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1D & 2D Supramolecular assemblies dominated by crystal structure of Pb(II) oxoanion (and) complexes with 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine (PDPT)

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1D & 2D Supramolecular assemblies dominated by crystal structure of Pb(II) oxoanion (NO_3^- and ClO_4^-) complexes with 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine (PDPT)

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The two lead(II) complexes, $[\text{Pb}(\text{PDPT})(\text{NO}_3)_2]_n$ and $[\text{Pb}(\text{PDPT})_2(\text{ClO}_4)_2\text{EtOH}] \cdot \text{CH}_3\text{OH}$, PDPT = 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine, have been synthesized and characterized. 1D & 2D supramolecular assemblies of these compounds in the solid state are discussed via covalent and noncovalent donor...acceptor interactions.

Keywords: Lead; 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine; 1D & 2D polymers

1. Introduction

Numerous compounds containing 1,2,4-triazine moieties are well known in natural materials and show interesting biological, pharmacological and medicinal properties. One of the important classes of N-containing heterocycles is the 3,5,6-trisubstituted-1,2,4-triazines. Some of them can be active as blood platelet aggregation inhibitors and others exhibit antiviral inhibitory activity (against influenza viruses for example), significant activity towards leukaemia and ovarian cancer, and anti-HIV activity [1].

A wide range of 1,2,4-triazine complexes has been reported in the literature [2]. Among them, only a few papers have considered the potential biological or pharmaceutical activity of such complexes and more specifically of copper complexes [3, 4].

It is interesting to find further examples of these complexes with Pb^{2+} . The lead(II) complexes are interesting and frequently discussed when considering the coordination and stereo-activity of heavy metals, that is the “stereo-chemical activity” of the valence shell electron lone pairs [5–10]. Extensive recent structural studies on lead(II)

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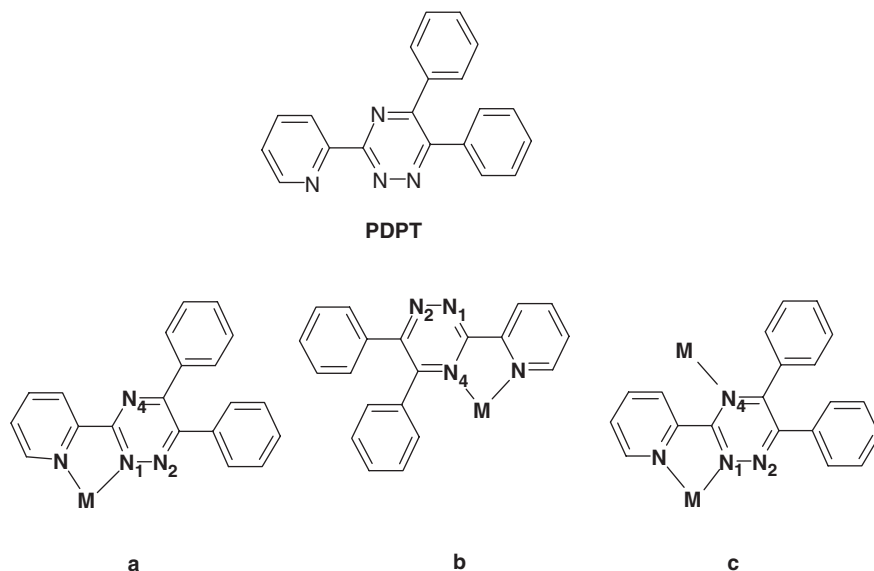


Figure 1. The general structure of the PDPT ligand and its modes of coordination.

compounds [11] have in particular provided a basis for a rather detailed analysis for the evidence of coordination sphere distortions as a consequence of the presence of such lone-pairs.

This work focuses on the nature of the adducts formed between lead(II) salts (perchlorate and nitrate) and 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine (PDPT) and on the role of the counter ion in the stoichiometry of the resulting complexes.

The general structure of the ligand (PDPT) and its multifunction coordination modes are shown as in figure 1. The presence of a triazine nitrogen adjacent to the pyridyl group helps PDPT to bind in a bidentate fashion forming one five-membered chelate ring either through N1-bonding figure 1(a), or through N4-bonding figure 1(b). PDPT has two other sites for coordinating, and may produce polymeric structures, figure 1(c).

2. Experimental

2.1. Physical 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.

2.2. Preparation of $[Pb(PDPT)(NO_3)_2]_n$ (1)

3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine (0.331 g, 1 mmol) was placed in one arm of a branched tube and lead(II) nitrate(0.331 g; 1 mmol) in the other. Methanol was carefully added to fill both arms, the tube sealed and the ligand-containing arm immersed in a bath at 60°C while the other was at ambient temperature.

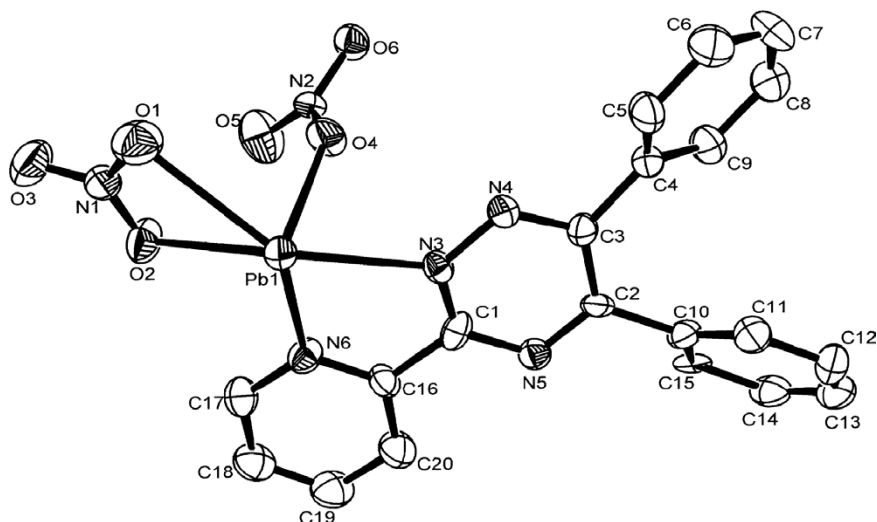


Figure 2. ORTEP diagram of $[\text{Pb}(\text{PDPT})(\text{NO}_3)_2]_n$ (**1**).

After 3 days, yellow crystals deposited in the cooler arm and were filtered off, washed with acetone and ether, and air dried (0.288 g, yield 60%), m.p. = 215°C. (Found: C: 37.50, H: 2.00, N: 13.20%, calculated for $\text{C}_{20}\text{H}_{14}\text{N}_6\text{O}_6\text{Pb}$: C: 37.44, H: 2.18, N: 13.10).

IR (cm^{-1}) selected bands: 694(m), 763(m), 1010(m), 1265(s), 1380(vs), 1442(s), 1511(s), 1596(m), 1735(w), 1765(w), and 3062(w).

$^1\text{H NMR}$ (DMSO, d): 7.65(m, 10H), 7.75(t, 1H), 8.20(t, 1H), 8.60(d, 1H), and 8.85(d, 1H).

2.3. Preparation of $[\text{Pb}(\text{PDPT})_2(\text{ClO}_4)_2\text{EtOH}] \cdot \text{CH}_3\text{OH}$ (**2**)

PDPT (0.662 g, 2mmol) was placed in one arm of a branched tube and lead(II) nitrate (0.331 g, 1 mmol) and sodium perchlorate (0.246 g, 2 mmol) in the other. Methanol and ethanol (ratio of 1/1) was carefully added to fill both arms, the tube sealed and the ligand-containing arm immersed in a bath at 60°C while the other was at ambient temperature. After 1 day, red crystals deposited in the cooler arm and were filtered off, washed with acetone and ether, and air dried (0.288 g, yield 60%), m.p. = 176°C. (Found C, 46.64; H, 3.12; N, 10.00%, calculated for $\text{C}_{43}\text{H}_{37}\text{Cl}_2\text{N}_8\text{O}_{10}\text{Pb}$: C: 46.74, H: 3.35, N: 10.14).

IR (cm^{-1}) selected bands: 610(m), 625(m), 702(m), 763(s), 1103(vs), 1396(s), 1511(m), 1596(s), 2969(w), and 3062(w).

$^1\text{H NMR}$ (DMSO, d): 1.04 (t, 3H), 3.42(m, 2H), 3.70(d, 3H), 4.32(t, 1H), 4.60(q, 1H), 7.45(m, 10H), 7.60(m, 10H), 7.65(t, 2H), 8.08(t, 2H), 8.55(d, 2H), and 8.85(d, 2H).

3. Results and discussion

Reaction between PDPT and lead(II) nitrate/perchlorate provided crystalline materials $[\text{Pb}(\text{PDPT})(\text{NO}_3)_2]_n$ and $[\text{Pb}(\text{PDPT})_2(\text{ClO}_4)_2\text{EtOH}] \cdot \text{CH}_3\text{OH}$, respectively. The characteristic IR active band for $\nu(\text{ClO}_4^-)$ of the complex appeared at 615, 625, 1103 cm^{-1} .

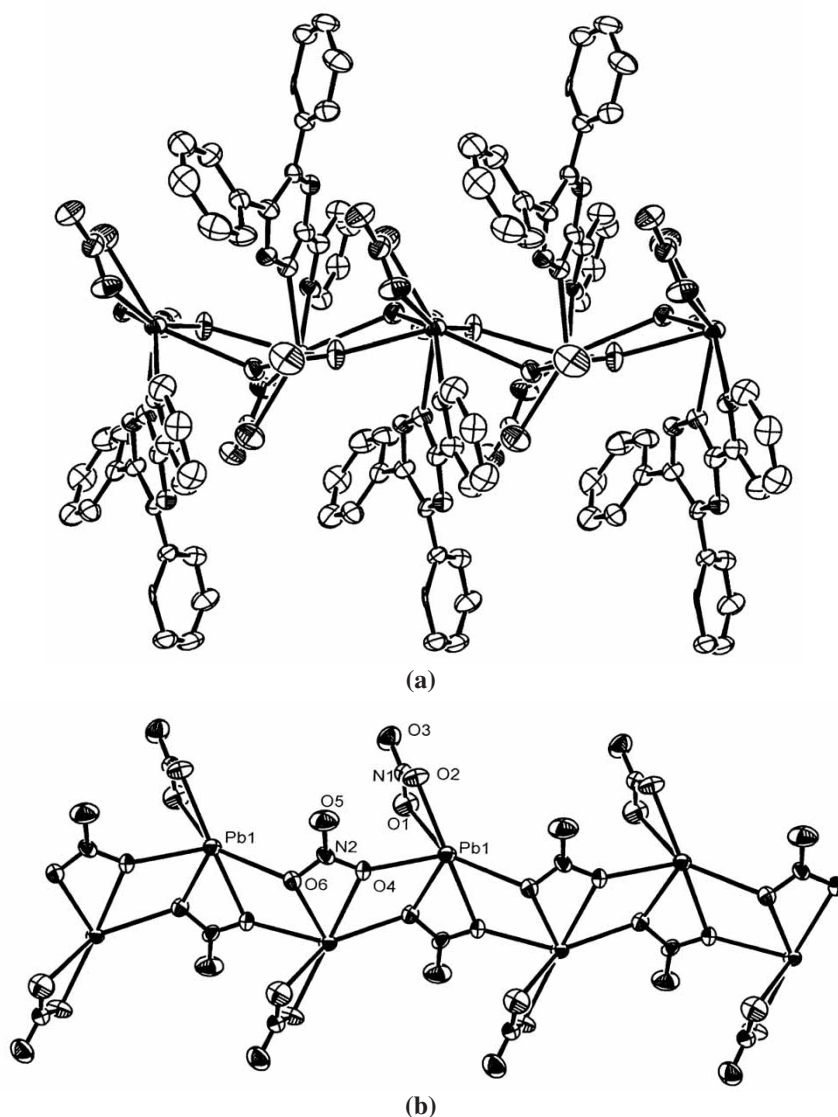


Figure 3. (a) A fragment of 1D coordination polymer in $[\text{Pb}(\text{PDPT})(\text{NO}_3)_2]_n$ (**1**), (b) A view of the nitrate coordination mode in the $[\text{Pb}(\text{PDPT})(\text{NO}_3)_2]_n$ (**1**), and (c) A view of the $\text{Pb}\cdots\text{N4}$ interaction in the $[\text{Pb}(\text{PDPT})(\text{NO}_3)_2]_n$ (**1**), the PDPT ligands are omitted for clarity in the figure (b) and figure (c).

In general, IR absorption bands in the region $1300\text{--}1500\text{ cm}^{-1}$ are due to the presence of NO_3^- stretching vibrations [12]. The medium intensity bands that appeared at 1380, 1396, 1511, 1596 cm^{-1} were assigned to C=C, C=N stretching mode. The bond of $\nu(\text{C-H aromatic})$ appeared at 3062 cm^{-1} in accordance with the literature [13].

In numerous instances, a change in stoichiometry from PbX_2L to PbX_2L_2 , where X is an anion and L a bidentate ligand such as 2,2'-bipyridine or 1,10-phenanthroline, is associated with a change from a lattice composed of oligomeric or polymeric entities to a lattice composed of molecular species [14].

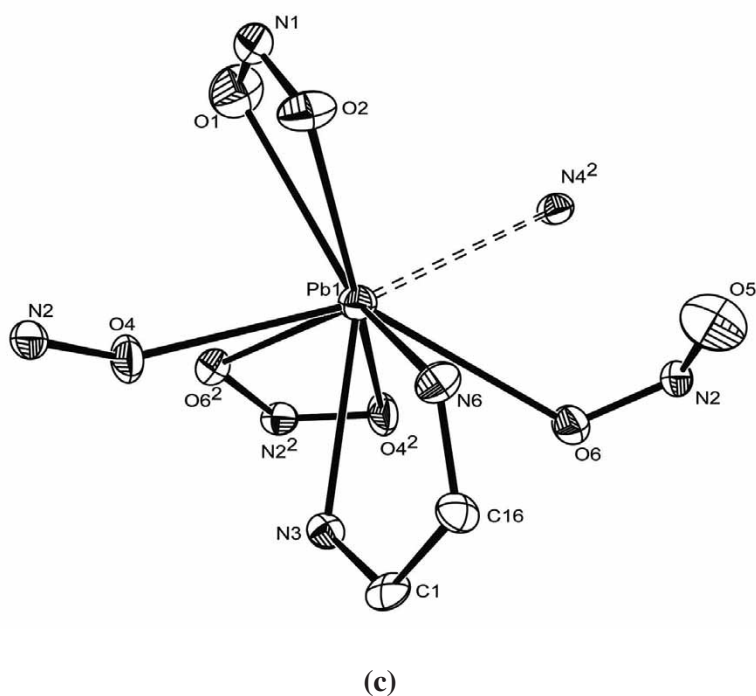
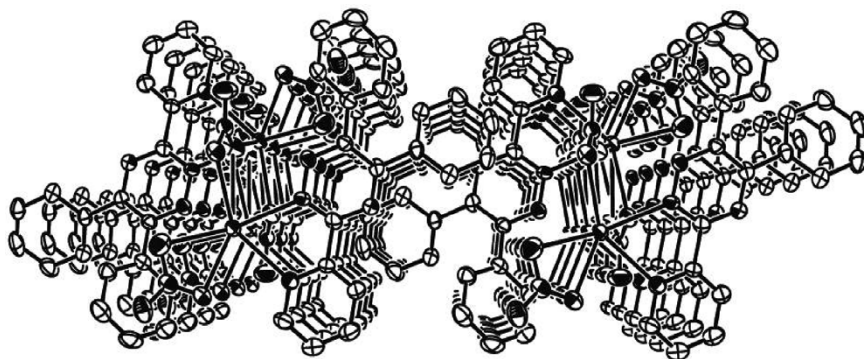


Figure 3. Continued.

Figure 4. The packing of the $[\text{Pb}(\text{PDPT})(\text{NO}_3)_2]_n$ (**1**).

This again appears to be the case where X is an anion such as nitrate or perchlorate and L is PDPT, and the nature of lattice of the complexes $[\text{Pb}(\text{PDPT})(\text{NO}_3)_2]_n$ and $[\text{Pb}(\text{PDPT})_2(\text{ClO}_4)_2\text{EtOH}] \cdot \text{CH}_3\text{OH}$ is a focus of the present discussion.

The ORTEP diagram of **1** is shown in figure 2. The crystal structure of **1** consists of polymeric units of $\text{Pb}(\text{PDPT})(\text{NO}_3)_2$ (figure 3). If a limit of 2.85 Å is placed upon Pb(II)-donor atom separation for coordinate bonding, each lead atom is chelated by the nitrogens of PDPT with Pb–N distances of 2.573 and 2.566 Å and bonded to

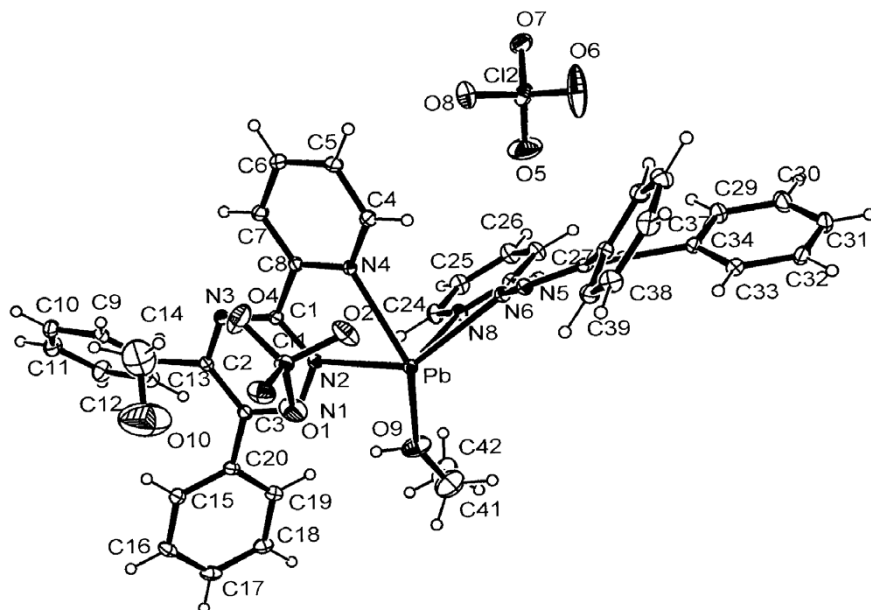


Figure 5. ORTEP diagram of $[\text{Pb}(\text{PDPT})_2(\text{ClO}_4)_2\text{EtOH}] \cdot \text{CH}_3\text{OH}$ (2).

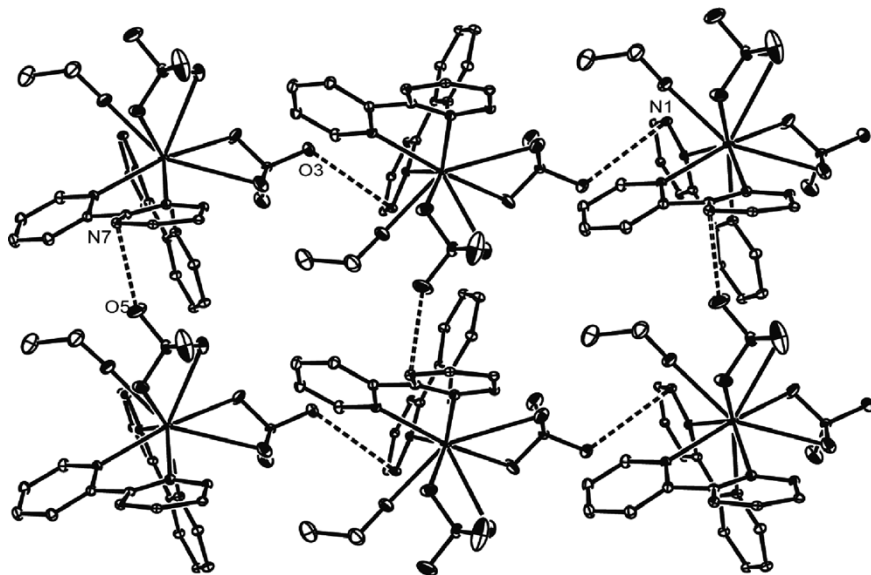


Figure 6. Packing of $[\text{Pb}(\text{PDT})_2(\text{ClO}_4)_2\text{EtOH}]_n \cdot \text{MeOH}$ (2) complex, Down b, dashed bond are O3–N1 and O5–N7 donor···acceptor interactions and phenyl groups was omitted for clarity.

the oxygen atoms of four nitrates with Pb–O distances of 2.554, 2.646, 2.704, 2.733, 2.788 and 2.845 Å. Indeed, each lead atom is coordinated to the nitrogen atom of the pyridyl ring and one nitrogen atom of the triazine ring adopting the coordination mode shown in figure 1(a). Therefore, Pb atoms are eight-coordinate

Table 1. Crystal data and structure refinement for [Pb(PDPT)₂EtOH](ClO₄)₂·CH₃OH and [Pb(PDPT)(NO₃)₂]_n.

Identification code	[Pb(PDPT) ₂ EtOH](ClO ₄) ₂ ·CH ₃ OH	[Pb(PDPT)(NO ₃) ₂] _n
Empirical formula	C ₄₃ H ₃₇ Cl ₂ N ₈ O ₁₀ Pb	C ₂₀ H ₁₄ N ₆ O ₆ Pb
Formula weight	1103.90	641.56
Temperature (K)	120(2)	293(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	<i>P</i> 2(1)/ <i>c</i>	<i>P</i> 21
Unit cell dimensions (Å, °)	<i>a</i> = 30.297(4) <i>b</i> = 8.3331(11) <i>c</i> = 17.113(2) α = 90 β = 93.059(2) γ = 90	<i>a</i> = 11.714(2) <i>b</i> = 7.1380(14) <i>c</i> = 12.393(3) α = 90 β = 103.62(3) γ = 90
Volume (Å ³)	4314.3(10)	1007.1(3)
<i>Z</i>	4	2
Density (calculated) (Mg m ⁻³)	1.700	2.116
Absorption coefficient (mm ⁻¹)	4.103	8.430
<i>F</i> (000)	2188	612
Crystal size (mm ³)	0.22 × 0.18 × 0.14	0.3 × 0.3 × 0.1
Theta range for data collection (°)	2.02 to 27.89	3.32 to 25.99
Index ranges	-37 ≤ <i>h</i> ≤ 39, -10 ≤ <i>k</i> ≤ 10, -22 ≤ <i>l</i> ≤ 22	0 ≤ <i>h</i> ≤ 14, 0 ≤ <i>k</i> ≤ 8, -15 ≤ <i>l</i> ≤ 14
Reflections collected	37959	2176
Independent reflections	9407 [<i>R</i> (int) = 0.0180]	2072 [<i>R</i> (int) = 0.0639]
Completeness to theta	97.6%	96.1%
Absorption correction	Semi-empirical from equivalents	Psi-scan
Max. and min. transmission	0.5974 and 0.4655	0.704 and 0.126
Refinement method	Full-matrix least-squares on <i>F</i> ²	Full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	9407/0/579	2072/1/295
Goodness-of-fit on <i>F</i> ²	1.038	1.050
Final <i>R</i> indicate [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ = 0.0273, <i>wR</i> ₂ = 0.0744	<i>R</i> ₁ = 0.0347, <i>wR</i> ₂ = 0.0887 [for 3740 reflections. <i>I</i> > 2σ(<i>I</i>)]
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0285, <i>wR</i> ₂ = 0.0752	<i>R</i> ₁ = 0.0368, <i>wR</i> ₂ = 0.0906
Largest diff. Peak, hole (e Å ⁻³)	1.358, -0.764	1.988, -1.489

in “hemidirected” array [11], consistent with the presence of a lone pair in the apparent coordination-sphere vacancy remote from the bound atoms (figures 2 and 3a).

In the present complex only one of nitrate acts as a truly bidentate ligand and another is bidentate and bridging between three lead centers rather than a chelate to one. It is interesting, nonetheless, to view links of three Pb atoms *via* two Pb₂O₂ rhombs, geometrically a feature of many Pb(II) complex structures [15, 16], involving these three nitrate-O atoms (a novel behavior of nitrate anion) (figure 3b).

To find all potential donor centers, it is necessary to extend the bonding limits to values above 3.5 Å [17]; PDPT-N4 atoms approach each Pb with a distance of Pb ··· N4 = 3.474 Å, thus giving to each metal a very asymmetric environment of nine possible donor centers (figure 3c). There are two sets of phenyls in PDPT; one set close to Pb with planes at 7.138 Å indicating no interaction, and another set lies between triazine rings and at 3.451 and 3.710 Å. Therefore, there is edge-to-edge stacking between parallel aromatic rings of adjacent chains [18] (figure 4).

The crystal structure of **2** consists of monomeric units (figure 5), similar to complex 1. If a limit of 2.85 Å is placed upon Pb(II)-donor atom separation for bonding, each lead atom is chelated by the nitrogen atoms of PDPT with Pb–N distances of 2.481, 2.581, 2.617 and 2.599 Å and bonded to the oxygen atom of ethanol ligand with

Table 2. Bond lengths (Å) and bond angles (°) for [Pb(PDPT)(NO₃)₂]_n.

Pb1–O2	2.553(7)	O2–Pb1–N6	76.5(2)
Pb1–N6	2.565(7)	O2–Pb1–N3	127.1(2)
Pb1–N3	2.573(7)	N6–Pb1–N3	63.7(2)
Pb1–O4	2.646(9)	O2–Pb1–O4	78.5(3)
Pb1–O6#1	2.704(9)	N6–Pb1–O4	86.5(3)
Pb1–O1	2.733(9)	N3–Pb1–O4	66.2(2)
Pb1–O6#2	2.788(9)	O2–Pb1–O6#1	121.7(3)
Pb1–O4#1	2.844(10)	N6–Pb1–O6#1	139.3(3)
N1–O3	1.214(12)	N3–Pb1–O6#1	77.8(2)
N1–O2	1.235(11)	O4–Pb1–O6#1	65.1(3)
N1–O1	1.262(13)	O2–Pb1–O1	47.8(3)
N2–O5	1.185(11)	N6–Pb1–O1	124.0(3)
N2–O6	1.275(13)	N3–Pb1–O1	143.2(3)
N2–O4	1.277(14)	O4–Pb1–O1	77.9(3)
O4–Pb1#3	2.844(10)	O6#1–Pb1–O1	79.9(3)
O6–Pb1#3	2.704(9)	O2–Pb1–O6#2	131.3(3)
O6–Pb1#4	2.788(9)	N6–Pb1–O6#2	74.5(3)
		N3–Pb1–O6#2	70.5(2)
		O4–Pb1–O6#2	136.7(3)
		O6#1–Pb1–O6#2	105.75(18)
		O1–Pb1–O6#2	144.6(3)
		O2–Pb1–O4#1	156.1(3)
		N6–Pb1–O4#1	126.9(3)
		N3–Pb1–O4#1	74.2(2)
		O4–Pb1–O4#1	105.2(2)
		O6#1–Pb1–O4#1	45.7(2)
		O1–Pb1–O4#1	109.1(3)
		O6#2–Pb1–O4#1	61.5(2)

Symmetry transformation used to generate equivalent atoms: (#1) $-x+1, y+1/2, -z+1$. (#2) $x, y+1, z$. (#3) $-x+1, y-1/2, -z+1$. (#4) $x, y-1, z$.

Pb–O distance of 2.615 Å. Therefore Pb atom is five-coordinate in a very strongly “hemidirected” [11] array (figure 5). To find any potential donor center, extending the bonding limit to <3.5 Å shows two perchlorate anions approaching each Pb, with Pb–O distances of 3.046, 3.100, 3.211, 2.860 Å, thus giving each metal an asymmetric environment of nine donors.

Sterically bulky groups present on PDPT ligand force formation of monomeric complex and the lone pair on lead, is apparently the reason why perchlorate anions lie far from lead. Lead–oxygen separations similar to the present case have been observed in several other perchlorate complexes [19]. The lattice of complex **2** shows distances of O5–N7 = 2.996 Å and O3–N1 = 3.285 Å, with significant distortion of the aromatic triazine ring, possibly indicative of donor...acceptor interactions into a supramolecular 2D network [20] (figure 6).

Attempts to isolate 1:2 adducts of Pb(PDPT)₂(NO₃)₂ and 1:1 adducts of Pb(PDPT)(ClO₄)₂, were not successful; each time the 1:1 adduct of **1**, and the 1:2 adduct **2** was isolated. This is consistent with the assumption that nitrate anion is stronger ligand than perchlorate anion (NO₃⁻ > ClO₄⁻), resulting in the nitrate anion coordinate to lead(II) atom preventing formation of 1:2 adducts with these anions.

Determination of the structures of the [Pb(PDPT)(NO₃)₂]_n (**1**) and [Pb(PDPT)₂(ClO₄)₂EtOH]·CH₃OH (**2**), by X-ray crystallography showed that the complexes in the solid state are polymeric (tables 1–3). The [Pb(PDPT)(NO₃)₂]_n is a 1D coordination polymer, the monomeric units are linked by covalent O_{nitrate}–Pb interactions and [Pb(PDPT)₂(ClO₄)₂EtOH]·CH₃OH is a 2D coordination polymer,

Table 3. Bond lengths (Å) and bond angles (°) for [Pb(PDPT)₂(ClO₄)₂EtOH]·CH₃OH₂.

Pb–N6	2.617(3)	N8–Pb–N4	77.76(9)
Pb–N8	2.481(3)	N8–Pb–N2	83.79(9)
Pb–N2	2.599(3)	N4–Pb–N2	62.74(8)
Pb–N4	2.581(3)	N8–Pb–O9	78.22(10)
Pb–O9	2.615(3)	N4–Pb–O9	128.87(9)
Cl1–O1	1.429(3)	N2–Pb–O9	70.31(10)
Cl1–O3	1.428(3)	N8–Pb–N6	64.74(9)
Cl1–O2	1.436(3)	N4–Pb–N6	81.73(8)
Cl1–O4	1.425(3)	N2–Pb–N6	137.04(8)
Cl2–O6	1.394(3)	O9–Pb–N6	125.48(10)
Cl2–O5	1.422(4)	N8–Pb–O7	78.91(8)
Cl2–O8	1.434(3)	N8–Pb–O1	151.63(9)
Cl2–O7	1.448(3)	N8–Pb–O2	136.04(8)
Pb–O1	3.046(4)	N4–Pb–O7	145.12(8)
Pb–O2	3.100(3)	N4–Pb–O1	81.19(9)
Pb–O7	2.860(3)	N4–Pb–O2	65.12(9)
Pb–O8	3.211(3)	N2–Pb–O7	139.06(8)
O5–N7	2.996(3)	N2–Pb–O1	69.66(8)
		N2–Pb–O2	98.42(9)
		O9–Pb–O7	69.96(8)
		O9–Pb–O1	101.07(9)
		O9–Pb–O2	143.96(8)
		N6–Pb–O7	64.92(8)
		N6–Pb–O1	130.46(9)
		N6–Pb–O2	86.57(9)
		O7–Pb–O1	127.98(9)
		O7–Pb–O2	119.68(9)
		O1–Pb–O2	44.18(8)

with the monomeric units linked by N_{triazine}–O_{perchlorate} noncovalent donor ··· acceptor interactions.

Supplementary material

Crystallographic data for the structures reported in the paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no, CCDC-262580 for [Pb(PDPT)(NO₃)₂]_n (**1**) and 262581 for [Pb(PDPT)₂(ClO₄)₂EtOH].CH₃OH (**2**). Copies of the data can be obtained on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: +44–1223/336033; E-mail: deposit@ccdc.cam.ac.uk].

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