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NOTE

Hydrothermal Synthesis and Structure of a 3D-Supramolecular Complex: phen-Cu-C_2O_4

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The complex $Cu[(phen)(C_2O_4)(H_2O)]\cdot H_2O$, where phen = 1,10-phenanthroline, has been synthesized and its crystal structure were determined by X-ray diffraction. Crystal data: monoclinic, space group P2(1)/n, a = 8.457 Å, b = 9.70580(10) Å, c = 17.45100(10) Å, α = 90º, β = 103.8190(10)º, γ = 90º, V = 1391.016(16)Å^3, Z = 4, Mr = 367.80, Dc = 1.756 Mg/cm^3, μ = 1.605 mm^{-1}, F(000) = 748, T = 273(2) K, R = 0.0292, wR = 0.0735 for 2460 observed reflections ($I > 2\sigma(I)$). The center Cu(II) ion is coordinated by five atoms in a slightly distorted square pyramidal geometry. The compound is in 3D supramolecular state.

Key Words: Copper(II) complex, Crystal structure, Phenanthroline, Oxalate, Superamolecular.

The copper complexes with ligands containing nitrogen and oxigen as donors is currently of great interest in the field of coordination chemistry[1-8]. Herein, the preparation and crystal structure of $Cu[(phen)(C_2O_4)(H_2O)]\cdot H_2O$ is reported.

IR spectra were record on a Nexus-870 spectrophotometer. Elemental analyses were performed on a Elemental Vario EL-III elemental analyzer.

Synthesis: A mixture of $CuSO_4$ (0.4 mmol), 1,10-phenanthroline (0.6 mmol), oxalic acid (0.4 mmol) and H_2O (25 mL) were carried out in a autoclave and heated to 150 ºC for 48 h. After cooling, the mixture was filtered and the filtrate was standing at 25 ºC for three weeks. The product was blue pillar-shaped crystal. IR(KBr, ν_{max}, cm^{-1}): (O-H) 3460, (C=O) 1690, 1660, (C=C) 1520, 1400, (phen) 868, 723. Elemental Anal. Calcd. (%) for $C_{14}H_{12}CuN_2O_6$: C, 45.70; H, 3.29; N, 7.62; Found (%): C, 45.66; H, 3.42; N, 7.71.

Crystal structure determination: A single crystal of compound with dimensions of 0.56 mm × 0.30 mm × 0.28 mm was selected for crystallographic data collection at 293(2) K and structure determination on a Siemens SMART CCD area-detector diffractometer with graphite-monochromatic MoK_α radiation (λ = 0.71073A). A total of 6886 reflections were

collected in the range of $2.40^\circ \le \theta \le 25.04^\circ$, out of which 2460 reflections were unique with $R_{int} = 0.0240$ and R = 0.0292, wR = 0.0735; where w = $1/[\sigma^2(F_0^2) + (0.0315P)^2 + 1.4238P]$ and P = $(F_0^2 + 2FC^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.292 and -0.558e/Å^3 and the CCDC number is 270813, respectively.

The atomic coordinates and thermal parameters are listed in Table-1, and the selected bond lengths and bond angles in Table-2. The molecular structure of the Cu[(phen)(C_2O_4)(H_2O)] is shown in Fig. 1, in which the center copper(II) ion is five-coordinated with two N atoms of the 1,10-phenanthroline and two O atoms of a $C_2O_4^{2-}$ group and one O atom of the coordinated water in a slightly distorted square pyramidal geometry. The hydrogen-bonded is characterized in Table-3. The molecular packing arrangement in the unit cell is shown in Fig. 2. The complex molecules form a chain-link structure and many chains make up of a two-dimensional polymer layer by the hydrogen bond. Then through the π-π stacking interactions, many of these layers pack in a 3D net structure supramolecular compound.

TABLE-1
ATOMIC COORDINATES (×10^4) AND THERMAL PARAMETERS (×10^3 Å^2)

Atom	X	Y	Z	U(eq)
CU	1447(1)	196(1)	1542(1)	29(1)
N(1)	2338(2)	-1201(2)	913(1)	29(1)
N(2)	1552(2)	1417(2)	623(1)	31(1)
C(1)	-504(3)	881(3)	2504(1)	32(1)
C(2)	118(2)	1453(2)	1983(1)	35(1)

TABLE-2
SELECTED BOND DISTANCES (Å) AND ANGLES (º)

Bond	Length	Angle	(º)	Angle	(º)
CU-O(1)	1.9382(16)	O(1)-CU-O(2)	85.18(7)	C(12)-N(2)-CU	129.84(17)
CU-N(1)	2.0010(2)	O(1)-CU-N(1)	166.94(8)	O(4)-C(1)-O(1)	125.30(2)
C(1)-O(1)	1.2810(3)	N(1)-CU-N(2)	82.44(8)	O(3)-C(2)-O(2)	125.40(3)
C(2)-O(3)	1.2160(3)	N(1)-CU-O(5)	98.36(8)	C(1)-C(1)-CU	112.60(16)

TABLE-3
HYDROGEN BOND DISTANCE (Å) AND ANGLES (º)

D–H···A	D–H	H···A	D···A	∠DHA
O(5)–H(1)···O(6)	0.920(18)	1.831(19)	2.751(3)	180(4)
O(5)–H(2)···O(2)#1	0.903(19)	2.070(2)	2.952(3)	166(3)
O(6)–H(3)···O(3)#2	0.887(18)	2.190(2)	3.008(3)	153(3)
O(6)–H(4)···O(1)#3	0.904(18)	1.993(19)	2.886(3)	169(3)

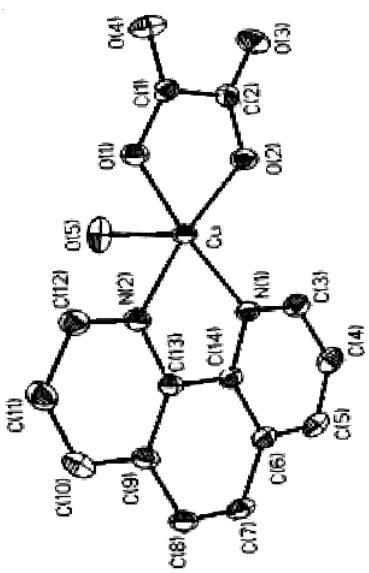

Fig. 1. Molecular structure of the $Cu[(phen)(C_2O_4)(H_2O)]$

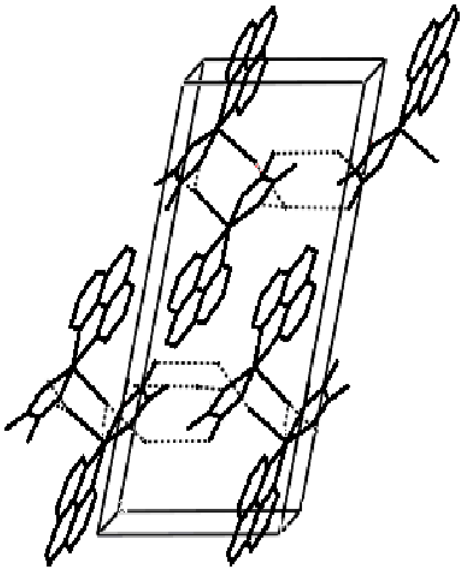

Fig. 2. Molecular packing arrangement in the unit cell

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