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NOTE

Synthesis and Structure of a 2D-Supramolecular Compound: $[Cu(Phen)_2Cl]\cdot Cl\cdot 3.5H_2O$

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A new 2D-supramolecular compound of formula $[Cu(Phen)_2Cl]\cdot Cl\cdot 3.5H_2O$ (phen = 1,10-phenanthroline) has been synthesized and determined by X-ray diffraction. The crystal is monoclinic, space group C2/c with unit cell parameters: a = 23.354(2)Å, b = 30.289(3)Å, c = 7.4973(6)Å. $\alpha = 90^o$, $\beta = 97.744(4)^o$, $\gamma = 90^o$, v = 5255.1(8)Å^3, z = 8, Dc = 1.441 Mg/m^3, Mr = 569.90, F(000) = 2336 and μ = 1.072 mm^{-1}. The final R and wR are 0.0734 and 0.2139, respectively for 5997 observed reflections with $I>2\sigma(I)$. The center copper(II) ion is five-coordinated in a square pyramidal geometry. The compound is in 2D-supramolecular state.

Key Words: Copper(II), 1,10-Phenanthroline, Supramolecular, Crystal structure.

Recently, the metal-organic framework materials (MOFs) has many advantages and a wide range of uses, the synthesis of MOFs is being more and more attention[1,2]. Many MOFs supramolecular compounds containing copper(II) have been synthesized, because Cu ion is soft acid and it favour to be coordinated by soft base such as unsaturated N-containing ligands[3-6]. In this communication, the preparation and crystal structure of a novel 2D-supramolecular compound $[Cu(Phen)_2Cl]\cdot Cl\cdot 3.5H_2O$ are reported.

All reagents were of AR grade and used without further purification. IR spectra was record on a Nexus-870 spectrophotometer. Elemental analyses for C, H and N were performed on a Elementar Vario EL-III analyzer. The crystal structure was determined by Siemens SMART CCD area-detector diffractometer.

Synthesis: A mixture of $CuSO_4$ (0.3 mmol), 1,10-phenanthrolline (0.6 mmol), potassium hydrogenphthalate (0.3 mmol) and H_2O (25 mL) was carried out in a lautoclave, then heated to 150 ºC for 48 h. The product was

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bright green slender four-prism crystal. IR (KBr, ν_{max}, cm^{-1}): 3500-3200 [s, ν(O-H)], 1560, 1400 [s, ν(C=C, C=N)], 849, 725 [m, ν(phen)]. Anal. calcd. (%) for $C_{24}H_{23}N_4O_{4.25}Cl_2Cu$: C, 50.57; H, 4.07; N, 9.83. Found: C, 50.45; H, 4.31; N, 9.96.

Structure determination: A single crystal (0.45 mm × 0.07 mm × 0.05 mm) was selected for crystallographic data collection at 293(2)K and structure determinated with graphite-monochromatic MoKα radiation (λ = 0.71073Å). A total of 20453 reflections were collected in the range of $2.20^o \leq \theta \leq 27.48^o$, of which 5997 reflections were unique with $R_{int} = 0.0337$. The final full-matrix least-squares refinement including 336 variable parameters for 5997 reflections with $I>2\sigma(I)$ and converged with unweighted and weighted agreement factors of R = 0.0734 and wR = 0.2139, where $w = 1/[\sigma^2(F_0^2) + (0.1332P)^2 + 4.3736P]$, and $P = (F_0^2 + 2FC^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.985 and -0.395e/$Å^3$ (CCDC No. 646142), respectively.

The atomic coordinates and thermal parameters are given in Table-1 and the selected bond lengths and bond angels are listed in Table-2. The molecular structure of $[Cu(Phen)_2Cl]\cdot Cl\cdot 3.5H_2O$ is shown in Fig. 1, in which the center copper(II) ion is five-coordinated in a distorted square pyramidal geometry and the axial position is occupied by one coordinated Cl^-. Fig. 2 depicts the packing diagram in the unit cell and shows that the moleculars are linked to the neighbours by π-π stacking interactions and form 1D chain-like structure. Many of these chains packed in 2D-supramolecular state by H-bonds.

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

Atom	X	Y	Z	U(eq)
Cu	2751(1)	1198(1)	3392(1)	51(1)
N(1)	2092(1)	841(1)	2229(5)	54(1)
N(2)	3093(1)	565(1)	4027(4)	49(1)
N(3)	2263(1)	1548(1)	5074(4)	48(1)
N(4)	3379(1)	1561(1)	4697(5)	53(1)
C(1)	1606(2)	983(2)	1301(7)	68(1)
C(3)	1241(2)	263(2)	724(7)	70(1)

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

Bond	Dist.	Bond	Dist.	Angles	(º)	Angles	(º)
Cu-N(1)	1.984(3)	Cu-Cl(1)	2.3375(12)	N(1)-Cu-N(2)	81.42(13)	N(3)-Cu-Cl(1)	121.64(9)
Cu-N(2)	2.108(3)	N(1)-C(1)	1.319(5)	N(1)-Cu-N(3)	95.01(13)	N(3)-Cu-N(2)	122.96(12)
Cu-N(3)	2.097(3)	N(1)-C(12)	1.371(5)	N(1)-Cu-Cl(1)	91.04(11)	N(4)-Cu-N(1)	176.06(14)
Cu-N(4)	1.983(3)	N(2)-C(10)	1.328(5)	N(2)-Cu-Cl(1)	115.35(9)	N(4)-Cu-Cl(1)	92.19(11)

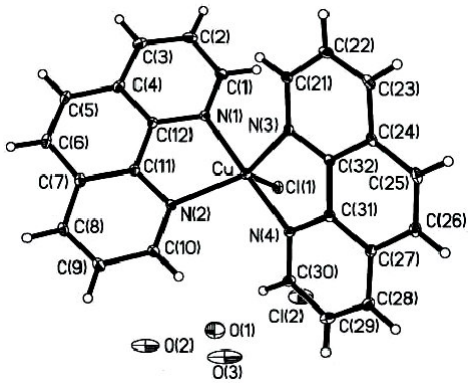

Fig. 1. Structure of the title compound

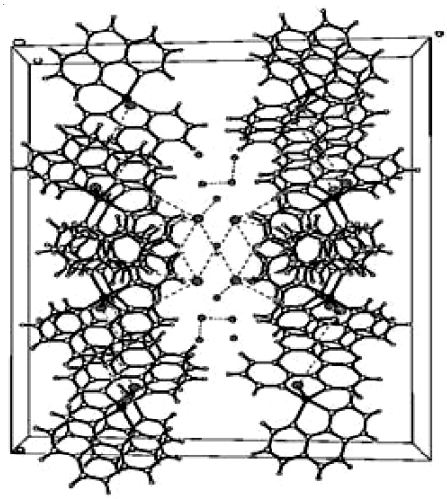

Fig. 2. Molecular packing arrangement

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