

## Research Article

# Synthesis, Crystal Structure and Thermal Properties of Lead(II) Complex with Bathophenanthroline and Benzoyltrifluoroacetate Ligands

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Received 13 April 2012; Accepted 15 May 2012

Academic Editor: Jorge Barros-Velazquez

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A new Pb(II) complex,  $[\text{Pb}(\text{bp})_2(\text{btfa})_2]$  **1**, has been synthesized with bathophenanthroline (bp) and benzoyltrifluoroacetone (Hbtfa) and characterized by elemental analysis IR and  $^1\text{H}$  NMR spectroscopy as well as by thermal properties and X-ray crystallography. The coordination number of the Pb(II) ions in **1** is eight, with the  $\text{PbN}_4\text{O}_4$  coordination polyhedron containing a stereochemically “inactive” electron lone pair with holodirected coordination spheres. In solid state, there are  $\pi \cdots \pi$ , C–H $\cdots$ F, C–H $\cdots$ O, and F $\cdots$ F interactions between adjacent units to generate 3D supramolecular structure.

## 1. Introduction

The  $\text{Pb}^{2+}$  cation has a  $[\text{Xe}]4f^{14}5d^{10}6s^2$  electronic configuration and exhibits an especially versatile character with respect to the HSAB theory [1] for which it appears as a borderline acid, able to bind to wide families of ligands [2] within very flexible coordination modes [3, 4]. The coordination chemistry of Pb(II) is mainly determined by two factors: (i) its large size (ionic radius 1.32 Å [5], covalent radius 1.54 Å [5] and van der Waals radius 2.00 Å [6]), which permits coordination numbers that range from quatione [7] to twelve [8] and (ii) its 6s electron pair, which may or may not play a role in the stereochemistry of lead(II) complexes [9]. The lone-pair activity can depend on (1) hard or soft ligands, (2) attractive or repulsive interactions among ligands, and (3) the number of electrons (charge) transferred from the ligands to the metal atom [3]. It has been proposed that holodirected structures do not necessarily exclude an inactive lone-pair [10].

The design and synthesis of new materials with desired chemical and physical properties have been of interest, and this involves the generation and study of structural motifs in crystals, which is essentially guided by precise topological control through the manipulation of weak intermolecular interactions [11]. There is a rich variety of intermolecular interactions that serve as tools in engineering such molecular assemblies [12]. Recently, in an effort to explore the role of weak intermolecular interactions among ligands in the stereochemical activity of valence shell electron lone pairs, the lead(II) complexes with fluorinated  $\beta$ -diketonate and neutral aromatic diamine-chelating ligands have been synthesized and characterized by X-ray crystal structure determination [13–25]. Fluorinated  $\beta$ -diketonate ligands are very good probes because of their ability for forming intermolecular C–H $\cdots$ F, F $\cdots$ F, and  $\pi \cdots \pi$  interactions. In this paper we report the synthesis and crystal structures of  $[\text{Pb}(\text{bp})_2(\text{btfa})_2]$  **1**, where bp and  $\text{btfa}^-$  are the abbreviations of bathophenanthroline and benzoyltrifluoroacetate ligands, respectively.

## 2. Experimental

**2.1. Material and Measurements.** All chemicals were reagent grade and used without further purification. FT-IR spectra were collected on a Mattson 1000 spectrophotometer using KBr pellets in the range of 4000–450  $\text{cm}^{-1}$ . Elemental analyses (CHN) were performed using a Carlo ERBA model EA 1108 analyzer and  $^1\text{H}$  NMR spectra were obtained with a Bruker spectrometer at 250 MHz in [D6]DMSO. Thermal analyses were carried out on a Perkin-Elmer instrument (Seiko Instruments).

**2.2. Crystallography.** For the crystal structure determination, the single crystal of compound 1 was used for data collection on a four-circle Rigaku R-AXIS RAPID-S diffractometer equipped with a two-dimensional area IP detector. The graphite-monochromatized Mo-K $\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ) and oscillation scans technique with  $\Delta\omega = 5^\circ$  for one image were used for data collection. The lattice parameters were determined by the least-squares methods on the basis of all reflections with  $F^2 > 2\sigma(F^2)$ . Integration of the intensities, correction for Lorentz, polarization effects, and cell refinement was performed using CrystalClear (Rigaku/MSI Inc., 2005) software [26]. The structures were solved by direct methods using SHELXS-97 [27] and refined by a full-matrix least-squares procedure using the SHELXL-97 program [27]. The final difference Fourier maps showed no peaks of chemical significance. Details of crystal data, data collection, structure solutions, and refinements are given in Table 1.

**2.3. Preparation of [Pb(bp) $_2$ (btfa) $_2$ ] (1).** Bathophenanthroline (0.184 g, 1 mmol) was placed in one arm of a branched tube [28] and lead(II) acetate (0.190 g, 0.5 mmol), and "Hbtfa" (0.206 g, 1 mmol) in the other. Methanol and ethanol ratio (2 : 3) was carefully added to fill arms, the tube sealed, and the ligand containing arm immersed in a bath at 60°C while the other was at ambient temperature. After 3 days, crystals had been deposited in the cooler arm which were filtered off, washed with acetone and ether, and dried in air, yield: 56%. M.p. 250°C Analysis: found: C: 62.54, H: 3.20, N: 4.51%. Calculated for  $\text{C}_{68}\text{H}_{44}\text{F}_6\text{N}_4\text{O}_6\text{Pb}$ : C: 62.66, H: 3.38, N: 4.30%. IR ( $\text{cm}^{-1}$ ) selected bands: 680 (versus), 845 (versus) (C–H), 956 (m), 1032 (s), 1156 (s), 1224 (versus, C–F), 1400, 1480, 1585 (s, aromatic ring), 1629 (versus, C=O), 3062 (w, C–H aromatic).  $^1\text{H}$  NMR (DMSO,  $\delta$ ): 9.00–9.20 (m, 4H), 7.00–8.00 (m, 18H), 5.86 (s, 2H, =CH–) ppm.  $^{13}\text{C}$  NMR (DMSO,  $\delta$ ): 97.33 (=CH–), 117.60 (–CF $_3$ ), 124.52–138.11 (aromatic carbons), 137.57–153.10 ((hetero)aromatic C-atoms), 168.50 (C=O), 179.90 (C=O) ppm.

## 3. Result and Discussion

**3.1. Preparation and Thermal Behavior.** The reaction conditions always have great effect on the single-crystal growth. Interestingly, the molar ratio of solvents is important for the growth of single crystals.

TABLE 1: Crystal data and structure refinement for (1).

Identification code	[Pb(dpphen) $_2$ (tfpb) $_2$ ]
Empirical formula	$\text{C}_{68}\text{H}_{46}\text{F}_6\text{N}_4\text{O}_4\text{Pb}$
Formula weight	1304.3
Crystal system	monoclinic
Space group	C2/c
Unit cell dimensions	$a = 12.0945 (2) \text{ \AA}$ $b = 31.6546 (9) \text{ \AA}$ $c = 15.2474 (4) \text{ \AA}$ $\beta = 94.955 (3)^\circ$
Volume	$5815.6 (2) \text{ \AA}^3$
Z	4
Density (calculated)	$1.487 \text{ g cm}^{-3}$
Absorption coefficient	$2.973 \text{ mm}^{-1}$
$F(000)$	2600
$\Theta$ -range for data collection	2.16 to 26.47
Index ranges	$-15 \leq h \leq 15$ $-39 \leq k \leq 39$ $-19 \leq l \leq 19$
Reflections collected	57689
Independent reflections	5963 [ $R(\text{int}) = 0.14$ ]
Completeness to theta	99.1%
Data/restraints/parameters	3795/0/383
Goodness of fit on $F^2$	1.043
Final R. [ $I_0 > 2\sigma(I_0)$ ]	$R_1 = 0.092$ , $wR_2 = 0.1996$
R indices (all data)	$R_1 = 0.144$ , $wR_2 = 0.228$
Largest diff. peak, hole	1.576, $-0.808 \text{ e. \AA}$

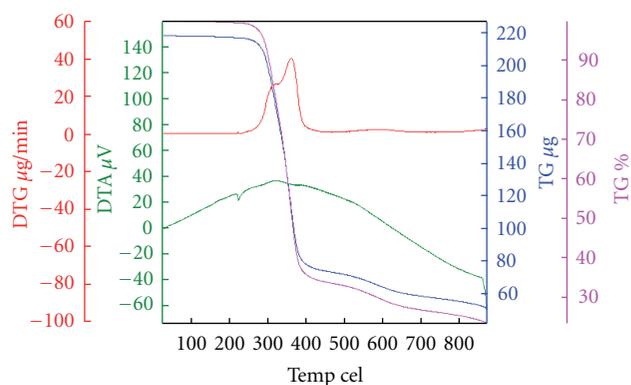


FIGURE 1: Thermal behavior of [Pb(bp) $_2$ (btfa) $_2$ ] (1).

In order to examine the thermal stability of the compound, thermal gravimetric (TG) and differential thermal analyses (DTA) were carried out for complex 1 between 30 and 800°C in the static atmosphere of air (Figure 1). The TG curve of compound 1 indicates that this compound decomposes at 300°C (1 has good thermal stability related to reported structures of Pb(II) with  $\beta$ -diketonates [13–25]). The ligands bp and btfa $^-$  decompose at 300–800°C with exothermic effect. The solid residue formed is suggested to be PbO (observed 19.50%, calcd. 17.12%).

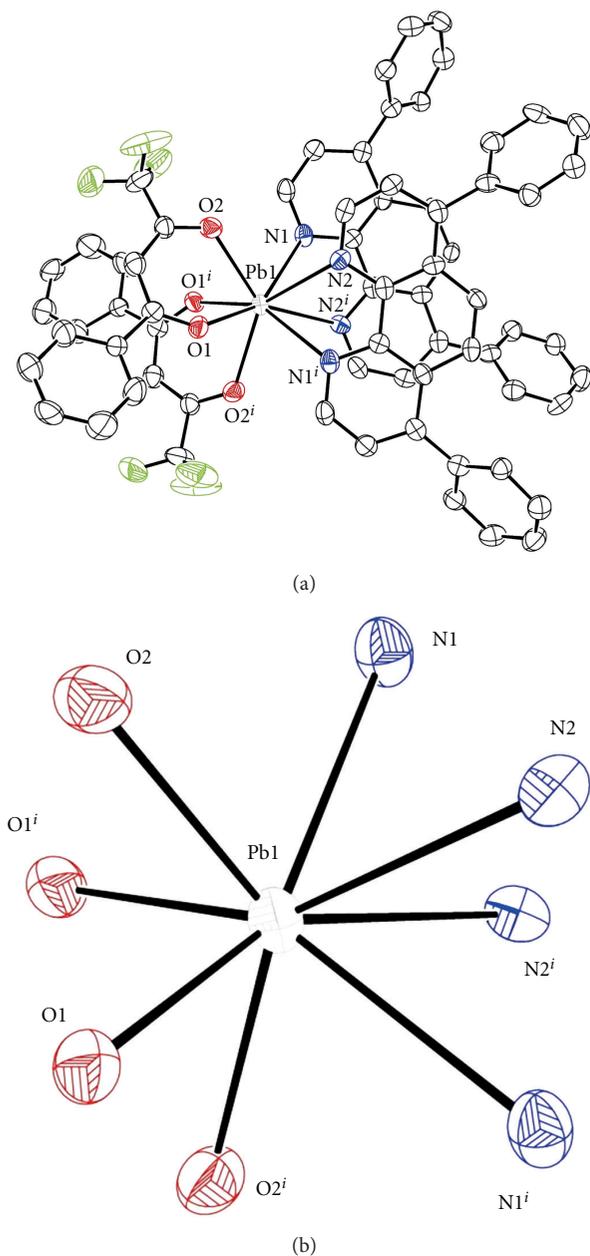


FIGURE 2: (a) Ortep diagram of complex **1**. (b) Representation of the hole in the coordination sphere of **1**. The coordination geometry is as holodirected [symmetry code (*i*):  $-x, y, 1/2 - z$ ].

**3.2. Structural Description.** The solid-state structure of compound **1** was determined by single-crystal X-ray diffraction. The Pb(II) complex crystallizes in the monoclinic space group  $C2/c$  (Table 1). The asymmetric unit contains half of the molecule and it has the  $C_2$  symmetry (Figure 2). Selected bond lengths, and angles are listed in Table 2. The molecule contains one Pb(II), two “Bathophenanthroline” ligands and two benzoyltrifluoroacetates anions (Figure 2(a)). The coordination number of Pb(II) in this complex is eight and each lead atom is chelated by four nitrogens of “bp” ligands with Pb-N distances of 2.779 (3), 2.871 (3) Å, and four

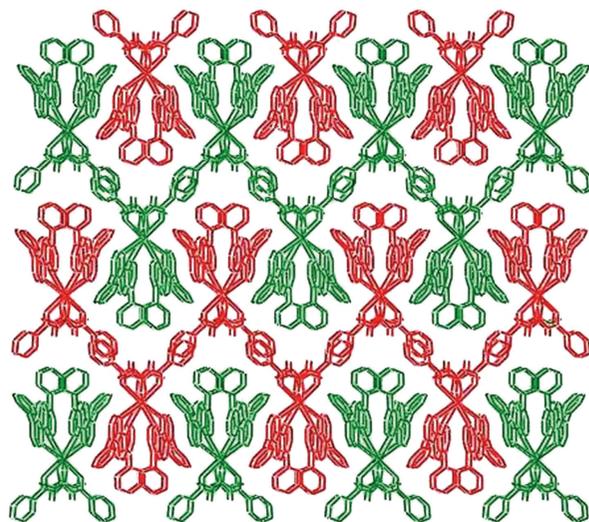


FIGURE 3: Perspective view of the complex down the *a*-axis, packing of mononuclear complex **1** forms 3D networks via  $\pi$ - $\pi$  interactions.

TABLE 2: Selected bond lengths (Å) and angles ( $^\circ$ ) for (**1**).

Pb-N1	2.779 (9)	Pb-O1	2.482 (8)
Pb-N1 <sup><i>i</i></sup>	2.779 (9)	Pb-O1 <sup><i>i</i></sup>	2.482 (8)
Pb-N2	2.871 (10)	Pb-O2	2.506 (8)
Pb-N2 <sup><i>i</i></sup>	2.871 (10)	Pb-O2 <sup><i>i</i></sup>	2.506 (8)
O1-Pb1-O1 <sup><i>i</i></sup>	91.8 (4)	O2 <sup><i>i</i></sup> -Pb1-N1	130.2 (3)
O1-Pb1-O2	71.1 (3)	N1-Pb1-N1 <sup><i>i</i></sup>	113.3 (4)
O1 <sup><i>i</i></sup> -Pb1-O2	75.8 (3)	O1-Pb1-N2 <sup><i>i</i></sup>	153.5 (3)
O1 <sup><i>i</i></sup> -Pb1-N1	84.4 (3)	O1 <sup><i>i</i></sup> -Pb1-N2 <sup><i>i</i></sup>	99.2 (3)
O2-Pb1-N2 <sup><i>i</i></sup>	135.0 (3)	N1-Pb1-N2 <sup><i>i</i></sup>	56.3 (3)
O2-Pb1-O2 <sup><i>i</i></sup>	131.7 (4)	N1 <sup><i>i</i></sup> -Pb1-N2 <sup><i>i</i></sup>	73.9 (3)
O1-Pb1-N1	149.6 (3)	O2-Pb1-N2	85.0 (3)
O2-Pb1-N1	78.7 (3)	N2 <sup><i>i</i></sup> -Pb1-N2	81.5 (4)

*i*:  $-x, y, 1/2 - z$ .

oxygens of two “btf<sup>-</sup>” anions with Pb-O distances of 2.482 (4) and 2.506 (4) Å (Figure 2(b)).

The arrangement of bathophenanthrolines and benzoyltrifluoroacetates does not suggest any gap or hole in coordination geometry around the metal ion (Figure 1(b)), indicating that the lone pair of electrons on lead(II) is inactive and the geometry around Pb(II) sphere is holodirected [29]. According to suggestions of Shimoni-Livny et al. [3, 4] the hemidirected geometry is present in all lead(II) compounds with low coordination numbers (2–5) and also quite common for coordination numbers of 6 to 8, while holodirected geometry becomes dominant at high coordination numbers of 9 and 10. Since the presence of four bulk ligands increases steric crowding around lead(II) and results in strong interligand repulsions. This may be the reason of the disappearance of the gap in the coordination polyhedron, thereby resulting in less common holodirected geometry.

TABLE 3: Inter- and intramolecular interactions for **1**.

A...H-B	A...H/Å	A...B/Å	A...H-B/ <sup>o</sup>
<b>1</b>			
O1...H28-C28 ( $-x, -y, 1-z$ )	2.725	3.360 (2)	146.86
F3...H19-C19 ( $x, -y, -1/2 + z$ )	2.707	3.308 (2)	123.10
F2...F2( $1-x, y, 1/2-z$ )	—	2.868	—
$\pi \cdots \pi$ (inter, slipped face-to-face between bp ligands)	—	3.496	—
$\pi \cdots \pi$ (slipped edge-to-edge between aromatic rings of btfa <sup>-</sup> )	—	3.507	—
$\pi \cdots \pi$ (intra, slipped face-to-face between bp ligands)	—	3.586	—

A search was generally made for intermolecular approaches in the complex [Pb(bp)<sub>2</sub>(btfa)<sub>2</sub>] (**1**). The interesting feature is that there are F...F interactions, the weak hydrogen bonding, between the fluorine atoms of btfa<sup>-</sup> with the distances of F2...F2( $1-x, y, 1/2-z$ ) = 2.868 Å and F...H-C, O...H-C interactions (Table 3) values suggest strong interactions within this class of weak noncovalent contacts [30]. There are  $\pi$ - $\pi$  stacking interactions between the aromatic rings that belong to adjacent chains in **1**. The interplanar distances range between aromatic rings in this compound is the normal  $\pi$ - $\pi$  stacking interaction (Table 3) [31, 32]. Consequently, F...F, C-H...O [33], C-H...F [34-37], and  $\pi$ - $\pi$  interactions allow the mononuclear complexes to form a hybrid three-dimensional network (Figure 3).

In spite of the recent structural study of fluorine  $\beta$ -diketonates complexes of Pb(II) with heteroaromatic ligands (1,10-phenanthroline, 2,9-dimethyl-1,10-phenanthroline) and other Pb(II) compounds using N-donor ligands and diketonates [13-25], this complex is as discrete mononuclear complex, and coordination geometry of **1** is holo-directed. In recent structural study of dinuclear complexes, fluorine  $\beta$ -diketonates anion has a role of bridging ligand [13-25].

The obvious question then is whether the weak interactions have stretched coordinate bonds to result in ligand stacking or whether it is the stacking interaction which has imposed a positioning of the donor atoms for forming the weak interactions in the packing. In conclusion, a subtle interplay among lone-pair activity and strong and weak interactions appears to control the packing motifs in the crystal structure of **1**. Our current results suggest that while interactions involving "organic fluorine" have a significant influence in generating supramolecular assemblies in inorganic solids, the general use of these interactions for the a priori prediction of packing motifs is yet to be fully understood and harnessed.

## Disclosure

Crystallographic data for the structure reported in the paper have been deposited in the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-864329 for the complex **1**. Copies of this information can be obtained for free from The Director, CCDC, 12 Union Road,

Cambridge, CB2 IEZ, UK (fax: 044-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/).

## Acknowledgment

Support of this investigation by Payame Noor University is gratefully acknowledged by F. Marandi.

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