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Synthesis and Crystal Structure of a New 2D-Supramolecular Complex: $[Cd(C_{17}H_{13}N_2O_2)_2(C_2H_6O)_2]$

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The molecule of the cadmium(II) complex *i.e.*, *bis*(l-phenyl-3-methyl-4-benzoyl-5-pyrazolone)-*bis*(ethanol)cadmium(II), $[Cd(C_{17}H_{13}N_2O_2)_2(C_2H_6O)_2]$, is rigorously centrosymmetric [symmetry codes: -x, -y, -z]. The Cd(II) center exhibits a slightly distorted octahedral geometry with two ligand units forming the basal O_2O_2 coordination plane and two other oxygen atoms from two coordinated ethanol molecules in the axial position. In the crystal structure, intermolecular O-H···N hydrogen bonds and C-H···π stacking interactions link the complex molecules into infinite 2D supramolecular structure.

Key Words: Cadmium(II) complex, l-Phenyl-3-methyl-4-benzoyl-5-pyrazolone, Supramolecular structure.

INTRODUCTION

1-Phenyl-3-methyl-4-benzoyl-5-pyrazolone was known to possess a high chelating ability with the metal ions and high pharmaceutical, biological, clinical and analytical activities[1-3]. It existed as two forms: 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 Cd(II) complex, *bis*(l-phenyl-3-methyl-4-benzoyl-5-pyrazolone)*bis*(ethanol)cadmium(II) was obtained and structurally characterized by X-ray crystallography.

EXPERIMENTAL

l-Phenyl-3-methyl-4-benzoyl-5-pyrazolone was purchased from Alfa Aesar was used without further purification. The other 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 (0.01 mmol) was dissloved in an anhydrous EtOH solution (5 mL) then added dropwise to the same solvent (2 mL) of cadmium(II) acetate hydrate (0.01 mmol). Then the mixture was stirred and refluxed at 428 K for 6 h. The resulting solution was then filtered and the filtrate was kept in air for 2 weeks at room temperature. Colourless block-like single crystals of Cd(II) complex suitable for X-ray crystallographic analysis was obtained. Anal. calcd. (%) for $C_{38}H_{38}N_4O_6Cd$: C, 60.12; H, 5.05; N, 7.38; Cd, 14.81. Found (%): C, 60.23; H, 5.01; N, 7.16; Cd, 14.95.

X-Ray structure determination: The single crystal of the title complex, with approximate dimensions of 0.47 mm × 0.35 mm × 0.23 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_α radition (λ = 0.71073 Å) at 298(2) K. The structure was solved by using the program SHELXS-97[9] and Fourier difference techniques and refined by full-matrix least-squares method on F^2 using SHELXL-97[10]. Details of the data collection and refinements of Cd(II) complex are given in Table-1. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added theoretically.

RESULTS AND DISCUSSION

The X-ray crystallography indicates that the crystal structure of Cd(II) complex is built up only by $C_{38}H_{38}N_4O_6Cd$ molecules (Fig. 1), in which all bond lengths are in normal ranges. The Cd(II) center is located in the centre of the equatorial plane defined by the two oxygen atoms (O1 and $O1^{\#1}$) in enol-form from pyrazolone ring and the two carbonyl oxygen atoms (O2 and $O2^{\#1}$) and the other two oxygen atoms (O3 and $O3^{\#1}$) from two coordinated ethanol molecules lie in the axial position of the coordination plane. Thus, the six oxygen donors construct an octahedral geometry around Cd(II) atom. The

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

Empirical formula	$C_{38}H_{38}N_4O_6Cd$
Formula weight	759.12
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Cell dimensions	a = 9.1410(10) Å, b = 10.3201(13) Å, c = 10.9079(14) Å, α = 106.7810°, β = 107.613(2)°, γ = 104.6090°
Volume	871.56(18) $Å^3$
Z	1
Density (calculated)	1.446 mg/m^3
Absorption coefficient	0.679 mm^{-1}
$F_{(000)}$	390
Index ranges	$-10 \le h \le 10, -10 \le k \le 12, -12 \le l \le 12$
Reflections collected/unique	4530/3015 [R_{int} = 0.0203]
Data/restraints/parameters	3015/0/245
Goodness-of-fit on F^2	1.075
Final R indices [I > 2σ(I)]	R_1 = 0.0345, wR_2 = 0.0780
R indices (all data)	R_1 = 0.0399, wR_2 = 0.0827
Largest diff. peak and hole	0.447 and -0.362 e. Å

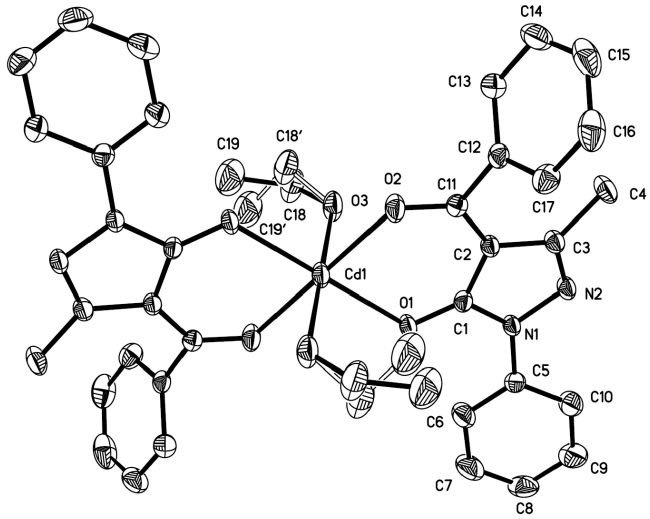

Fig. 1. Molecule structure of the Cd(II) complex

distance of O1-Cd1 ($O1^{\#1}$-Cd1), O2-Cd1 ($O2^{\#1}$-Cd1) and O3-Cd1 ($O3^{\#1}$-Cd1) is 2.226, 2.280 and 2.345 Å, respectively (Table-2), so the coordination sphere of Cd(II) complex can be described as an elongate octahedron geometry. Moreover, the whole Cd(II) complex is rigorously centrosymmetric [symmetry codes: -x, -y, -z] and Cd(II) atom lies in the crystallographic inversion center. In the crystal, the complex molecules is linked by a pair of intermolecular O3-H3···N2 hydrogen bonds into a 1D infinite chain parallel to the a-axis (Fig. 2). This linkage is further stabilized by a pair of intermolecular C9-H9···π (C12-C17) hydrogen bonds interactions to form the other 1D infinite chain (Fig. 3). Thus, every complex molecule links four other molecules into an infinite 2D-layer supramolecular structure *via* intermolecular O-H···N and C-H···π hydrogen-bonding interactions.

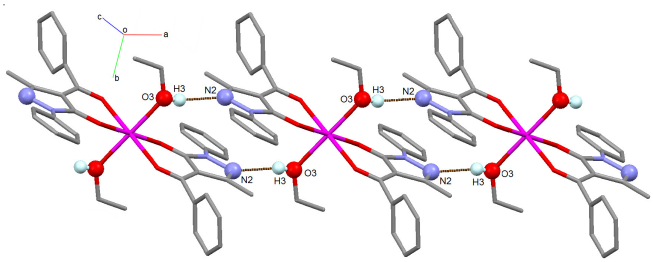

Fig. 2. View of the 1D chain along a-axis linked by O3-H3···N2 hydrogen bonds

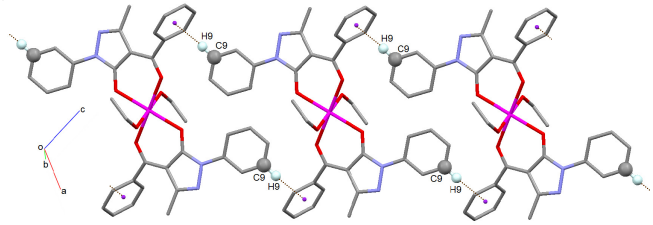

Fig. 3. View of the 1D chain linked by C9-H9···p(C12-C17) hydrogen bonds

TABLE-3
HYDROGEN-BONDING DATA [Å, °]

D-H···A	d(D-H)	d(H···A)	d(D···A)	∠D-H···A
O3-H3···N2	0.82	2.08	2.835(4)	153
C9-H9···Cg[a]	0.93	2.78	3.549(5)	141

[a]Cg is the C12–C17 ring centroid.

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TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (°) FOR THE Cd(II) COMPLEX

Bond	Lengths	Bond	Lengths	Bond	Lengths
Cd1-$O1^{\#1}$	2.2264(2)	Cd1-O1	2.2264(2)	Cd1-O2	2.280(2)
Cd1-$O2^{\#1}$	2.280(2)	Cd1-O3	2.345(2)	Cd1-$O3^{\#1}$	2.345(2)
Bond	Angles	Bond	Angles	Bond	Angles
$O1^{\#1}$-Cd1-O1	180.00(0)	$O1^{\#1}$-Cd1-O3	91.08(8)	O2-Cd1-$O3^{\#1}$	93.65(8)
$O1^{\#1}$-Cd1-O2	95.95(7)	O1-Cd1-O3	88.92(8)	$O2^{\#1}$-Cd1-$O3^{\#1}$	86.35(8)
O1-Cd1-O2	84.05(7)	O2-Cd1-O3	86.35(8)	O3-Cd1-$O3^{\#1}$	180.00(0)
$O1^{\#1}$-Cd1-$O2^{\#1}$	84.05(7)	$O2^{\#1}$-Cd1-O3	93.65(8)	C1-O1-Cd1	122.41(2)
O1-Cd1-$O2^{\#1}$	95.95(7)	$O1^{\#1}$-Cd1-$O3^{\#1}$	88.92(8)	C11-O2-Cd1	128.31(2)
O2-Cd1-$O2^{\#1}$	180.00(0)	O1-Cd1-$O3^{\#1}$	91.08(8)	C18-O3-Cd1	133.1(1)
C18'-O3-Cd1	125.0(1)				

Symmetry transformations used to generate equivalent atoms: $^{\#1}$ –x + 1, -y + 1, -z + 3.

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