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NOTE

Supramolecular Structure of *Bis*(4-benzoyl-3-methyl-1-phenyl-5-(2*H*)-2-pyrazolonato)copper(II)

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The molecule of the Cu(II) complex with the empirical formula $C_{34}H_{26}N_4O_4Cu$, *bis*(4-benzoyl-3-methyl-1-phenyl-5(2*H*)-2-pyrazolonato)copper(II), is rigorously centrosymmetric [symmetry codes: -x + 1, -y + 1, -z + 1]. Each Cu(II) atom is four-coordinated by two oxygen atoms (O1 and O1#) from pyrazolone ring in deproton enol-form and other two oxygen atoms (O2 and O2#) in carbonyl-form forming a rigorously square planar geometry. In the crystal structure, weak intermolecular C-H···O hydrogen bonds link the title molecules into an infinite 1D supramolecular chains structure.

Key Words: β-Diketonate, Copper(II) complex, Supramolecular structure.

β-Diketonate compound was known to possess a high chelating ability with the metal ions and high pharmaceutical, biological, clinical and analytical activities[1-3]. It existed in two tautomeric forms *i.e.*, the enol form and the keto form[4]. When it coordinated with metal ions, the complexes showed various structures such as facial (*fac*-) and meridional (*mer*-) forms[5,6]. As an extension of our research work[7,8] on the structural characterization of transition metal complexes, a single crystal of unexpected complex, *bis*(4-benzoyl-3-methyl-1-phenyl-5(2*H*)-2-pyrazolonato)copper(II) was obtained and structurally characterized by X-ray crystallography.

l-Phenyl-3-methyl-4-benzoyl-5-pyrazolone was purchased from Alfa Aesar and used without further purification. The reagents and solvents were analytical grade reagents from Tianjin Chemical Reagent Factory. C, H and N analyses were carried out with a GmbH VariuoEL V3.00 automatic elemental analyzer. X-Ray single crystal structure was determined on a Bruker Smart 1000 CCD area detector.

General procedure: The ligand l-phenyl-3-methyl-4-benzoyl-5-pyrazolone was dissloved in DMF (0.01 mmol) then added dropwise to an anhydrous EtOH solution (2 mL) of copper(II) acetate hydrate (0.01 mmol) at room temperature. The brilliant yellow solution obtained was placed at room temperature for about seven days. Black block-like single crystals of *bis*(4-benzoyl-3-methyl-1-phenyl-5(2*H*)-2-pyrazolonato)copper(II) complex suitable for X-ray crystallographic analysis was obtained. Anal. calcd. (%) for $C_{34}H_{26}N_4O_4Cu$: C, 66.09; H, 5.85; N, 8.56; Cu, 9.71. Found (%): C, 66.81; H, 5.94; N, 8.4; Cu, 9.63.

TABLE-1
CRYSTAL DATA AND REFINEMENT PARAMETERS FOR THE Cu(II) COMPLEX

Empirical formula	$C_{34}H_{26}N_4O_4Cu$
Formula weight	618.13
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/c
Cell dimensions	a = 6.8384(8) Å, b = 23.855(2) Å, c = 9.1591(12) Å, β = 108.948(2)
Volume	1413.1(3) Å^3
Z	2
Density (calculated)	1.453 mg/m^3
Absorption coefficient	0.820 mm^{-1}
$F_{(000)}$	638
Index ranges	$-8 \le h \le 8$, $-28 \le k \le 23$, $-10 \le l \le 10$
Reflections collected	7365 [$R_{(int)}$ = 0.0366]
Independent reflections	2494
Data/restraints/parameters	2494/0/197
Goodness of fit indicator	0.981
R [I > 2σ (I)]	R_1 = 0.0335, wR_2 = 0.0777
Largest diff. peak and hole	0.223 and -0.266 e Å

X-Ray structure determination: The single crystal of the Cu(II) complex, with approximate dimensions of 0.24 mm × 0.15 mm × 0.07 mm was placed on a Bruker Smart 1000 diffractmeter equipped with Apex CCD area detector. The diffraction data were collected using a graphite monochromated MoK$_\alpha$ radition (λ = 0.71073 Å) at 298(2) K. The structure was solved by using the program SHELXS-97 and Fourier

difference techniques and refined by full-matrix least-squares method on F^2 using SHELXL-97. Details of the data collection and refinements of Cu(II) complex are given in Table-1. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added theoretically.

The crystal structure of the Cu(II) complex is built up only by $C_{34}H_{26}N_4O_4Cu$ molecules (Fig. 1), in which all bond lengths are in normal ranges. The dihedral angle of the two coordination molecules O1-Cu1-O2 and O1#-Cu1-O2# is 0º, indicating the two oxygen atoms (O1 and O1#) from pyrazolone ring in deproton enol-form and other two oxygen atoms (O2 and O2#) in carbonyl-form are rigidly coplanar. Cu(II) atom is located in the centre of the planar quadrilateral defined by the two pyrazolone ring oxygen atoms (O1 and O1#) and the two carbonyl oxygen atoms (O2 and O2#). The distance of Cu1 and O1 (Cu1 and O1#), Cu1 and O2 (Cu1 and O2#) is 1.909(3) Å and 1.920(3) Å, respectively. The central Cu(II) atom in the title complex sits on a crystallographic inversion center, therefore, the whole Cu(II) complex is rigorously centrosymmetric [symmetry codes: -x + 1, -y + 1, -z + 1]. In the crystal structure, the Cu(II) complex are linked by two pairs of intermolecular C4-H4C···O2 hydrogen bond into an infinite 1D supramolecular chain along the a-axis (Fig. 2, Tables 2 and 3) [9-11].

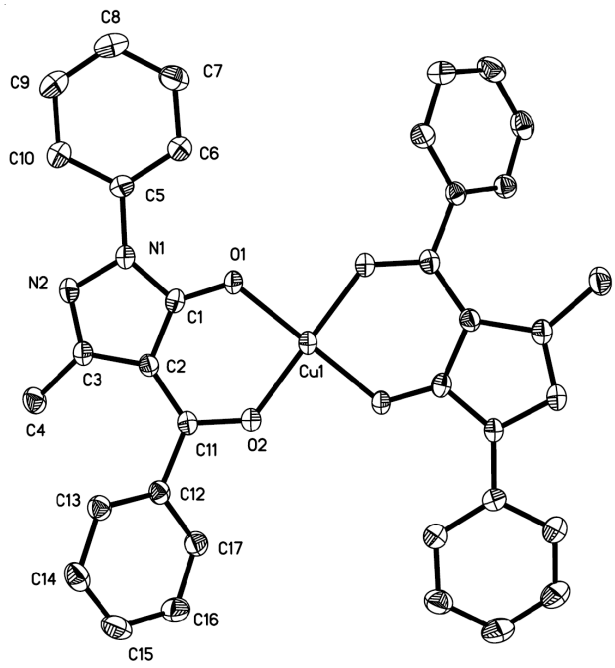

Fig. 1. Molecule structure of the Cu(II) complex

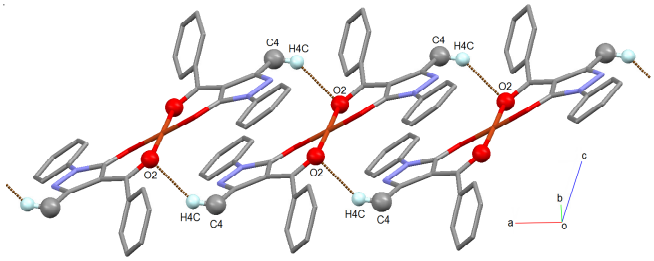

Fig. 2. View of the 1D supramolecular chain within the Cu(II) complex (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity)

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (º) FOR THE Cu(II) COMPLEX

Bond	Lengths	Bond	Lengths
Cu(1)-O(1)	1.9092(15)	Cu(1)-O(1)$^{\#}$	1.9092(15)
Cu(1)-O(2)	1.9203(15)	Cu(1)-O(2)$^{\#}$	1.9203(15)
Bond	**Angles**	**Bond**	**Angles**
O(1)$^{\#}$-Cu(1)-O(1)	180.00(8)	O(1)$^{\#}$-Cu(1)-O(2)	86.69(6)
O(1)-Cu(1)-O(2)	93.31(7)	O(1)$^{\#}$-Cu(1)-O(2)$^{\#}$	93.31(7)
O(1)-Cu(1)-O(2)$^{\#}$	86.69(6)	O(2)-Cu(1)-O(2)$^{\#}$	180.00(7)
C(1)-O(1)-Cu(1)	120.24(15)	C(11)-O(2)-Cu(1)	129.15(16)

Symmetry transformations used to generate equivalent atoms: $^{\#}$-x + 1, -y + 1, -z + 1.

TABLE-3
HYDROGEN-BONDING DATA [Å, º]

D-H···A	d(D-H)	d(H···A)	d(D···A)	∠D-H···A
C4-H4C···O2	0.96	2.56	3.339(3)	143

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