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Synthesis and Crystal Structure of a New 3D-Supramolecular Complex: $[Cd(C_{10}H_{12}N_2O)_2I_2(H_2O)_2]$

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A novel complex $[Cd(C_{10}H_{12}N_2O)_2I_2(H_2O)_2]$, was synthesized and characterized by IR spectra, elemental analysis and single-crystal X-ray. The crystal structure analysis shows that the Cd(II) is a six-coordinated in a slightly distorted octahedral geometry environment, then complex packs in 3D-superamolecular network through intermolecular hydrogen bonds. The crystal is monoclinic, space group C2/c with unit cell parameters: a = 21.861(2)Å, b = 8.642(8)Å, c = 14.732(2)Å, α = 90º, β =114.4(1)º, γ = 90º, V = 2534.1(4)Å^3, Z = 4, Mr = 754.7, Dc = 1.978 Mg/cm^3, μ = 3.330 mm^{-1}, F(000) = 1448, T = 291(2) K, R = 0.0472, wR = 0.1235 for 2484 reflections with I > 2σ(I).

Key Words: Cadmium(II) complex, Crystal structure, Hydrogen bonds, Superamolecular.

INTRODUCTION

In recent years, researchers showed considerable interest in the photophysical properties of mono- and polynuclear complexes of transition metals having the d^{10} electronic configuration[1-8]. These are the derivatives of Cu(I) which show brightly luminescent with an emissive behaviour varying markedly with structure and environment[1-4]. However little attention has been paid to complexes derived from organic ligands with CdI_2[5-8]. Herein reports a mononuclear complex having formula as $[Cd(C_{10}H_{12}N_2O)_2I_2(H_2O)_2]$.

EXPERIMENTAL

All reagents were of AR grade and used without further purification. 3-(Dimethylamino)-1-(pyridin-4-yl)prop-2-en-1-one was prepared by similar procedure reported in the literature[9]. Analyses for carbon, hydrogen and nitrogen were performed on a Perkin-Elmer 1400C analyzer. Infrared spectra (4000-400 cm^{-1}) were recorded with a Bruker Vector 22 FT-IR spectrophotometer on KBr disks.

Synthesis: To a stirred solution of ligand, 3-(dimethylamino)-1-(pyridin-4-yl)-prop-2-en-1-one (0.50 mmol) in 40 mL of methanol, the solution of CdI_2 (0.25 mmol) in methanol (25 mL) was added. A white precipitate formed immediately which was filtered under vacuum and dried in air (yield: 70 %). Anal. Calcd for $C_{20}H_{28}N_4O_4I_2Cd$: C, 31.83; H, 3.74; N, 7.42 %. Found: C, 31.94; H, 3.63; N, 7.35

%. IR (KBr, cm^{-1}): 3370 (w), 1627 (s), 1604 (m), 1557 (w), 1500 (s), 1444 (m), 1419 (m), 1375 (w), 1324 (w), 1263 (s), 1138 (w), 785 (w). The filtrated solution evaporated slowly to give the pale yellow single crystals at room temperature.

Crystal structure determination: A single crystal of compound with dimensions of 0.31 mm × 0.26 mm × 0.18 mm was selected for crystallographic data collection at 291(2) K and structure determination on a Siemens SMART CCD area-detector diffractometer with graphite-monochromatic MoKα radiation (λ = 0.71073 Å). A total of 6584 reflections were collected in the range of $2.0^o \leq \theta \leq 26^o$, of which 2484 reflections were unique with $R_{int} = 0.056$. Lp effects and empirical absorption were applied in data corrections. The structure was solved by direct methods and expanded using Fourier techniques and SHELXS-97 program system was used in the solution and refinement of the structure. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added according to theoretical model. The final full-matrix least-squares refinement including 152 variable parameters for 2484 reflections with $I > 2\sigma(I)$ and converged with unweighted and weighted agreement factors of

$$R_1 = \Sigma(||F_0|-|F_c||)/\Sigma|F_0| = 0.0472 \quad (1)$$

and

$$wR_2 = \{\Sigma[w(F_0^2-F_C^2)^2]/\Sigma w(F_0^2)^2\}^{1/2} = 0.1235 \quad (2)$$

where $w = 1/[\sigma^2(F_0^2) + (0.0734P)^2 + 4.665P]$ and $P = (F_0^2 + 2F_C^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.92 and -2.03, e/Å^3, respectively.

RESULTS AND DISCUSSION

The selected bond lengths and bond angles are presented in Table-1. Fig. 1 shows the molecular structure of the title compound. Fig. 2 shows the packing diagram of $[Cd(C_{10}H_{12}N_2O)_2I_2(H_2O)_2]$. In present complex, each Cd(II) ion is coordinated by two N donors from different ligands and two coordinated iodide anions as well as two coordinated water molecules, yielding a slightly distorted octahedron environment with Cd-N and Cd-I distance in the range of those found in the other complexes[10,11]. Additionally, two pyridyl units are obviously coplanar.

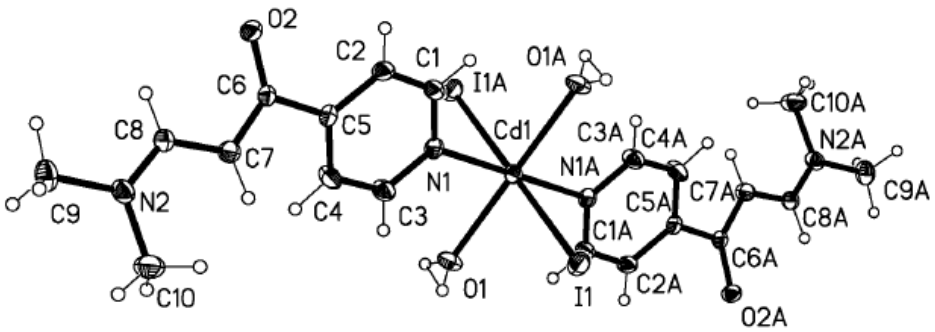

Fig. 1. Molecular structure of the $[Cd(C_{10}H_{12}N_2O)_2I_2(H_2O)_2]$

TABLE-1
SELECTED BOND DISTANCES (Å) AND ANGLES (°) OF [Cd($C_{10}H_{12}N_2O$)$_2$$I_2$($H_2O$)$_2$]

Cd1-O1	2.384(4)	O1-Cd1-O1#A	180.000(1)
Cd1-O1#A	2.384(4)	O1-Cd1-N1	90.64(14)
Cd1-N1	2.400(4)	O1#A-Cd1-N1	89.36(14)
Cd1-N1#A	2.400(4)	O1-Cd1-N1#A	89.36(14)
Cd1-I1#A	2.8514(4)	O1#A-Cd1-	90.64(14)
Cd1-I1	2.8514(4)	N1#A	180.000(1)
O1-Cd1-I1#A	91.91(11)	N1-Cd1-N1#A	89.76(9)
O1#A-Cd1-I1#A	88.09(11)	N1#A-Cd1-I1#A	88.09(11)
N1-Cd1-I11	90.24(9)	O1-Cd1-I1	91.91(11)
N1-Cd1-I1	89.76(9)	O1#A-Cd1-I1	180.0
N1#A-Cd1-I1	90.24(9)	I1#A-Cd1-I1	

#A: -X+1,-Y+1,-Z+1.

TABLE-2
HYDROGEN BOND DISTANCES (Å) AND ANGLES (°) OF [Cd($C_{10}H_{12}N_2O$)$_2$$I_2$($H_2O$)$_2$]

D–H···A	D–H	H···A	D···A	∠DHA
O1–H1A···O2	0.69(6)	2.11(7)	2.796(5)	177(10)
O1–H1B···O2	0.79(9)	2.05(9)	2.809(6)	162(10)
C4–H4···I1	0.930	3.050	3.953(5)	165.00
C9–H9···I1	0.960	3.00	3.957(6)	177.00

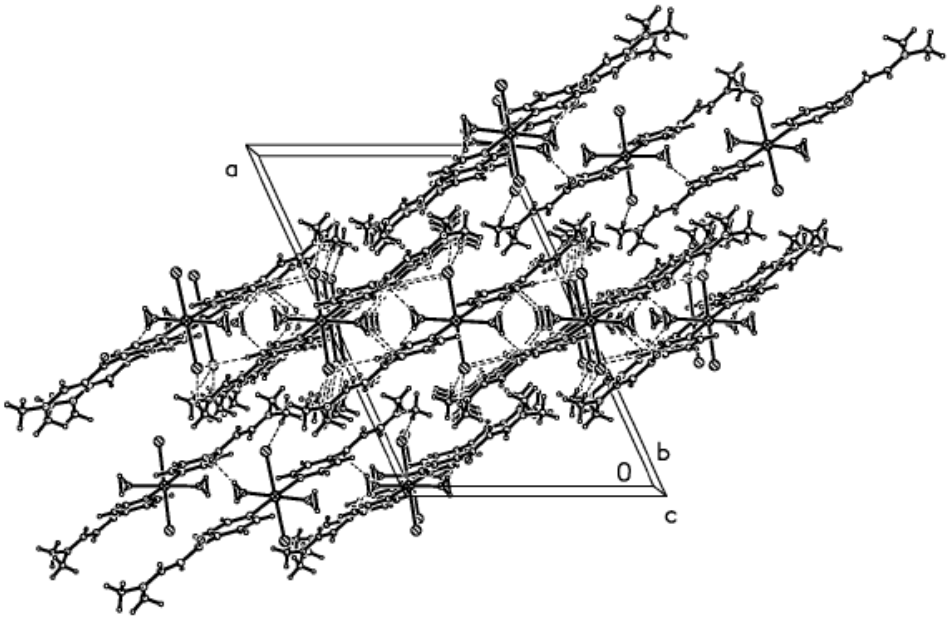

Fig. 2. View of a 3D supramolecular framework of [Cd($C_{10}H_{12}N_2O$)$_2$$I_2$($H_2O$)$_2$] showing the intermolecular hydrogen bonding

In crystal packing, it is interesting to observe that the O–H···O and C–H···I intermolecular hydrogen bonds are formed between adjacent molecules resulting in a 3D supramolecular framework.

Conclusion

Crystal structure of a novel 3D-superamolecular cadmium(II) complex has been synthesized and characterized by IR, elemental analysis and X-ray diffraction analysis.

Supplementary material: Crystallographic data for the structure reported in this communication have been deposited with the Cambridge Crystallographic Data Center as supplementary publication No. CCDC 706836.

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