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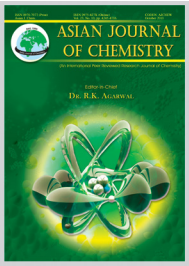

Synthesis and Crystal Structure of Complex $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$

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A mixed complex $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$ was synthesized based on the reaction of cadmium acetate and 2-aminobenzothiazole ($C_7H_6N_2S$) in methanol medium. The structure of this complex was characterized by elemental analysis and IR spectrum. The crystal structure of the compound $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$ was also determined by X-ray single crystal diffraction. The crystal belongs to crystal orthorhombic system with space group Pna2(1) and crystallographic data of the compound are: a = 0.88831(15) nm, b = 2.7449 (3) nm, c = 0.90103(16) nm, $\alpha = \beta = \gamma = 90°$, V = 2.1970(6) nm^3; D_c = 1.605 g/cm^3; Z = 4; F(000) = 1064; m = 1.214 mm^{-1}.

Key Words: Cd(II) complex, 2-Aminobenzothiazol, Crystal structure.

INTRODUCTION

Cadmium is one of the transition elements, whose positive ion is Cd^{2+} with empty valence electron tracks of 5*s* and 5*p* that can form complexes employed as insecticide, bactericide, pigment and paint. Cd^{2+} complexes are popular for new structures of one dimension, two dimension and netted supera-molecule and special property of polyporus materials, radiation and catalyst[1-8]. As an important industry intermediate in medicine,farm insecticide and dyestuff, 2-aminobenzothiazol ($C_7H_6N_2S$) is of biological activitity and good coordination for *n*-donor of the N atom and S atom on benzothiazol ring as well as the N atom of $-NH_2$[9-12]. Herein the synthesis and X-ray crystal structure of the mixed complex $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$ will be reported, which utlized 2-aminobenzothiazol and cadmium acetate as synthetic materials in methanol medium.

EXPERIMENTAL

Elemental analysis of C, H and N were carried out with a Perkin Elmer 240 elemental analyzer. IR spectra were measured by using KBr discs on a spectrum one BFT-IR spectrophotometer. The single crystal structure was determined by Siemens Smart-1000 CCD diffractometer. All starting materials were of chemical purity grade.

Synthesis of the complex: In 12 mL methanol medium, 0.5230 g (2 mmol) cadmium acetate and 1.2 g (4 mmol) 2-aminobenzothiazol were added, then the mixed solution was refluxed for 1 h to get a colourless transparent solution. After filtered, the solution was kept at room condition for two days and then the colourless pillar crystals resulted from it. The crystals were suitable for analysis of X-ray single crystal diffraction. IR (KBr, ν_{max}, cm^{-1}): 3321 m, 3127 m, 2981 w, 1900 w, 1645 m, 1591 s, 1435 s, 1419 s, 1340 w, 1300 m, 751 m, 677 w. Anal. calcd. (%) for $C_{18}H_{18}N_4O_4S_2Cd$: C 40.72, H 3.42, N 10.55. Found: C 40.63, 40.68, H 3.35, 3.39, N 10.51, 10.48.

Structure determination: A single crystal of $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$ with dimensions of 0.58 mm × 0.27 mm × 0.22 mm was selected for the experiment. X-ray diffraction data collection was performed by Siemens Smart-1000 CCD diffractometer with graphite-monochromatized MoK_α radiation (λ = 0.071073 nm) at 298(2) K,using Φ-ω scand mode. A total of 9195 reflections were collected in the range of $1.48° \le \theta \le 25.01°$, of which 2073 reflections were unique with Rint = 0.0460. The crystal structure was solved by the direct methods and fourier synthesis with SHELXS-97 program system[12] and refined by full-matrix least squares techniques on F^2 with SHELXS-97 program system[13]. The non-hydrogen atoms were refined anisotropycally and hydrogen atoms were added according to theoretical models. The final refinement converged at R = 0.0304 and wR = 0.0593 (ω=1/$[\sigma^2(F_0^2)^2 + (0.0464P)^2 + 5.5696P]$, where P = ($F_0^2 + 2F_C^2$)/3) for 1807 observed reflections with $I > 2\sigma(I)$. S = 1.068, (Δ/σ) max = 0.09(4), $(\Delta\rho)$ max = 401 and $(\Delta\rho)$ min = -356 e nm^{-3}. Crystallographic data of the mixed complex $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$ are listed in Table-1.

TABLE-1
CRYSTALLOGRAPHIC DATA OF THE COMPLEX $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$

Empirical	$C_{18}H_{18}N_4O_4S_2Cd$
Formula weight	530.88
Crystal system	Orthorhombic
Space group	Pna2(1)
a (nm)	0.888831(15)
b (nm)	2.7749(3)
c (nm	0.9013(6)
α (º)	90
β (º)	90
γ (º)	90
V (nm^3)	2.1970(6)
Z	4
Dc (g cm^{-3})	1.605
μ(MoKα) mm^{-1}	1.214
F(000)	1064
Range of θ/(º)	1.48 to 25.01
Index range	$-9 \le h \le 10$; $-22 \le k \le 32$; $-10 \le l \le 10$
Reflections collected/unique R(int))	9195/2073(0.0460)
Observed data ($I > 2\sigma(I)$)	1807
R1, wR2 ($I > 2\sigma(I)$)	0.0304, 0.0593
R1, wR2 (all data)	0.0369, 0.0611
GOF	1.068
Largest diff. peak and hole/(e nm^{-3})	401 and – 356

RESULTS AND DISCUSSION

IR spectrum: In the IR spectrum,symmetric and antisymmetric vibrations of $-NH_2$ of the 2-aminobenzothiazol in the complex exist at 3321 cm^{-1} and 3127 cm^{-1}, respectively. A strong absorption at 1645 cm^{-1} can be attributed to distortion vibration of $-NH_2$ in 2-aminobenzothiazol, which do not coordinate to the central cadmium atom. There are some framework vibrations of 2-aminobenzothiazol at 1591, 1435 and 1419 cm^{-1}. A strong absorption at 751 cm^{-1} means that adjacent double replaced benzene ring (benzene ring of 2-aminobenzothiazol)exist. The weak absorption at 2981 cm^{-1} can be attributed to $-CH_3$ of acetate, whereas there is a difference of 40 cm^{-1} between symmetric and antisymmetric vibrations of -COO group of acetate in the complex at 1340 cm^{-1} and 1300 cm^{-1}, respectively, which indicate the fact of double coordination of acetate.

Molecular and crystal structure: Single-crystal X-ray diffraction analysis reveal that mixed complex $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$ **1** crystallizes in orthorhombic system with Pna2(1) space group. The atomic coordinates and thermal parameters are listed in Table-2, the selected important bond parameters are given in Table-3, the molecule structure and packing diagram of the title compound in a unit cell are shown in Figs. 1 and 2. From the Fig. 1, the mixed complex $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$ **1** is a no electric charge complex, the central cadmium atom is coordinated by four O atoms from two acetate and two N atoms from two 2-aminobenzothiazol, where the N atom of $-NH_2$ do not coordinate to the central cadmium atom, forming a distorted octahedral coordination structure, which bond lengths of Cd(1)-O(1), Cd(1)-O(2), Cd(1)-O(3), Cd(1)-O(4), Cd(1)-N(1) and Cd(1)-N(3) are 0.2298(4), 0.2431(4), 0.2473(5), 0.2280(4), 0.2263(5) and 0.2271(5) nm, respectively. The angles around the cadmium atom range from 54.20(14)º to 150.28(17)º. From Fig. 2, the cell diagram of the titled complex **1** consist of four complexes molecules of $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$, one O atom of acetate in a title complex molecule acts as a hydrogen-bond donor to the H atom of $-NH_2$ group of 2-aminobenzothiazol in another title complex molecule forming the hydrogen bond of N-H···O with length rang from 0.2036 nm to 0.2126 nm, one S atom of 2-aminobenzothiazol in a title complex molecule acts as a hydrogen-bond donor to the H atom of $-NH_2$ group of the 2-aminobenzothiazol in same complex molecule forming the hydrogen bond of N-H···S with length of 0.301 5 nm, these hydrogen bonds data were given in Table-4.

TABLE-2
ATOMIC COORDINATES ($\times 10^4$) AND EQUIVALENT ISOTROPIC DISPLACEMENT PARAMETERS ($nm^2 \times 10^1$)

Atom	x	y	z	U (eq)
Cd(1)	8809(1)	6205(1)	5644(1)	42(1)
N(1)	8130(6)	5556(2)	4249(6)	48(1)
N(2)	10387(7)	5112(2)	4397(8)	78(2)
N(3)	8056(6)	6835(2)	4189(6)	45(1)
N(4)	6763(7)	7274(2)	5999(7)	65(2)
O(1)	11355(4)	6069(1)	5476(8)	54(1)
O(2)	10741(5)	6745(1)	6619(5)	51(1)
O(3)	8494(5)	5760(2)	8009(6)	60(1)
O(4)	6964(5)	6339(1)	7356(5)	51(1)
S(1)	8060(3)	4697(1)	3042(2)	74(1)
S(2)	7208(3)	7698(1)	3383(3)	84(1)
C(1)	8974(9)	5170(2)	3992(8)	57(2)
C(2)	6710(8)	5498(2)	3654(7)	50(2)
C(3)	6457(8)	5053(2)	2934(8)	62(2)
C(4)	5086(13)	4952(3)	2254(9)	82(3)
C(5)	3980(11)	5295(3)	2317(10)	88(3)
C(6)	4198(8)	5733(3)	3027(10)	75(2)
C(7)	5551(8)	5842(3)	3674(8)	59(2)
C(8)	7355(7)	7224(2)	4659(8)	46(2)
C(9)	8187(10)	7347(3)	2114(8)	72(2)
C(10)	8558(8)	6895(2)	2729(8)	53(2)
C(11)	9381(9)	6565(3)	1914(9)	68(2)
C(12)	9810(10)	6685(4)	497(14)	96(3)
C(13)	9427(14)	7127(5)	-122(12)	120(4)
C(14)	8623(12)	7466(4)	671(16)	111(3)
C(15)	11709(7)	6443(2)	6228(7)	45(2)
C(16)	13313(7)	6511(3)	6636(10)	76(2)
C(17)	7376(7)	6025(2)	8275(8)	44(2)
C(18)	6526(9)	5972(3)	9691(9)	76(2)

TABLE-3
SELECTED BOND LENGTHS (nm) AND ANGLES (º) OF $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$

Bond	Dist.	Bond	Dist.
Cd(1)-N(1)	0.2263(5)	Cd(1)-N(3)	0.2271(5)
Cd(1)-O(1)	0.2298(4)	Cd(1)-O(4)	0.2280(4)
Cd(1)-O(2)	0.2431(4)	Cd(1)-O(3)	0.2473(5)
Angle	(º)	Angle	(º)
N(1)-Cd(1)-N(3)	101.60(19)	N(1)-Cd(1)-O(4)	108.12(18)
N(3)-Cd(1)-O(4)	93.25(16)	N(1)-Cd(1)-O(1)	95.61(18)
N(3)-Cd(1)-O(1)	112.0(2)	O(4)-Cd(1)-O(1)	141.1(2)
N(3)-Cd(1)-O(2)	87.29(16)	O(4)-Cd(1)-O(2)	99.49(15)
O(1)-Cd(1)-O(2)	55.07(15)	N(1)-Cd(1)-O(3)	93.4(2)
N(3)-Cd(1)-O(3)	147.22(17)	O(4)-Cd(1)-O(3)	54.20(14)
O(1)-Cd(1)-O(3)	95.0(2)	O(2)-Cd(1)-O(3)	93.97(16)

TABLE-4
HYDROGEN BONDS OF THE TITLE COMPLEX $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$

D-H···A	d_{D-H}/nm	$d_{H..A}$/nm	θ_{DHA}/(°)	$d_{D..A}$/nm	Symmetry code
N2-H2A···O1	0.0860	0.2126	155.77	0.2931	
N2-H2A···S1	0.0860	0.3015	127.20	0.3601	[-x+2, -y+1, z+1/2]
N2-H2B···O3	0.0860	0.2055	159.76	0.2877	[-x+2, -y+1, z-1/2]
N4-H4A···O4	0.0860	0.2036	157.25	0.2849	
N4-H4B···O2	0.0860	0.2058	165.45	0.2898	[x-1/2, -y+3/2, z]

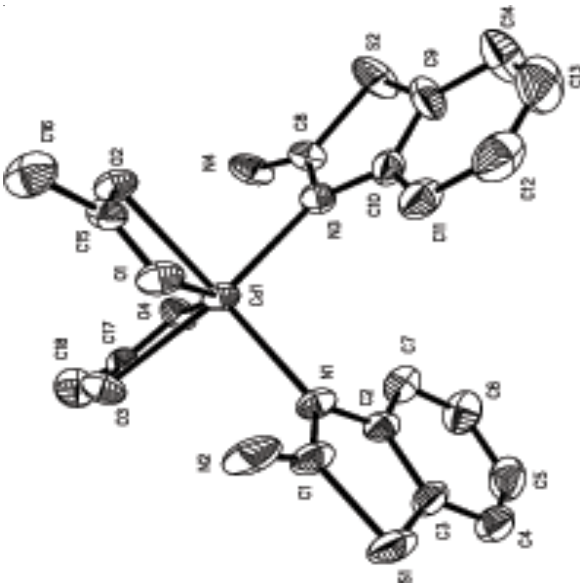

Fig. 1. A stereoview of the complex $[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$

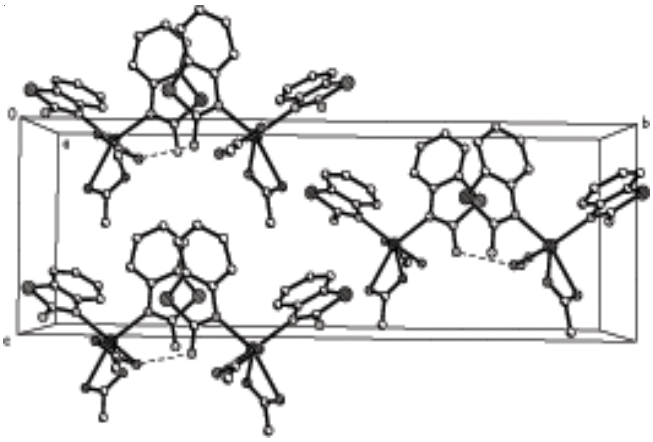

Fig. 2. Cell diagram of the complex$[Cd(CH_3COO)_2(C_7H_6N_2S)_2]$

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