Synthesis and crystal structure of one new Pb(II) complex constructed by 1,10-phenanthroline and two carboxylate ligands

Lu-Lin Zhang, Shi-Li Tang, De-Jun Li, Yuan-Zheng Cheng*, and Li-Ping Zhang*

Abstract: The complex [Pb(phen)(4-NB)(CH\textsubscript{3}COO)] of lead(II) was prepared and characterized by means of elemental analysis, FT-IR, and single crystal X-ray analysis, where phen = 1,10-phenanthroline, 4-NB = 4-nitrobenzoate. The single crystal X-ray analysis indicates that the complex is a monomeric species, including two carboxylate ligands, and adopts a hemidirected structure. It is further extended by intermolecular C−H···O hydrogen bonds, π−π interactions and secondary Pb···O interactions to form two-dimensional supramolecular architecture.

Keywords: lead(II), 1,10-phenanthroline, 4-nitrobenzoate, acetate, crystal structure

The coordination chemistry of lead(II) with N and O donor ligands has been studied extensively in recent years because of their intriguing structures and potential applications in some fields, such as luminescence, organic light-emitting diode, catalysis, nano materials, and antimicrobial (Alizadeh and Amani, 2011; Ding et al., 2008; Jin, 2020; Kazak et al., 2009; Kowalik et al., 2020; Li, 2019; Ma et al., 2018; Mirtamizdoust, 2017; Najafi et al., 2013; Sahu et al., 2013; Singh et al., 2016; Song et al., 2018; Wang et al., 2014; Xiong et al., 2012; Yang et al., 2016; Zhang et al., 2011; Zhao et al., 2007). Lead(II) has an electron configuration of [Xe]4f\textsuperscript{14}5d\textsuperscript{10}6s\textsuperscript{2}, with large ionic radius. It is able to form compounds with broad range of coordination numbers (Balch and Oram, 1987; Du et al., 2006; Fan and Zhu, 2006; Jin et al., 2010; Li et al., 2012). Due to the presence of the 6s lone-pair electrons, which may or may not be stereochemically active, Pb\textsuperscript{2+} complexes display either hemidirected or holodirected structures. The lone pair electron on lead(II) is stereochemically inactive and ligand-to-metal bonds are distributed evenly around the lead(II) ion is termed holodirected, otherwise it is called hemidirected (Janiak et al., 2000; Pellissier et al., 2007; Shimoni-Livny et al., 1998). Secondary bonds, as a kind of non-covalent interaction, usually existed in the hemidirected lead(II) complexes and formed by the stereochemically active lone pair, such as Pb···O, Pb···N, Pb···C, Pb···Cl, Pb···S, and Pb···π, which have some effect on the structures and properties of the lead(II) complexes (Catalano et al., 2015; Chen et al., 2015; Cheng et al., 2014b; Huang et al., 2017; Kowalik et al., 2020; Meng et al., 2009; Miao and Zhu, 2008; Mirdya et al., 2019, 2020; Yang et al., 2012; Zhao et al., 2007). Thus, Pb\textsuperscript{2+} ion is an ideal candidate for the construction of novel complexes (including monomeric compounds and metal–organic frameworks). In the reported lead(II) complexes, a number of compounds were tested for fluorescence properties, and the presence or absence of stereochemically active lone-pair Pb(II) has possible effect on luminescence of coordination compounds (Chen et al., 2015; Katz et al., 2008; Meng et al., 2009; Nugent et al., 2015). Therefore, some Pb(II) complexes exhibiting good luminescent properties have been observed (Alizadeh and Amani, 2011; Ding et al., 2008; Kowalik et al., 2020; Li, 2019; Ma et al., 2018; Sahu et al., 2013; Song et al., 2018; Xiong et al., 2012; Yang et al., 2016; Zhang et al., 2011; Zhao et al., 2007). Lead(II) compounds having catalytic and antimicrobial activities, fabricating organic light-emitting diode and nano materials have also been occasionally reported. For example, Wang et al. (2014) reported eleven germanium(II), tin(II), and lead(II) amino-(ether)-phenolate monomeric complexes and nine of them in the catalysis of the immortal ring-opening polymerisation (iROP) of \(\alpha\)-lactide (\(\alpha\)-LA) or racemic-lactide (rac-LA) upon addition of iPrOH were
tested, it is surprising to find the catalytic activity increases with metal size according to Ge$^{II}$ < Sn$^{II}$ < Pb$^{II}$. Singh et al. (2016) reported lead(II) complexes with semicarbazone and thiosemicarbazones derived from (2-hydroxyphenyl)(pyrrolidin-1-yl)methanone assessed as antibacterial and antifungal agents and the results interestingly showed the lead(II) complexes have higher activity than the free ligands. Najafi et al. (2013) synthesized a new complex, [Pb(µ-8-Quin)$_2$], and fabricated OLEDs by the prepared complex. The experiment results demonstrated the complex dye can serve as an electron transport layer and light emission layer along with the PVK as a hole transport layer in organic light-emitting diodes. Mirtamizdoust introduced one method of preparing PbO nanoparticles via thermolysis of lead(II) metal-organic coordination polymer (Mirtamizdoust, 2017). Most of the above compounds are constituted with N and O donor ligands. Which may be why Pb(II) compounds are increasingly synthesized and tested in some certain properties despite lead is a heavy toxic metal. 4-Nitrobenzoic acid has two functional groups, carboxyl (–COOH) and nitryl (–NO$_2$), whose carboxylate group can serve as good candidates to tune structures because of their versatile coordination properties, which can bind to metal ions with monodentate, bridging, and chelating modes (Kar et al., 2011; Lv et al., 2019; Ren et al., 2006; Sarma and Baruah, 2009; Tsaryuk et al., 2012; Zhu et al., 2012). In the complexes formed by 4-nitrobenzoate, the nitryl group can be uncoordinated but its oxygen atom can participate in generating the hydrogen bonds as H-acceptor, which often acts as a contribution in the construction of interesting supramolecular architectures (Ren et al., 2006; Saha et al., 2019; Srinivasan et al., 2009; Xu et al., 2012). Moreover, the introduction of the neutral ligands can adjust the coordination modes of 4-nitrobenzoate with metal ions resulting in diverse structures. 1,10-Phenanthroline, as an excellent N-donor bidentate chelating ligand, not only was often used to construct novel fascinating molecular architectures and build supramolecular structures through weak interactions (Liu et al., 2004; Wan et al., 2003), but also to produce compounds of various properties (Erxleben, 2018; Lemercier et al., 2018; Sammes and Yahioğlu, 1994). Based on the reasons above mentioned, 4-nitrobenzoic acid was chosen for the construction of novel lead(II) complex containing the ligand 1,10-phenanthroline. 

Herein we report the preparation and crystal structure of [Pb(phen)(4-NB)(CH$_3$COO)] (Scheme 1), obtained by reactions of lead(II) acetate, 1,10-phenanthroline, and 4-nitrobenzoic acid.

The molecular structure of the complex determined by single crystal X-ray diffraction method and the result shows that compound crystallizes in the monoclinic P2$_1$/n space group, with four formula units in the unit cell, and exhibits monomeric species. As shown in Figure 1a, Pb(II) ion adopts a six-coordinated geometry completed by two nitrogen donors from one 1,10-phenanthroline molecule, two oxygen atoms from one 4-NB ligand, and two oxygen atoms from one acetate ion to generate a N$_2$O$_4$ environment with a deformed pentagonal pyramid, which is like an umbrella. The Pb(II) atom is located in the at the top of the umbrella (Figure 1b) and the bond Pb1–O5 is umbrella handle. The Pb1–O bond lengths fall in the range of 2.359(4)-2.656(4) Å (Table 1), which can be well comparable with those in the reported compounds (Lv et al., 2019; Mahjoub and Morsali, 2002; Morsali et al., 2003; Sarma and Baruah, 2009; Xu et al., 2012; Zhao et al., 2013). The Pb1–N bond distances [2.586(5) and 2.658(5) Å] (Table 1) are typical values and are similar to Pb–N
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bonds in previously published Pb(II)-complexes containing 1,10-phenanthroline ligand (Cheng et al., 2014a; Fan and Zhu, 2006; Mahjoub and Morsali, 2002; Morsali et al., 2003; Xu et al., 2012). IR spectra of the complex were analyzed on the basis of the crystal structure. The band at 3049 cm\(^{-1}\) can be assigned to the C−H stretching of phenyl ring. According to IR spectra of the reported complexes [Pb(phen)]\(_2\)(4-NBA)\(_2\)·2(NO\(_3\))·H\(_2\)O (Xu et al., 2012) and [Pb(8-OQ)(4-NB)] (Lv et al., 2019), the \(\nu_{as}(\text{COO}^-)\) and \(\nu_{s}(\text{COO}^-)\) at 1560 and 1385 cm\(^{-1}\) are attributed to the carboxylate group of 4-nitrobenzoate, the \(\nu_{as}(\text{COO}^-)\) and \(\nu_{s}(\text{COO}^-)\) at 1616 and 1427 cm\(^{-1}\) are attributed to the carboxylate group of acetate, respectively. The differences between \(\nu_{as}(\text{COO}^-)\) and \(\nu_{s}(\text{COO}^-)\) in the two carboxylate groups are approximately 180 cm\(^{-1}\), which is close to \(\Delta\nu = [\nu_{as}(\text{COO}^-) - \nu_{s}(\text{COO}^-)]\) values in [Pb(phen)]\(_2\)(4-NBA)\(_2\)·2(NO\(_3\))·H\(_2\)O and [Pb(8-OQ)(4-NB)] whose COO\(^-\) groups adopted chelating mode, indicating the COO\(^-\) groups from 4-nitrobenzoate and acetate in this complex all chelated Pb(II) ion. The characteristic absorption peaks at 1509 and 1340 cm\(^{-1}\) are assigned to the asymmetric stretching vibration peaks and the symmetric stretching vibration peaks of the NO\(_2\) group, respectively. The characteristic peaks of the 1,10-phenanthroline are at about 1404, 832, and 724 cm\(^{-1}\).

In the complex, weak intermolecular hydrogen bonds (Figure 2, Table 2) are observed: (1) One intramolecular C−H···O hydrogen bond exists between 1,10-phenanthroline and 4-NB ligand. (2) Four intermolecular C−H···O hydrogen bonds are found between 1,10-phenanthroline and two anion (4-NB, CH\(_3\)COO\(^-\)) ligands. There are five

Table 1: Selected bond lengths (Å) and angles (deg) for the compound

| Bond Lengths (Å) | Bond Angles (deg) |
|-----------------|------------------|
| Pb1-O1          | 2.656(4)         | Pb1-O2          | 2.488(4)         |
| Pb1-O5          | 2.359(4)         | Pb1-O6          | 2.615(4)         |
| Pb1-N1          | 2.586(5)         | Pb1-N2          | 2.658(5)         |
| O1-Pb1-O2       | 51.09(14)        | O1-Pb1-O5       | 77.42(14)        |
| O1-Pb1-O6       | 76.52(13)        | O2-Pb1-O5       | 80.06(14)        |
| O2-Pb1-O6       | 116.35(13)       | O5-Pb1-O6       | 52.48(14)        |
| O1-Pb1-N1       | 127.28(13)       | O1-Pb1-N2       | 149.99(13)       |
| O2-Pb1-N1       | 77.15(14)        | O2-Pb1-N2       | 134.48(14)       |
| O5-Pb1-N1       | 85.39(14)        | O5-Pb1-N2       | 75.62(14)        |
| O6-Pb1-N1       | 128.23(13)       | O6-Pb1-N2       | 76.74(14)        |
| N1-Pb1-N2       | 63.17(14)        | |

Figure 1: The molecular structure of complex at 50% ellipsoids for non-hydrogen atoms.
face to face π–π interactions (Figure 3, Table 3) (Janiak, 2000) between adjacent 1,10-phenanthroline molecules, between 1,10-phenanthroline ligand and 4-NB ligand, between neighboring 4-NB ligands. Finally, these hydrogen bonds and face to face π–π interactions extend the monomer structure into a 2-D supramolecular architecture.

The complex adopts a hemidirected structure suggesting that 6s lone pair electrons of Pb1 is stereochemically active (Figure 1b) (Janiak et al., 2000; Pellissier et al., 2007; Shimoni-Livny et al., 1998). The direction of Pb1–O and Pb1–N bonds is irregularly distributed through the entire sphere around the lead(II) ion, and an obvious gap trans to the CH$_3$COO$^-$ anion can be found in the lead coordination sphere. The coordinating atoms occupy less than a half of a sphere surrounding the lead atom with the dihedral angle of 152.7° between the Pb1 N1 N2 plane and the plane Pb1 O1 O2. In addition, two intermolecular longer secondary Pb⋯O interactions, Pb⋯O4$^i$ bond of 3.335 Å and Pb⋯O6$^i$ bond of 3.096 Å (symmetry code $i$: 2-x, 1-y, 1-z; $ii$: 2-x, 2-y, 1-z), are discovered (Figure 4) (Huang et al., 2017; Miao and Zhu, 2008), which connect the adjacent [Pb(phen)(4-NB)(CH$_3$COO)] molecular and further stabilize 2-D supramolecular architecture. The values of two Pb⋯O bonds are larger than the sum of the ionic radii but obviously less than the sum of the van der Waals radii (3.54 Å) (Bondi, 1964). The two secondary bonds also confirmed Pb(II) ion adopting hemidirected structure.

The Pb(II) complex based on 1,10-phenanthroline and 4-nitrobenzoic acid with the Pb(CH$_3$COO)$_2$ was obtained and structurally characterized. The compound was a monomeric species involving hemidirected Pb(II) atom, and displayed 2-D supramolecular architecture constructed via intermolecular hydrogen bonds, π–π interactions and secondary Pb⋯O interactions. In this complex, the coordinate bonds around Pb(II) ion and non-covalent interactions probably affect the energy transfer and improve the luminescence properties of the compound compared with those of the free ligands.

**Experimental**

A mixture of Pb(CH$_3$COO)$_2$·3H$_2$O (0.1910 g, 0.50 mmol), 1,10-phenanthroline monohydrate (0.1980 g, 1.0 mmol), 4-nitrobenzoic acid (0.1671 g, 1.0 mmol) and NaOH (0.0400 g, 1.0 mmol) in a water/DMF/methanol (2:1:1, v/v/v) solution (20 mL) was refluxed for 4 h and filtered. Yellow block crystals were obtained after 23 days. All

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**Table 2:** H-Bonding geometry parameters (Å and deg) for the compound

| Type   | D–H⋯A       | D–H (Å) | H⋯A (Å)  | D⋯A (Å) | D–H⋯A (deg) |
|--------|-------------|---------|----------|---------|-------------|
| Intra  | C2⋯H2⋯O2   | 0.93    | 2.40     | 3.079(8) | 129         |
| Inter  | C4⋯H4⋯O1$^i$ | 0.93    | 2.59     | 3.476(8) | 160         |
| Inter  | C6⋯H6⋯O6$^i$ | 0.93    | 2.42     | 3.326(8) | 165         |
| Inter  | C9⋯H9⋯O3$^ii$ | 0.93   | 2.59     | 3.365(9) | 141         |
| Inter  | C10⋯H10⋯O5$^iii$ | 0.93  | 2.51     | 3.325(7) | 147         |

Symmetry code: ($i$) ·1+x, y, z; ($ii$) ·1+x, 1+y, z; ($iii$) 3/2-x, 1/2+y, 1/2-z.

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**Figure 2:** Hydrogen bonds as green dashed lines. Symmetry code: ($i$) ·1+x, y, z; ($ii$) ·1+x, 1+y, z; ($iii$) 3/2-x, 1/2+y, 1/2-z.
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**Figure 3:** π–π interactions in complex. Symmetry code: (i) 1-x, 2-y, 1-z; (ii) 3/2-x, 1/2+y, 1/2-z; (iii) 2-x, 1-y, 1-z.

**Table 3:** π–π interactions for the compound

| Ring (I) → Ring (J) | Centroid to centroid distance (Å) | θ (deg) | Slippage distance (Å) |
|-------------------|----------------------------------|--------|----------------------|
| Cg1 → Cg2′        | 3.710 (3)                        | 23.3   | 1.465                |
| Cg3 → Cg3′        | 3.543 (3)                        | 18.0   | 1.095                |
| Cg2 → Cg4′        | 3.690 (3)                        | 22.1   | 1.391                |
| Cg3 → Cg4′        | 3.574 (3)                        | 16.8   | 1.033                |
| Cg4 → Cg4′        | 3.570 (3)                        | 22.2   | 1.351                |

Symmetry code: (i) 1-x, 2-y, 1-z; (ii) 3/2-x, 1/2+y, 1/2-z; (iii) 2-x, 1-y, 1-z. Ring code: Cg1: N1/C1 → C5; Cg2: N2/C8 → C12; Cg3: C1/C5 → C8/C12; Cg4: C14 → C19. θ is the angle between the Ring (I) → Ring (J) vector and the normal to Ring (I).

**Figure 4:** Secondary Pb⋯O interaction shown as green dashed lines. Symmetry code: (i): 2-x, 1-y, 1-z; (ii): 2-x, 2-y, 1-z.
Table 4: Crystal data and refinement parameters

| Parameter                  | Value            |
|----------------------------|------------------|
| Empirical formula          | \( C_{21}H_{15}N_3O_6Pb \) |
| Formula weight             | 612.56           |
| Crystal dimensions (mm)    | \( 0.20 \times 0.22 \times 0.25 \) |
| Crystal system             | Monoclinic       |
| Space group                | P21/n            |
| a (Å)                      | 10.6490(5)       |
| b (Å)                      | 13.9872(6)       |
| c (Å)                      | 13.6435(6)       |
| \( \alpha \) (°)           | 90.00            |
| \( \beta \) (°)            | 106.422(S)       |
| \( \gamma \) (°)           | 90.00            |
| V (Å³)                     | 1949.29(16)      |
| Z                          | 4                |
| \( D_c \) (g·cm⁻³)         | 2.807            |
| \( \mu \) (mm⁻¹)           | 8.702            |
| F (000)                    | 1168             |
| T (K)                      | 145(1)           |
| \( \lambda \) (Å)          | MoKα(0.71073)    |
| \( \theta \) Range (°)    | 3.4-25.0         |
| Measured reflections       | 9501             |
| Unique reflections         | 3444             |
| Observed reflections       | 2991             |
| No. of parameters refined  | 281              |
| R1, WR2 [I>2σ(I)]          | 0.0326, 0.0634   |
| R1, WR2 [all data]         | 0.0398, 0.0667   |
| GOOF                       | 1.020            |
| Largest peak and hole (e·Å⁻³) | 1.18, -1.55     |

X-ray crystallography

Single-crystal X-ray diffraction data was obtained using Agilent SuperNova diffractometer equipped with an Atlas CCD detector (Agilent Technologies Inc, Santa Clara, CA, USA) at 145(1) K with graphite monochromated MoKα radiation (\( \lambda = 0.71073 \) Å). The structure was solved by direct methods using SHELXL-2014 (Sheldrick, 2015b), refining on \( F^2 \). Selected bonds and angles are given in Table 1. Detailed crystal data and structure refinement are listed in Table 4. Crystallographic data has been deposited with the Cambridge Crystallographic Centre as supplementary publication number CCDC-2052415.

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