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NOTE

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

DONG XIE*, HUA CHENG and WEN-JIE BI

College of Chemistry and Chemical Engineering, Hefei Normal University, Hefei 230601, Anhui Province, P.R. China

*Corresponding author: E-mail: hxlw12@163.com

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A novel lead(II) complex $[Pb(phen)_2(NO_3)]\cdot NO_3$ 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 P2(1)/n with unit cell parameters: a =16.6321(18)Å, b = 7.7550(8)Å, c = 18.0809(19)Å, α = 90º, β = 98.2880(10)º, γ = 90º, V = 2307.8(4) Å^3, Z = 4, Mr = 691.62, Dc = 1.991 Mg/cm^3, μ = 7.366 mm^{-1}, F(000) = 1328, T = 298(2) K, R = 0.0795, wR = 0.1980 for 4068 reflections with I > 2σ(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 chemistry[1-6].

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 $Pb(NO_3)_2$ (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 × 0.44 × 0.40 mm was selected for crystallographic data collection at 293(2) K and structure determination on a Seimens SMART CCD area-detector diffractometer with graphite-monochromatic MoK_α radiation (λ = 0.71073 Å). A total of 11285 reflections were collected in the range of 2.47º ≤ θ ≤ 25.02º, of which 4068 reflections were unique with R_{int} = 0.0998. Lp effects and empirical absorption were applied in data corrections. The strucutre was solved by direct methods and expanded using Fourier techniques. SHELXS-97 program system[7] was used in the solution and refinement of the structure. The non-hydrogen atoms were refined anisotroically. 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 > 2σ(I) and gave the final R = 0.0795, wR = 0.1980. The weighting scheme was w = 1/[s^2(F_0^2) + (0.1470P)^2 + 0.0000P], P = (F_0^2+ 2F_0^2)/3. s = 1.026. The maximum and minimum peaks on the final diffe-

TABLE-1
NON-HYDROGEN ATOMIC COORDINATES (× 10^4) AND THERMAL PARAMETERS (× 10^{3}Å^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/Å^3 (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 $[Pb(phen)_2(NO_3)]\cdot NO_3$. 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.

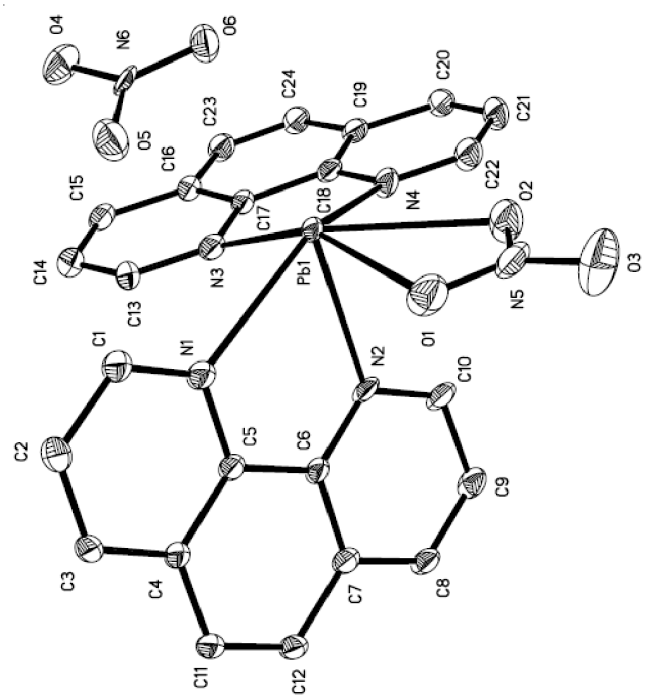

Fig.1. Molecular structure of the complex $[Pb(phen)_2(NO_3)]\cdot NO_3$

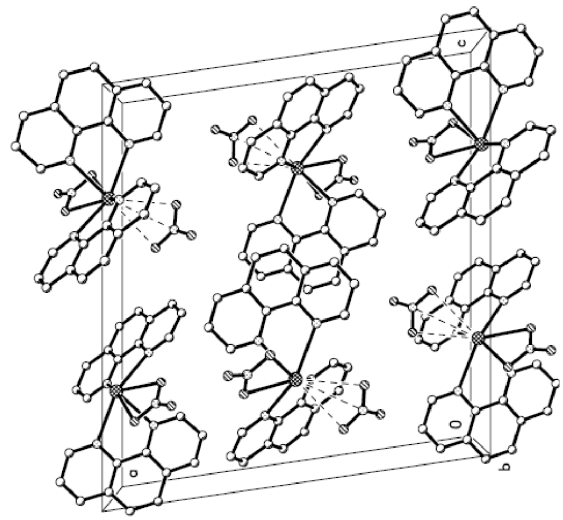

Fig. 2. Molecular packing arrangement in the unit cell

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