NOTE

Synthesis and Crystal Structural of 1,10-Phenanthroline Lead(II) Complex

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A novel lead(II) complex $[\text{Pb}(\text{phen})_2(\text{NO}_3)] \cdot \text{NO}_3$ has been synthesized, with 1,10-phenanthroline (phen) and nitrate (NO_3^-) as ligands and characterized by IR spectra and single-crystal X-ray diffraction measurements. The crystal is monoclinic, space group $\text{P2}_1/\text{n}$ with unit cell parameters: $a = 16.6321(18) \text{ \AA}$, $b = 7.7550(8) \text{ \AA}$, $c = 18.0809(19) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 98.2880(10)^\circ$, $\gamma = 90^\circ$, $V = 2307.8(4) \text{ \AA}^3$, $Z = 4$, $M_r = 691.62$, $D_c = 1.991 \text{ Mg/cm}^3$, $\mu = 7.366 \text{ mm}^{-1}$, $F(000) = 1328$, $T = 298(2) \text{ K}$, $R = 0.0795$, $wR = 0.1980$ for 4068 reflections with $I > 2\sigma(I)$. In the molecular structure unit, lead(II) cation is coordinated by six donor atoms.

Keywords: Lead(II) complex, 1,10-Phenanthroline, Crystal structure.

The organic lead compounds which are formed by nitrogenous ligands, have better biological and pharmacological activities and a wide range of applications in medicine, pesticides and analytical chemistry¹⁻⁶.

So we synthesized a lead(II) complex with 1,10-phenanthroline ligand. The structure of the complex were characterized by infrared and X-ray single crystal diffraction.

All the reagents were of AR grade and used without further purification. Infrared spectra were recorded with a Nicolet 380 FT-IR spectrophotometer on KBr disks. The X-ray structure was determined by Siemens SMART CCD area-detector diffractometer.

Synthesis: 15 mL ethanol solution of 1,10-phenanthroline (10 mmol) was, respectively added to 30 mL H_2O solution of $\text{Pb}(\text{NO}_3)_2$ (10 mmol) and *p*-tert-butyl benzoic acid-Na (10 mmol) under stirring for 3 h. After being filtered, the solution was stand at room temperature for 1 week. The product was colourless granular single crystals. Yield 35 %. IR (KBr, ν_{max} , cm^{-1}): 3444, 3048, 1514, 1383, 1281, 849, 723.

Crystal structure determination: A single crystal of compound with dimensions of $0.45 \times 0.44 \times 0.40 \text{ mm}$ was selected for crystallographic data collection at $293(2) \text{ K}$ and structure determination on a Seimens SMART CCD area-detector diffractometer with graphite-monochromatic $\text{MoK}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). A total of 11285 reflections were collected in the range of $2.47^\circ \leq \theta \leq 25.02^\circ$, of which 4068 reflections were unique with $R_{\text{int}} = 0.0998$. L_p effects and empirical absorption were applied in data corrections. The structure was solved by direct methods and expanded using Fourier techniques. SHELXS-97 program system⁷ was used in the solution and refinement of the structure. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added according to the theoretical model. The final full-matrix least-squares refinement including 335 variable parameters for 4068 reflections with $I > 2\sigma(I)$ and gave the final $R = 0.0795$, $wR = 0.1980$. The weighting scheme was $w = 1/[s^2(F_o^2) + (0.1470P)^2 + 0.0000P]$, $P = (F_o^2 + 2F_c^2)/3$. $s = 1.026$. The maximum and minimum peaks on the final diffe-

TABLE-1
NON-HYDROGEN ATOMIC COORDINATES ($\times 10^4$) AND THERMAL PARAMETERS ($\times 10^3 \text{ \AA}^2$)

Atom	X	Y	Z	U(eq)
PB(1)	5028(1)	8714(1)	2394(1)	25(1)
N(1)	4473(8)	8421(15)	3651(7)	32(3)
N(2)	6003(7)	7534(16)	3420(6)	30(3)
N(3)	4591(7)	5487(16)	2247(7)	32(3)
O(1)	5664(8)	11626(16)	3120(7)	54(3)
O(2)	6336(7)	10636(16)	2286(7)	51(3)
O(3)	6703(10)	13050(20)	2822(11)	90(5)

TABLE-2
SELECTED BOND LENGTHS (Å) AND BOND ANGLES (°)

Bond	Length	Angle	(°)	Angle	(°)
PB(1)-N(2)	2.459(11)	N(2)-PB(1)-N(1)	65.4(4)	N(1)-PB(1)-O(1)	79.3(4)
PB(1)-N(1)	2.583(13)	N(2)-PB(1)-N(3)	82.1(4)	N(3)-PB(1)-O(1)	156.9(4)
PB(1)-N(3)	2.609(13)	N(1)-PB(1)-N(4)	136.9(4)	O(2)-PB(1)-O(1)	46.6(4)
PB(1)-N(4)	2.611(13)	N(3)-PB(1)-N(4)	63.5(4)	O(2)-PB(1)-O(5)	145.2(4)
PB(1)-O(2)	2.668(12)	N(2)-PB(1)-O(2)	78.8(4)	N(1)-PB(1)-O(6)	114.5(4)
PB(1)-O(1)	2.746(12)	N(1)-PB(1)-O(2)	120.8(4)	O(2)-PB(1)-O(6)	119.1(4)

rence fourier map are corresponding to 5.554 and -7.508 e/Å³ (CCDC No. 908533), respectively.

The atomic coordinates and thermal parameters are listed in Table-1 and the selected bond lengths and bond angles in Table-2, respectively. Fig. 1 shows diagram of the molecular structure of the complex [Pb(phen)₂(NO₃)]·NO₃. Fig. 2 shows a perspective view of the crystal packing in the unit cell. As shown in the Fig. 1, the center lead(II) cation is six-coordinated with four nitrogen atoms of the two phen ligands and two oxygen atoms of a nitrate anions.

Fig.1. Molecular structure of the complex [Pb(phen)₂(NO₃)]·NO₃

Fig. 2. Molecular packing arrangement in the unit cell

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