NOTE

Synthesis and X-Ray Diffraction of a Ternary Plumbum Complex

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A new ternary plumbum complex $[\text{Pb}(\text{phen})_2(\text{NO}_3)_2]$ (phen = 1,10-phenanthroline) has been synthesized and characterized by IR spectra and single-crystal X-ray diffraction. The crystal belongs to monoclinic crystal system and has space group $P2(1)/n$ with unit cell parameters: $a = 16.7304(15) \text{ \AA}$, $b = 7.7529(7) \text{ \AA}$, $c = 18.1308(17) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 98.2170(10)^\circ$, $\gamma = 90^\circ$, $V = 2327.6(4) \text{ \AA}^3$, $Z = 4$, $M_r = 691.62$, $D_c = 1.974 \text{ Mg/cm}^3$, $\mu = 7.303 \text{ mm}^{-1}$, $F_{(000)} = 1328$, $T = 298(2) \text{ K}$, $R = 0.0306$, $wR = 0.0574$ for 11276 reflections with $I > 2\sigma(I)$. In terms of the crystal structure, Pb(II) ion is eight-coordinated with four nitrogen atoms of the two phen ligands and four oxygen atoms of the two nitrate radical anions.

Key Words: Plumbum(II) complex, 1,10-Phenanthroline, Crystal structure.

Because of their rich structure and versatility, the design and synthesis of the phen(1,10-phenanthroline) coordinated complexes have attracted considerable interest in recent years¹⁻⁴. They have potential applications in biological activity, electrochemistry, spectrum and fluorescent, catalysis and molecular magnetism^{2,3,6-9}.

In this paper, we report the synthesis and X-ray structure of a ternary plumbum complex $[\text{Pb}(\text{phen})_2(\text{NO}_3)_2]$.

All reagents were of AR grade and used without further purification. IR spectra were recorded on a Nexus-870 spectrophotometer. The crystal structure was determined by Siemens SMART CCD area-detector diffractometer.

Synthesis of $[\text{Pb}(\text{phen})_2(\text{NO}_3)_2]$: A solution of 10 mmol of 1,10-phenanthroline in 10 mL ethanol, a solution of 5 mmol $\text{Pb}(\text{NO}_3)_2$ in 10 mL water and a solution of 5 mmol sodium ethylene glycol-bis-(2-amino-ethylether)-N,N,N',N'-tetraacetate in 10 mL water were mixed together under stirring for 2 h. After being filtered, the solution was kept at room condition for 6 d and then the well-shaped colourless four edges column single crystals were obtained. Yield 32 %. IR spectrum (KBr , cm^{-1}): 3500, 3420; 1630, 1560, 1420, 847, 717, 619.

Crystal structure determination: A single crystal (0.45 mm $\times$ 0.20 mm $\times$ 0.10 mm) was selected for crystallographic data collection at 298(2) K and structure determined with graphite monochromatic $\text{MoK}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). A total of 11276 reflections were collected in the range of $1.55^\circ \leq \theta \leq 25.02^\circ$, of which 4091 reflections were unique with $R_{\text{int}} = 0.0742$ and $R = 0.0306$ and $wR = 0.0574$, where

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)	3714(1)	2394(1)	35(1)
N(1)	4482(3)	3435(6)	3646(2)	35(1)
N(2)	6006(3)	2506(6)	3418(2)	36(1)
N(3)	5839(3)	1789(6)	1571(2)	40(1)
O(1)	6348(3)	5641(6)	2281(2)	68(1)
O(2)	5687(3)	6620(6)	3118(2)	67(1)
C(1)	3752(4)	3918(8)	3768(3)	45(2)
C(2)	3481(4)	3821(8)	4459(3)	52(2)

Fig. 1. Molecular structure of the complex $[\text{Pb}(\text{phen})_2(\text{NO}_3)_2]$

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

Bond	Length	Angle	(°)	Angle	(°)
Pb(1)-N(1)	2.574(4)	N(2)- Pb(1)-N(1)	65.59(14)	O(1)- Pb(1)-O(2)	46.20(13)
Pb(1)-N(2)	2.477(4)	N(2)- Pb(1)-N(4)	82.25(14)	O(2)- Pb(1)-O(5)	118.76(13)
Pb(1)-O(1)	2.698(4)	N(1)- Pb(1)-O(1)	120.72(13)	C(1)-N(1)- Pb(1)	125.1(3)
Pb(1)-O(2)	2.757(4)	N(1)- Pb(1)-O(2)	79.44(14)	C(6)-N(2)- Pb(1)	120.2(3)
N(5)-O(1)	1.243(6)	O(1)- Pb(1)-O(2)	46.20(13)	O(3)-N(5)-O(1)	121.8(7)

$w = 1/[s^2(F_0^2) + (0.0137P)^2 + 0.0000P]$, $P = (F_0^2 + 2F_{0^2})/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 1.881 and -1.327 e/Å³ (CCDC No. 908532), 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)₂]. Fig. 2 shows a perspective view of the crystal packing in the unit cell. As shown in the Fig. 1, the center plumbum(II) cation is eight-coordinated with four nitrogen atoms of the two phen ligands and four oxygen atoms of the two nitrate radical anions.

Fig. 2. Molecular packing arrangement in the unit cell

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