Syntheses and Characterization of Two New Lead(II) Acetate Complexes, $Pb(L)(CH_3COO)_2$, L = 2,2':6',2''-Terpyridine (tpy) and 2,4,6-Tris(2-pyridyl)-1,3,5-Triazine (trz), Crystal Structure of $Pb(tpy)(CH_3COO)_2$

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Lead(II) complexes with 2,2':6',2"-terpyridine (tpy) and 2,4,6-tris(2-pyridyl)-1,3,5-triazine (trz) ligands, $[Pb(tpy)(CH_3COO)_2]$ and $[Pb(trz)(CH_3COO)_2]$, have been synthesized and characterized by IR, CHN elemental analysis and ^{207}Pb NMR. The structure of $Pb(tpy)(CH_3COO)_2$ was confirmed by single crystal X-ray data. The complex is monomeric and the Pb atom has an unsymmetrical seven–coordinate geometry, being coordinated by three nitrogen atoms of the 2,2':6',2"-terpyridine ligand and four oxygen atoms of the CH_3COO^- ligands. The arrangement of the ligands in the two complexes exhibits a coordination gap around the Pb(II) ion, occupied possibly by a stereoactive lone pair on lead(II). The coordination around lead atoms is hemidirected. The parallel aromatic rings in $Pb(tpy)(CH_3COO)_2$ show $\pi - \pi$ stacking.

Key words: 2,4,6-Tris(2-pyridyl)-1,3,5-triazine, 2,2':6',2"-Terpyridine, Lone Pair, Lead(II), $\pi - \pi$ Stacking

1. Introduction

The coordination chemistry of lead(II) with N and O-donor ligands has been investigated in the past decade and frequently discussed with respect to the coordination and stereoactivity of the valence shell lone pairs [1 – 4]. Extensive recent structural studies of lead(II) compounds [5 – 6] in particular have provided a basis for a rather detailed analysis of the evidence for coordination sphere distortions, which may be a consequence of the presence of such electron pairs. It appears that in complexes of lead(II) {and probably in those of related species such as Tl(I) [7 – 9] and Bi(III) [10 – 13]}, the nature and form of the coordination sphere are generally determined by a number of factors, including lone-pair bond-pair repulsions, so that seemingly minor differences in ligands can have quite marked effects on the coordination stereochemistry. Since the presence of a lone pair is not directly detected but inferred on the basis of the spatial distribution of donor atoms surrounding the metal, the identification of these donor atoms is fundamental to the analysis of any particular system.

The recent reports of the crystal structure of the 1:1 adducts $[Pb(phen)(O_2CCH_3)_2)]$ [5], $[Pb(phen)(O_2CCH_3)(O_2ClO_2)]$ [14], $[Pb(phen)(O_2CCH_3)(O_2NO)]$ [15], $[Pb(phen)(O_2CCH_3)(NCS)]$ [16] and the 1:2 adduct $[Pb(phen)_2(CH_3COO)]X$ (X = NCS^-, NO_3^- and ClO_4^-) [17] described the presence of bridging acetate and its influence on the coordination stereochemistry of the lead(II) ion. In the present paper, we describe the syntheses and characterization of lead(II) complexes with 2,2':6',2"-terpyridine (tpy) and 2,4,6-tris(2-pyridyl)-1,3,5-triazine (trz) ligands.

2. Experimental Section

2.1. Physical property measurements

IR spectra were recorded as nujol mulls using Perkin-Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus CHN-O- Rapid analyzer. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. The ^{207}Pb solution NMR spectra were recorded on a Bruker DRX-500 AVANCE spectrometer at 104.6 MHz using a 5 mm broadband probe. Lead chemical shifts are reported (ppm) downfield from tetramethyllead using $PbPh_4$ ($\delta_{Pb} = -178.0$ ppm, saturated in $CDCl_3$) as an external standard at a 0.01 M concentration and at 25 °C. Each lead spectrum was acquired in 3 – 10 hours.

2.2. Preparation of [Pb(tpy)(O_2CCH_3)$_2$]

Lead(II) acetate (0.36 g, 1 mmol) was dissolved in water by heating and the solution added dropwise with stirring to an aqueous solution of the 2,2':6',2''-terpyridine(tpy) (0.234 g, 1 mmol). The resulting solution was stirred for 3 h at r. t. On standing the product precipitated and was collected by filtration, washed with a little ice-cold water and recrystallized from a concentrated aqueous solution. The pure product was washed with ice-cold ethanol, then diethyl ether, before drying in air. Yield: 0.356 g, 60%. m. p. 135 °C. $C_{19}H_{17}N_3O_4Pb$: calcd. C 40.82, H 3.04, N 7.51; found C 40.62, H 3.01, N 7.81. – IR (cm^{-1}) selected bands were: 740(s), 1010(s), 1380(vs), 1538(vs), 1618(s), 2991(w), 3040(w). ^{207}Pb NMR (DMSO) $\delta = -816.68$ ppm.

2.3. Preparation of [Pb(trz)(O_2CCH_3)$_2$]

Lead(II) acetate (0.36 g,1 mmol) was dissolved in water by heating and the solution added dropwise with stirring to an aqueous solution of the 2,4,6-tris(2-pyridyl)-1,3,5-triazine (trz) ligand (0.31 g, 1 mmol). The resulting solution was stirred for 2 h at r.t. On standing, the product precipitated and was collected by filtration, washed with a little ice-cold water and recrystallised from a concentrated aqueous solution. The pure product was washed with ice-cold ethanol, then diethyl ether, before drying in air. Yield :0.328 g, 50% .m.p. 180 °C. $C_{25}H_{22}N_3O_4Pb$: calcd. C 47.16, H 3.46, N 6.60; found C 47.20, H 3.50, N 6.75. – IR (cm^{-1}) selected bands were: 740(s), 1010(s), 1360(vs), 1545 (vs), 1620(s), 2991(w), 3040(w). ^{207}Pb NMR (DMSO) $\delta = -886.2$ ppm.

2.4. Crystallography

2.4.1. Crystal data and refinement details

[Pb(tpy)(CH_3CO_2)$_2$]: $C_{19}H_{17}N_3O_4Pb$, M 558.55, monoclinic, space group $P2_1/c$, $a = 10.506(2)$, $b = 20.428(4)$, $c = 8.3879(16)$ Å, $\beta = 98.845(4)^\circ$, $V = 1778.7(6)$ Å^3, D_c $Z = 4$, $d_c = 2.086$ g/cm^3, $F(000) = 1064$, crystal size: $0.40 \times 0.2 \times 0.07$ mm; $T_{max,min} = 0.265, 0.109$, N = 13871, $N_0 = 3154$, $R = 0.0513$, $R_w = 0.1185$.

2.4.2. Determination of the structure

Crystallographic measurements were made at 120(2) K using a Siemens R3m/V diffractometer. The data were collected within the range of $1.96^\circ \leq \theta \leq 30.03^\circ$ for [Pb(tpy)(CH_3CO_2)$_2$] using graphite monochromated Mo-K$_\alpha$ radiation ($\lambda = 0.71073$ Å). Accurate unit cell parameters and the orientation matrix for data collection was obtained from a least-squares refinement. Intensities of 13871 unique reflections were measured, of which 3154 with $I > 2\sigma(I)$ were used in the refinement for [Pb(tpy)(CH_3CO_2)$_2$]. The structure has been solved by direct methods and refined by full-matrix least-squares refinements on F^2. The positions of hydrogen atoms were included in idealized positions in the calculations of the structure factors as fixed contributions. Each hydrogen atom was assigned an isotopic displacement parameter. R, R_w, with goodness of fit on F^2 of 0.939 are 0.0513 and 0.1185. The final difference density map showed a maximum peak and hole of 3.856 and -2.718 eÅ^{-3}. Corrections for Lorentz and polarization effects as well as an empirical correction for absorption using the Sadabs program were applied. All structural calculations were carried out with a PDP – 11/23+ computer using the SDP – PLUS program package [18, 19].

Table 1. Crystal data and structure refinement of [Pb(tpy)(CH_3COO)$_2$].

Empirical formula	C_{19} H_{17} N_3 O_4 Pb
Formula weight	558.55
Temperature	120(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Unit cell dimensions	$a = 10.506(2)$ Å $b = 20.428(4)$ Å $c = 8.3879(4)$ Å $\beta = 98.845(4)^\circ$
Volume	1778.7(6) Å^3
Z	4
Density (calculated)	2.086 g/cm^3
Absorption coefficient	9.516 mm^{-1}
$F(000)$	1064
Crystal size	$0.40 \times 0.20 \times 0.07$ mm^3
Theta range for data collection	1.96 to 30.03°
Index ranges	$-14 \leq h \leq 14$, $-32 \leq k \leq 28$ $-11 \leq l \leq 11$
Reflections collected	13871
Independent reflections	5095 [R(int) = 0.0512]
Completeness to θ	97.8%
Absorption correction	semi-empirical
Max. and min. transmission	0.285 and 0.109
Refinement method	full-matrix least-squares on F^2
Data / restraints / parameters	5065/0/244
Goodness-of-fit on F^2	0.939
Final R indices [$I > 2\sigma(I)$]	$Rl = 0.0513$, $wR2 = 0.1185$
R Indices (all data)	$R1 = 0.0863$, $wR2 = 0.1306$
Largest diff. Peak and hole	3.856, -2.718 e Å^{-3}

Crystal data and structure refinement data are given in Table 1. Selected bond lengths and angles are given in Table 2. Anisotropic displacement parameters, observed and calculated structure factors, full lists of bond distances, bond angles and torsion angles are given in the supplementary material*. ORTEP diagram and a perspective view of the packing of the molecule in the unit cell are shown in Figures 1 and 2.

*Complete bond lengths and angles, co-ordinates and displacement parameters have been deposited at Cambridge Crystallography Data Centre. Supplementary data are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition number 185735.

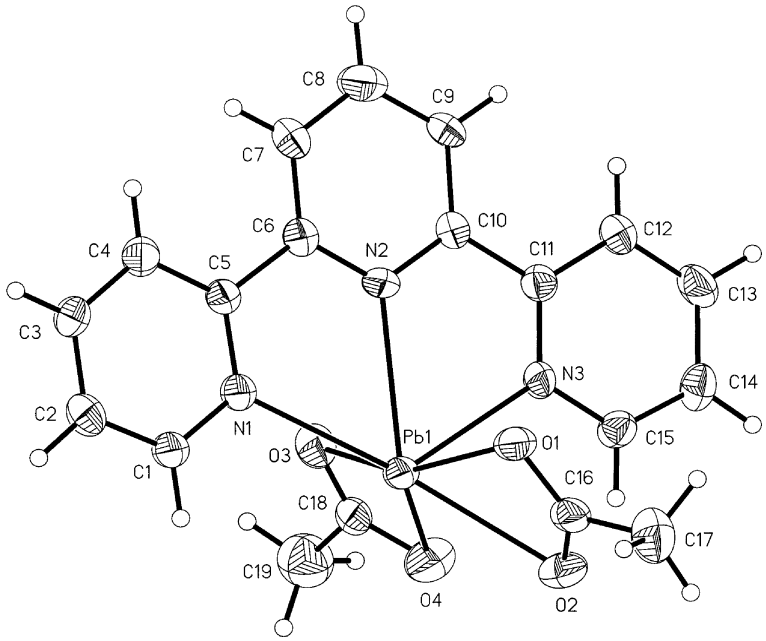

Fig. 1. The ORTEP diagram of the $Pb(tpy)(CH_3COO)_2$.

Table 2. Selected bond lengths (Å) and angles (°) for the $[Pb(tpy)(CH_3CO)_2]$.

Pb-O(1)	2.565(6)	Pb-O(2)	2.676(6)
Pb-O(3)	2.609(6)	Pb-O(4)	2.687(7)
Pb-N(1)	2.727(5)	Pb-N(2)	2.642(5)
Pb-N(3)	2.473(6)		
N(3)-Pb-O(1)	78.7(2)	N(3)-Pb-O(3)	83.1(2)
O(1)-Pb-O(3)	159.65(18)	N(3)-Pb-N(2)	64.41(19)
O(1)-Pb-N(2)	83.73(17)	O(3)-Pb-N(2)	80.27(19)
N(3)-Pb-O(2)	77.37(18)	O(1)-Pb-O(2)	49.74(16)
N(2)-Pb-O(2)	124.97(18)	O(3)-Pb-O(2)	134.10(18)
O(1)-Pb-O(4)	132.98(19)	N(3)-Pb-O(4)	75.9(2)
O(3)-Pb-O(4)	48.60(19)	N(2)-Pb-O(4)	118.24(19)
O(2)-Pb-O(4)	86.21(19)	N(1)-Pb-N(3)	124.73(19)
O(1)-Pb-N(1)	92.67(18)	O(3)-Pb-N(1)	90.28(19)
N(2)-Pb-N(1)	60.40(18)	O(2)-Pb-N(1)	134.75(19)

3. Results and Discussion

3.1. Synthesis

Reaction between $Pb(CH_3CO_2)_2$ with 2,2':6',2"-terpyridine (tpy) and 2,4,6-tris(2-pyridyl)-1,3,5-triazine (trz) provided powder materials which analyzed as $[Pb(tpy)(CH_3CO_2)_2]$ and $[Pb(trz)(CH_3COO)_2]$ complexes. The IR spectra of the $[Pb(tpy)(CH_3COO)_2]$ and $[Pb(trz)(CH_3COO)_2]$ complexes show ν(COO) bonds at about 1380, 1538 and 1360, 1545 cm^{-1}. Attempts to grow single crystals of $[Pb(trz)(CH_3COO)_2]$ suitable for X-ray crystallography were not successful.

3.2. Crystal structure of [Pb(tpy)(CH$_3$COO)$_2$]

The crystal structure of this compound consists of monomeric units of $[Pb(tpy)(CH_3COO)_2]$. Each lead atom is chelated by the nitrogen atoms of the "tpy" ligand with Pb–N distances of 2.473, 2.642 and 2.727 Å, and the oxygen atoms of the acetate anions with Pb–O distances of 2.565, 2.609, 2.676 and 2.678 Å (Table 2). The coordination number in this complex is seven (three from the 'tpy' ligand, four from the acetate anions). A weak interaction of lead(II) with the oxygen atoms of an adjacent molecule produces a polymer in the solid state. Each Pb atom in this structure forms one "weak" Pb···O bond. The presence of a lone pair at the lead atom is apparently the reason for the large space not occupied by ligand atoms and the inability of the complex to adopt higher symmetry.

The arrangement of the 2,2':6',2"-terpyridine (tpy) ligand and the acetate anions suggests a gap in the coordination around the metal ion [the O(2)-Pb-N(1) angle is 134.75° and the O(4)-Pb-N(1) angle is 134.26°], possibly due to the stereoactive lone pair. The shortening of the Pb-N bonds on the side of the Pb(II) ion opposite to the lone pair (2.473 and 2.565 Å compared with 2.727 and 2.687 Å adjacent to the lone pair) supports the presence of this feature [20]. Hence, the geometry of the nearest coordination environment of every lead atom is likely caused by the geometri-

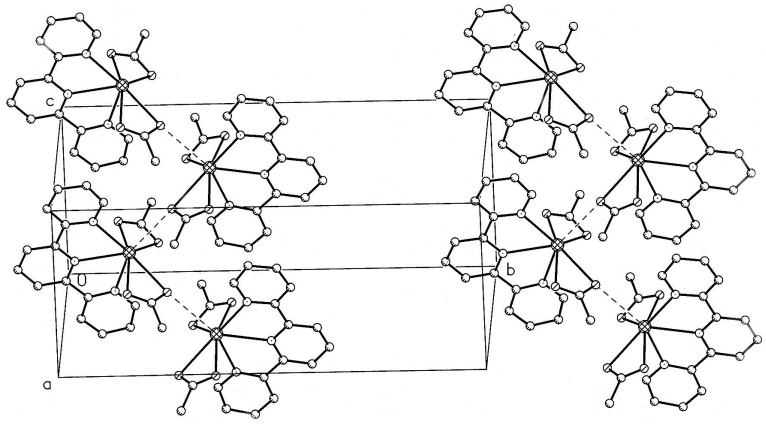

Fig. 2. The unit cell of $Pb(tpy)(CH_3COO)_2$.

cal constraints of the "tpy" ligand and the acetate anions and by the influence of a stereochemically active lone pair. Such an environment leaves space for bonding to oxygen atoms of the acetate anion of an adjacent molecule (Fig. 2). These intermolecular interactions of each unit bonded to two neighbors assembles the molecules into an one-dimensional chain parallel to the *a* axis.

The possible stereochemical activity of the lone pair in divalent lead compounds has recently been discussed by Shimoni-Livny *et al.* based on a thorough review of crystal data available from the Cambridge Structural Database (CSD) [6]. They classify lead coordination as holodirected, which refers to complexes in which the bonds to the ligand atoms are directed throughout the surface of the encompassing sphere, and as hemidirected in those cases in which the bonds to the ligand atoms occupy only a part of the coordination sphere, leaving a gap in the metal atoms coordination sphere. The latter, present in all Pb(II) compounds with coordination numbers 2 to 5 or 6, 7 and 8, does not exist in lead complexes with higher coordination numbers, here the holodirected geometry is the rule. For the structure described here, coordination around the lead atoms is hemidirected with a significant gap trans to the chelating "tpy" ligand.

There is a $\pi - \pi$ stacking [21–22] interaction between the parallel aromatic rings belonging to adjacent chains as shown in Fig. 2. The pyridyls are almost parallel and separated by a distance of about 3.5 Å, close to that of the layers in graphite. Parallel arrays of the planes of the aromatic moieties indicate that these interactions are of the "π-stacking" type, rather than "edge-to-face" or "vertex-to-face" types [23–26]. Projection of the structure perpendicular to the ring plane shows the overall form of "slipped" stacking [26–27], which can be rationalized qualitatively in terms of optimizing the attraction between atoms of opposite charges [28].

A simple model to describe the nature of $\pi - \pi$ interactions has been developed by Hunter *et al.* [28]. This model predicts that face-to-face π-stacked interactions will be disfavored due to the dominance of $\pi - \pi$ repulsion. However, in offset π-stacked and edge-on or T-shaped geometries, favourable $\pi - \sigma$ attractions dominate. The polarization of aromatic systems through the introduction of heteroatoms, electron-withdrawing groups or electron-donating groups, alters the nature of any $\pi - \pi$ interactions. For example, although it is well known that neither benzene [29] nor hexafluorobenzene tend to form stacked arrangements, the adduct between these two molecules adopts a structure comprising inclined stacks of alternating molecules [30]. Here, the hexafluorobenzene π-system is electron-deficient with respect to benzene and this reduces the $\pi - \pi$ repulsion, making the formation of offset π-stacked molecules more favorable. The introduction of heteroatoms can lead to a similar perturbation of aromatic interactions [25]. Specifically nitrogen atoms have been shown to remove electron density from the π-system, and hence have a similar effect as electron-withdrawing groups. It has been demonstrated that electron-deficient aromatic groups interact most strongly with electron-rich aromatic groups. Hence, since the molecules of this packing includes equally or almost equally electron-deficient, or indeed electron-rich, rings, it can be expected that within the molecule discussed here face-to-face π-stacking inter-

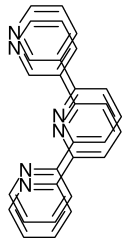

Fig. 3. Projection of nearest neighbor pairs in the $\pi-\pi$ stacks of the heteroaromatic bases in [Pb(tpy)$(CH_3COO)_2$].

actions must be disfavored due to the dominance of $\pi-\pi$ repulsion.

Thus two factors, lone pair activity and $\pi-\pi$ stacking, may control the coordination sphere of this complex. The obvious question then is, whether the lone pair activity has stretched coordinating bonds to result in ligand stacking or whether it is the stacking interaction, which imposes gap formation in the coordination sphere. The model of face-to-face π-stacking interactions must be disfavored due to the dominance of $\pi-\pi$ repulsion. This possibly suggests that the lone pair activity is the most important factor and affects the formation of $\pi-\pi$ stacking.

A comparison of the coordination type of the acetate anions in this complex with that in reported analogous lead(II) complexes may illustrate the influence of the kind of the ligand on the structural geometry.

It is interesting that there is a correlation between strong ligands presence in the coordination sphere of lead(II) complexes and the ability of acetate anion for formation of bridges. The complexes [Pb(phen)$(O_2CCH_3)(O_2ClO_2)$] [14] and [(phen)Pb$(O_2CCH_3)(O_2NO)$] [15] are polymeric, while [Pb(phen)$(O_2CCH_3)_2$] [5] and [(phen)Pb(O_2CCH_3)(NCS)][16] are dimeric and [Pb(phen)$_2$ $(CH_3COO)](ClO_4)$ [17] is monomeric. The complex reported here is monomeric with weak interactions of the oxygen atoms of the acetate ligands with neighboring lead atoms.

The Pb_2O_2 rings in [Pb(tpy)$(CH_3COO)_2$] are similar to the central Pb_2O_2 rings of dimeric [Pb(phen)$(O_2CCH_3)_2$] [5], [(phen)Pb(O_2CCH_3)(NCS)] [16], polymeric [Pb(phen)$(O_2CCH_3)(O_2ClO_2)$] [14] and [(phen)Pb$(O_2CCH_3)(O_2NO)$] [15], though the long edges of the rings in [Pb(phen)$(O_2CCH_3)(O_2NO)]_n$ (2.804 Å) and [Pb(phen)$(O_2CCH_3)(O_2ClO_2)]_n$ (2.736 Å), are considerably shorter than in [(phen)Pb(O_2CCH_3)(NCS)]$_2$ (3.190 Å) and [Pb(phen)$(O_2CCH_3)_2]_2$ (3.366 Å). This is consistent with the assumption that the acetate and thiocyanate anions are stronger ligands than nitrate and perchlorate, ($CH_3COO^- > NCS^- > NO_3^- > ClO_4^-$). It is interesting, that when stronger ligands like CH_3COO^- and NCS^- are coordinated to lead(II) like in [(phen)Pb(O_2CCH_3)X] (X= CH_3COO^- and NCS^-), the structures are dimeric and the acetate ligands bridge only one side, whereas when weaker ligands like NO_3^- and ClO_4^- are coordinated to the lead(II) ion like in [(phen)Pb(O_2CCH_3)X] (X= NO_3^- and ClO_4^-), the structures are polymeric and the acetate ligands bridge two sides. In the [Pb(phen)$_2(O_2CCH_3)](ClO_4)$ complex, the acetate ligand is not bridging and the complex is monomeric. This point is consistent with the assumption that 1,10-phenanthroline is a strong ligand. The [Pb(tpy)$(CH_3COO)_2$] complex is monomeric and the acetate ligand is weakly interacting with the neighboring lead atom from one side. This shows that one tri-dentate ligand such as "tpy" in [Pb(tpy)$(CH_3COO)_2$] affects the acetate ligand more than one bidentate ligand such as "phen" in dimeric [Pb(phen)$(CH_3COO)_2$] and two bidentate ligands such as "phen" in monomeric [Pb(phen)$_2$ $(CH_3COO)](ClO_4)$.

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