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Synthesis and Crystal Structure of a New Mononuclear Complex: $[Cd(C_{10}H_{12}N_2O)_2(NO_3)_2(H_2O)_2]$

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A novel three-dimensional hydrogen-bonding mononuclear complex $[Cd(C_{10}H_{12}N_2O)_2(NO_3)_2(H_2O)_2]$, where $C_{10}H_{12}N_2O$ is 3-(dimethylamino)-1-(pyridin-4-yl)prop-2-en-1-one, was synthesized and characterized by IR spectra, elemental analysis and single-crystal X-ray. The crystal structure analysis shows that the cadmium(II) is a six-coordinated in a slightly distorted octahedral geometry environment. The crystal is monoclinic, space group C2/c with unit cell parameters: a = 22.000(3)Å, b = 8.574(12)Å, c = 14.532(2)Å, α = 90º, β = 114.643(2)º, γ = 90º, V = 2491.6(5)Å^3, Z = 4, Mr = 620, Dc = 1.666 Mg/cm^3, μ = 0.942 mm^{-1}, F(000) = 1272, T = 273(2) K, R = 0.0417, wR = 0.0954 for 2184 reflections with $I > 2\sigma(I)$.

Key Words: Crystal structure, Hydrogen bonds, Superamolecular.

INTRODUCTION

In recent several decades researchers have shown remarkable attention in the rational design and assembly of supramolecules *via* hydrogen bonds in the field of coordination chemistry[1-5]. In our previous paper, we reported some 3D supramolecular complexes derived from a multidentate ligand[6-8]. Herein reported is a novel chiral cadmium complex produced by cadmium salt and 3-(dimethylamino)-1-(pyridin-4-yl)prop-2-en-1-one.

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[8]. 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 20 mL methanolic solution of $Cd(NO_3)_2 \cdot H_2O$ (1mmol) were successively added a 10 mL methanol solution of 3-(dimethylamino)-1-(pyridin-4-yl)prop-2-en-1-one (2 mmol) with stirring. The mixture was refluxed for 2 h to obtain a

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clear solution and after standing at room temperature for 1 week, well-shaped single crystals were obtained by slow evaporation. Anal. calcd for $C_{20}H_{28}N_6O_{10}Cd$: C, 38.44; H, 4.52; N, 13.45 %. Found: C, 38.48; H, 4.54; N, 13.41 %. IR (KBr, cm^{-1}): 3083 (m), 1611 (m), 1528 (m), 1467 (m), 1389 (s), 1230 (w), 1027 (w), 836(w).

Crystal structure determination: A single crystal of compound with dimensions of 0.30 mm × 0.24 mm × 0.21 mm was selected for crystallographic data collection at 273(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 5989 reflections were collected in the range of $2.0° \leq \theta \leq 25.0°$, of which 2184 reflections were unique with $R_{int} = 0.039$. The data were collected using SMART and reduced by the program SAINT. All the structures were solved by direct methods and refined by full-matrix least squares method on F^2_{obs} by using SHELXTL-PC software package. Non-hydrogen atoms were placed in geometrically calculated positions. Hydrogen atoms were added according to theoretical model. The final full-matrix least-squares refinement including 171 variable parameters for 2184 reflections with $I > 2\sigma(I)$ and converged with unweighted and weighted agreement factors of

$$R_1 = \Sigma(||F_0|-|F_c||)/\Sigma|F_0| = 0.0417 \quad (1)$$

and

$$wR_2 = \{\Sigma[w(F_0^2-F_C^2)^2]/\Sigma w(F_0^2)^2\}^{1/2} = 0.0954 \quad (2)$$

where $w = 1/[\sigma^2(F_0^2) + (0.0254P)^2]$ and $P = (F_0^2 + 2F_C^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.53 and - 0.35 e/Å^3, respectively.

RESULTS AND DISCUSSION

A molecular structure with atom-numbering scheme and the packing diagram of $[Cd(C_{10}H_{12}N_2O)_2(NO_3)_2(H_2O)_2]$ are shown in Figs. 1 and 2, respectively. Selected bond lengths and bond angles are listed in Table-1.

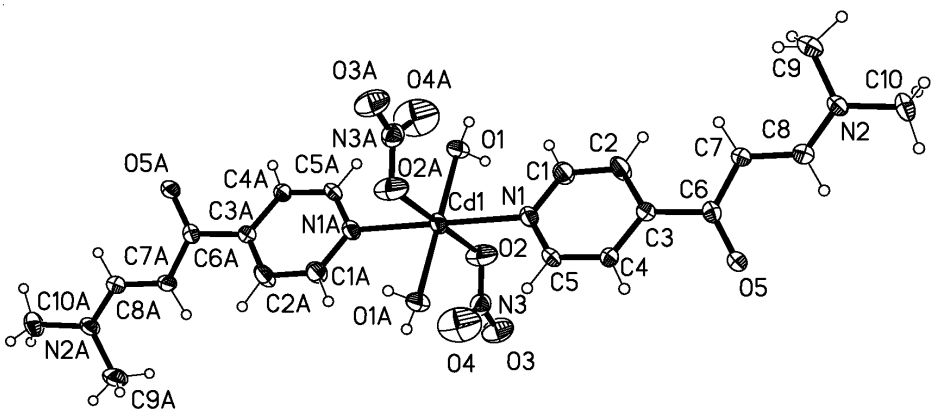

Fig. 1. Molecular structure of $[Cd(C_{10}H_{12}N_2O)_2(NO_3)_2(H_2O)_2]$

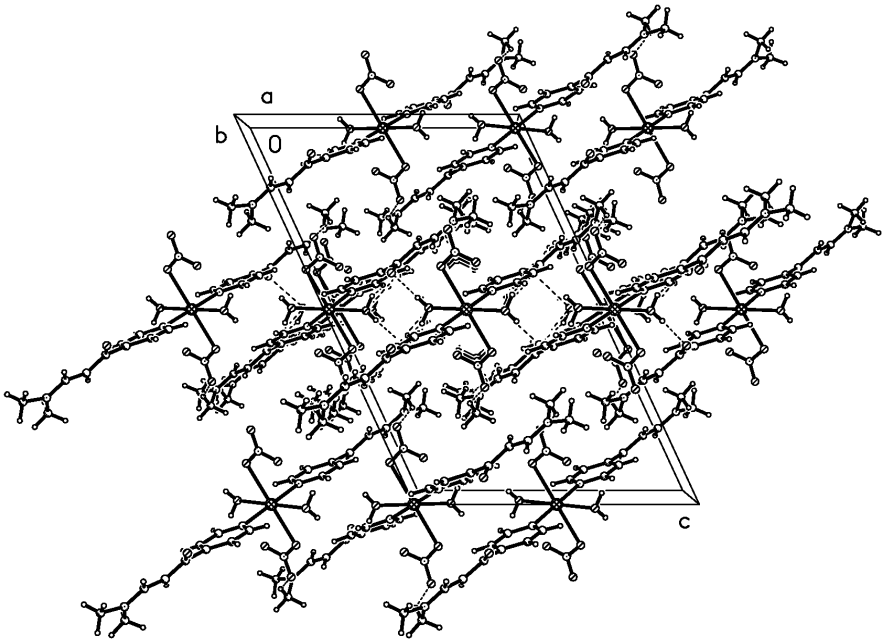

Fig. 2. Molecular packing arrangement in the unit cell

TABLE-1
SELECTED BOND DISTANCES (Å) AND ANGLES (º)

Cd(1)-N(1)	2.307(3)	O(1)-Cd(1)-O(4)	114.80(15)
Cd(1)-O(1)	2.369(3)	O(1)-Cd(1)-O(2a)	110.86(13)
Cd(1)-O(2)	2.473(5)	O(1)-Cd(1)-N(1a)	88.80(10)
N(1)-Cd(1)-O(1)	91.20(10)	N(1)-Cd(1)-O(2)	89.30(14)
O(1)-Cd(1)-O(1a)	180.00	O(2)-Cd(1)-O(2a)	180.00

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

D–H···A	D–H	H···A	D···A	∠DHA
O1-H1A···O5#1	0.850	2. 020	2.736(4)	141.0
O1-H1B···O5 #2	0.850	2.380	2.805(5)	112.0
C4-H4···O4 #3	0.930	2.590	3.490(7)	164.0
C5-H5···O1 #4	0.930	2.560	3.226(5)	129.0
C9-H9A···O3 #5	0.960	2.550	3.355(6)	141.0

Symmetry transformations used to generate equivalent atoms:
#1: x,-1+y,z; #2: -x,-1+y,1/2-z; #3: -x,1-y,-z; #4:-x,-y,-z; #5: -x,y,1/2-z.

The molecular structure of title compound owns a mononuclear motif (depicted in Fig. 1), where each Cd(II) ion is bound by two oxygen atoms from two nitrate, two water molecules and two nitrogen atoms from two organic ligands, in a octahedral fashion. Each ligand adopts a monodentate coordination pattern with a Z conformation.

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

Conclusion

Crystal structure of a new cadmium(II) complex with space group C2/c 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 723235.

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