (12)	0.3004 (2)	0.0386 (4)
H2	0.204274	0.612914	0.236072	0.046*
C3	0.2520 (2)	0.50492 (12)	0.27098 (19)	0.0378 (4)
H3	0.227091	0.491410	0.186292	0.045*
C4	0.3036 (2)	0.45097 (11)	0.36891 (19)	0.0341 (4)
C5	0.3196 (2)	0.37349 (12)	0.3487 (2)	0.0449 (5)
H5	0.294596	0.356112	0.265965	0.054*
C6	0.3708 (2)	0.32540 (11)	0.4490 (2)	0.0468 (5)
H6	0.378472	0.275323	0.433295	0.056*
C7	0.4131 (2)	0.34960 (10)	0.5778 (2)	0.0377 (4)
C8	0.4685 (2)	0.30315 (12)	0.6873 (3)	0.0487 (5)
H8	0.477850	0.252354	0.678445	0.058*
C9	0.5081 (3)	0.33335 (13)	0.8056 (2)	0.0518 (6)
H9	0.546168	0.303139	0.878032	0.062*
C10	0.4920 (2)	0.40936 (13)	0.8193 (2)	0.0440 (5)
H10	0.519433	0.428858	0.901152	0.053*
C11	0.39989 (18)	0.42523 (10)	0.60003 (19)	0.0301 (4)
C12	0.34191 (18)	0.47556 (9)	0.49417 (17)	0.0285 (3)
C13	0.2592 (2)	0.68464 (11)	0.7527 (2)	0.0445 (5)
C14	0.2027 (2)	0.62356 (10)	0.8084 (2)	0.0345 (4)
C15	0.1504 (2)	0.63967 (10)	0.8922 (2)	0.0368 (4)
H15	0.155501	0.687939	0.922651	0.044*
C16	0.08718 (19)	0.58244 (11)	0.93430 (18)	0.0327 (4)
C17	0.14387 (18)	0.50025 (10)	0.79847 (18)	0.0299 (4)
C18	0.12316 (19)	0.42607 (10)	0.73815 (19)	0.0314 (4)
C19	0.1159 (2)	0.36131 (11)	0.8013 (2)	0.0398 (4)
H19	0.128952	0.363262	0.885906	0.048*
C20	0.0891 (3)	0.29421 (12)	0.7365 (3)	0.0507 (6)
H20	0.083873	0.251000	0.778185	0.061*
C21	0.0699 (3)	0.29048 (13)	0.6108 (3)	0.0530 (6)
H21	0.053205	0.244909	0.568811	0.064*
C22	0.0756 (2)	0.35452 (13)	0.5477 (2)	0.0481 (5)
H22	0.061508	0.352229	0.462767	0.058*
C23	0.1025 (2)	0.42219 (11)	0.6111 (2)	0.0378 (4)
H23	0.106679	0.465238	0.568578	0.045*
Cl1	0.57305 (6)	0.58805 (4)	0.91027 (5)	0.05194 (15)
Cu1	0.39352 (3)	0.56232 (2)	0.71499 (3)	0.03241 (10)
N1	0.43852 (17)	0.45451 (9)	0.71863 (16)	0.0329 (3)
N2	0.32794 (16)	0.54735 (8)	0.52367 (15)	0.0292 (3)
N3	0.20351 (17)	0.55322 (8)	0.76230 (16)	0.0330 (3)
N4	0.09012 (16)	0.51310 (9)	0.88311 (15)	0.0313 (3)
H4	0.058 (3)	0.4800 (15)	0.904 (2)	0.044 (7)*
O1	0.02937 (16)	0.59064 (8)	1.00528 (14)	0.0424 (3)
O2	0.2351 (3)	0.74883 (9)	0.7696 (3)	0.0861 (8)
O3	0.32527 (16)	0.66331 (7)	0.68986 (14)	0.0397 (3)

[3] using Olex 2 [4]. All of the hydrogen atoms were placed in the calculated positions.

Comment

Metal complex have the advantages of biological activity, excellent porosity, high loading capacity, etc. and play an important role in biomedical applications. It could be used to effectively load drugs and is beneficial to package various compounds and therapeutic drugs [6, 7]. The complexes formed with Cu^{2+} have thousands of applications; for example as nucleic acid cleavage enzymes because of their cleavage activity to DNA, and some complexes have certain anticancer activities [8, 9]. As a new type of ligand, 6-oxo-2-phenyl-1,6-dihydropyrimidine-4-carboxylate coordinate with Cu^{2+} to form the title complex. A TG curve was obtained. From the thermo gravimetric curve it could be found that there were two parts in weight loss, indicating that each organic ligand in the complex is removed separately respectively. When the temperature was between 286 and 463°C, there was rapid weight loss, which meant the collapse of the whole system: weight loss 61.83%. Bond lengths and angles are all in the expected ranges.

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