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### Two lead coordination polymers with nitrilotriacetic acid and oxydiacetic acid: synthesis, characterization, and crystal structure

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## Two lead coordination polymers with nitrilotriacetic acid and oxydiacetic acid: synthesis, characterization, and crystal structure

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Two lead coordination compounds [Pb<sub>2</sub>(nta)]NO<sub>3</sub> (**1**) and [Pb(oda)] (**2**) have been synthesized by slow evaporation or hydrothermal conditions using nitrilotriacetic acid (nta) and 2,2'-oxydiacetic acid (oda) as ligands, respectively. Their structures were determined by single-crystal X-ray diffraction and further characterized by X-ray powder diffraction, infrared absorption spectrum, and thermogravimetric analysis. Compound **1** is a 2-D honeycomb-like layer structure with (6,3) topology. When the bonding limit of Pb–O extends from 2.76 to 2.90 Å, potential weak Pb–O bonds can be found in **1**, and the 2-D layer structure can be further linked to generate a 3-D 4-connected supramolecular **sra** net with the (4<sup>2</sup>.6<sup>3</sup>.8) Schläfli symbol. Compound **2** contains a 1-D infinite Pb–O chain which is connected through μ<sub>3</sub>-, μ<sub>4</sub>-, and μ<sub>5</sub>-coordination modes of oda to form a new 3-D structure.

**Keywords:** Coordination polymer; Lead; Nitrilotriacetic acid; 2,2'-Oxydiacetic acid

### 1. Introduction

Coordination polymers of lead receive attention because of their structural diversity and potential applications in nonlinear optics, ion exchange, luminescence, and biological activity [1–6]. Lead, bearing a stereochemically active 6s electron pair and large radius, can adopt variable coordination numbers ranging from 2 to 10 and versatile coordination modes such as hemidirected and holodirected geometries providing opportunity for the construction of coordination compounds [7].

Among lead coordination polymers, most products contain rigid ligands, for example, aromatic polycarboxylates, pyridyl carboxylic acids, aromatic ketones, and their N-oxide derivatives [8–14], while only a few examples have flexible ligands, such as citric acid, tartaric acid, N,N-bis[(6-carboxypyridin-2-yl)-methyl]ethylamine, and

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2,2'-oxydiacetic acid (oda) [15–18]. In our research, two flexible ligands, nitrilotriacetic acid (nta) and 2,2'-oda, are used to construct lead coordination polymers. Many coordination polymers containing nta and oda build frameworks with rare earth metals [16–23], transition metals [24–28], and alkaline earth metals [29, 30], but compounds with lead are less reported [31, 32]. Herein, we report two lead coordination polymers,  $[\text{Pb}_2(\text{nta})]\text{NO}_3$  (**1**) and  $[\text{Pb}(\text{oda})]$  (**2**), with nta and oda. Compound **1** was synthesized by slow evaporation and **2** by hydrothermal synthesis. Compound **1** is a 3-D 4-connected supramolecular **sra** framework with the  $(4^2,6^3,8)$  Schläfli symbol and **2** is a 3-D structure containing a 1-D infinite Pb–O chain.

## 2. Experimental

### 2.1. Materials and instrumentation

All chemicals were obtained from commercial sources and used without purification. Powder X-ray diffraction (XRD) data were obtained using a Shimadzu XRD-6000 diffractometer with Cu-K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). IR spectra were recorded from 400 to  $4000 \text{ cm}^{-1}$  on a Nicolet Impact 410 spectrometer, using KBr pellets. Thermogravimetric analyses (TGAs) were conducted on a Perkin Elmer TGA 7 thermogravimetric analyzer in air with a heating rate of  $10^\circ\text{C min}^{-1}$ .

### 2.2. Synthesis

**2.2.1. Synthesis of  $[\text{Pb}_2(\text{nta})]\text{NO}_3$  (**1**).** A mixture of  $\text{H}_3\text{nta}$  (0.096 g, 0.5 mmol) and  $\text{NH}_3 \cdot \text{H}_2\text{O}$  (3 mL,  $0.5 \text{ mmol mL}^{-1}$ ) dissolved in 5 mL of distilled water was added to an aqueous solution (5 mL) of  $\text{Pb}(\text{NO}_3)_2$  (0.0829 g, 0.5 mmol). After stirring for 30 min, the solution was concentrated by slow evaporation for a week, colorless crystals of **1** were obtained (yield = 69% based on Pb). IR (KBr pellet,  $\text{cm}^{-1}$ ): 3015(w), 2362(w), 1633(s), 1544(s), 1490(m), 1367(s), 1297(m), 811(s), and 578(m)  $\text{cm}^{-1}$ .

**2.2.2. Synthesis of  $[\text{Pb}(\text{oda})]$  (**2**).** A mixture of  $\text{Pb}(\text{NO}_3)_2$  (0.0415 g, 0.25 mmol), oda (0.0335 g, 0.25 mmol), NaOH (0.020 g, 0.5 mmol), triazole (0.035 g, 0.5 mmol), and 9 mL of water was sealed in a 23 mL Teflon-lined stainless steel container and heated to  $170^\circ\text{C}$  for 3 days. After cooling to room temperature, colorless crystals of **2** were obtained (yield = 56% based on Pb). IR (KBr pellet,  $\text{cm}^{-1}$ ): 2910(w), 1556(s), 1421(m), 1341(m), 1303(s), 1118(s), 1035(s), 918(s), 728(s), and 552(s)  $\text{cm}^{-1}$ .

### 2.3. Crystal structure determination

Single crystals of **1** and **2** with suitable dimensions were mounted onto thin glass fibers. Intensity data were collected on a Rigaku R-AXIS RAPID diffractometer equipped with graphite-monochromated Mo-K $\alpha$  ( $\lambda = 0.71073 \text{ \AA}$ ) radiation in the  $\omega$  scanning mode at room temperature. No significant decay was observed during the data collection. Data were processed on a Pentium PC, using Bruker AXS windows NT SHELXTL software package (version 5.10) [33, 34]. Neutral atom scattering factors were taken from Cromer and Waber [35]. The structures were solved by direct methods.

Table 1. Crystal data and structure refinement parameters for **1** and **2**.

	<b>1</b>	<b>2</b>
Identification code	<b>1</b>	<b>2</b>
Empirical formula	C <sub>6</sub> H <sub>6</sub> N <sub>2</sub> O <sub>9</sub> Pb <sub>2</sub>	C <sub>4</sub> H <sub>4</sub> O <sub>5</sub> Pb
Formula weight	664.51	339.27
Crystal system	Monoclinic	Triclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>c</i>	<i>P</i> $\bar{1}$
Unit cell dimensions (Å, °)		
<i>a</i>	8.3780(10)	7.6017
<i>b</i>	9.9054(10)	10.8930
<i>c</i>	13.6247(2)	12.9048
$\alpha$	90	114.369
$\beta$	99.921(10)	94.109
$\gamma$	90	91.4090
Volume (Å <sup>3</sup> ), <i>Z</i>	1113.77(2), 4	969.84, 4
Calculated density (g cm <sup>-3</sup> )	3.963	3.485
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.096	1.115
Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0396 <i>wR</i> <sub>2</sub> = 0.1062	<i>R</i> <sub>1</sub> = 0.0402 <i>wR</i> <sub>2</sub> = 0.1047
<i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.0422 <i>wR</i> <sub>2</sub> = 0.1083	<i>R</i> <sub>1</sub> = 0.0453 <i>wR</i> <sub>2</sub> = 0.1075

$$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|; wR_2 = \Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]^{1/2}.$$

All non-hydrogen atoms were refined anisotropically. Hydrogens were placed geometrically and located based on a riding model. Experimental details for the structural determinations of **1** and **2** are summarized in table 1.

### 3. Results and discussion

#### 3.1. Synthesis and characterization

The powder XRD patterns of **1** and **2** are consistent with the simulated ones on the basis of single-crystal structures, indicating phase purity of the synthesized samples. The differences in reflection intensities between the simulated and the experimental patterns are due to the variation in the crystal orientation of the powder samples.

The IR spectra of **1** and **2** in the range of 1544–1581 cm<sup>-1</sup> and 1367–1380 cm<sup>-1</sup> correspond to  $\nu_{\text{as}}(\text{C}-\text{O})$  and  $\nu_{\text{sym}}(\text{C}-\text{O})$  of carboxylate, and bands in the range of 2362–3023 cm<sup>-1</sup> and 578–1297 cm<sup>-1</sup> are related to  $\nu(\text{C}-\text{H})$  of nta and oda ligands.

The TGA of **1** and **2** was carried out in air (see figure S1 in “Supplementary material”). The TGA curve shows that **1** is stable at 280°C and a gradual weight loss of 34.66% occurs in the range of 280–500°C corresponding to the loss of nta and NO<sub>3</sub><sup>-</sup> (Calcd 32.83%). The remaining weight of 65.34% is attributed to the final product of PbO (Calcd 67.17%). The TGA curve of **2** shows a weight loss of 35.22% at 270–430°C corresponding to the loss of oda (Calcd 34.21%). The remaining weight of 64.78% is PbO (Calcd 65.79%).

#### 3.2. Structural description

**3.2.1. Crystal structure of [Pb<sub>2</sub>(nta)]NO<sub>3</sub> (**1**).** The asymmetric unit of **1** is composed of two leads, one nta, and one NO<sub>3</sub><sup>-</sup> (figure 1). Pb1 is hemidirected with four

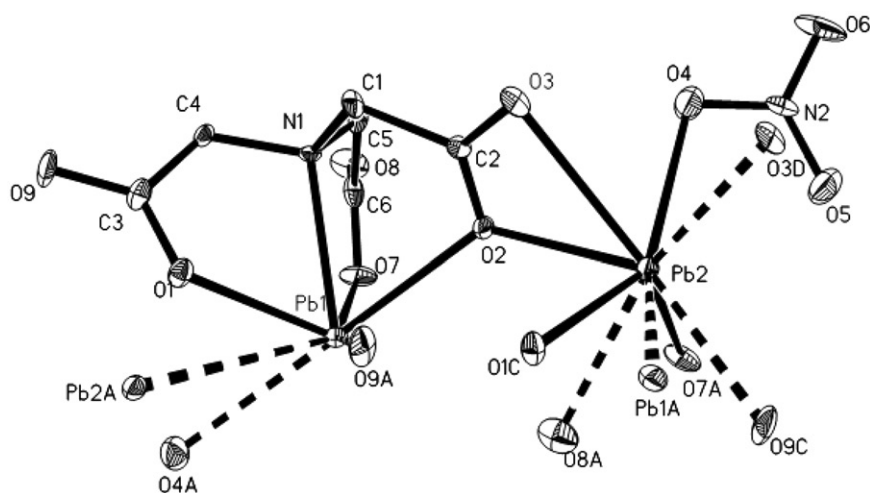


Figure 1. ORTEP of the coordination environments of Pb in **1** with 30% thermal ellipsoids. All hydrogens are omitted for clarity.

coordinates – N1, O1, O2, and O7 [Pb1–N1 = 2.525(8) Å, Pb1–O1 = 2.508(7) Å, Pb1–O2 = 2.563(7) Å, Pb1–O7 = 2.442(7) Å] from one nta. Pb2 is coordinated to five oxygens from three different nta ligands and one nitrate, in which O2 and O3 are in the same ligand in a bidentate chelate coordination with the Pb–O bond length 2.425(7) and 2.743(8) Å, O4 comes from a nitrate ion [Pb2–O4 = 2.651(9) Å] and O7A and O1C from a carboxyl of a different nta with the Pb–O bond length 2.528(7) and 2.549(7) Å.

In **1**, a hexanuclear unit  $Pb_6(nta)_3$  (figure 2a) is formed by the nta ligands through three bidentate-bridging carboxylates (scheme 1a) to connect Pb1 and Pb2 alternately. Parallel stacking units  $Pb_6(nta)_3$  build up 2-D honeycomb-like layer structure in the  $xy$ -plane (figure 2a). The interaction of adjacent parallel layers comes from weak Pb–O bonds. For **1**, when Pb–O bond distance extends from 2.76 to 2.90 Å, two weak bonds [Pb1–O4A = 2.816(7) Å, Pb1–O9A = 2.762(6) Å] are found. For Pb2, three weak bonds [Pb2–O9A = 2.862(8), Pb2–O3 = 2.858(7), and Pb2–O8A = 2.761(9) Å] are found in the direction of the void and the coordination geometry of Pb2 changes from hemidirected to holodirected. Through two weak bonds (Pb1–O4A and Pb2–O3), a 3-D supramolecular framework is formed (figure 2b). In **1**, there is another weak metallophilic Pb–Pb interaction at 4.140 Å which has been reported in  $[Pb_3(phen)_3(H_2O)(sip)_2]_n \cdot 3H_2O$  [36].

From a topological point of view, when Pb acts as a 3-connected node and nta are linkers, the topology of **1** is a (6,3) net (figure 2c) which is connected by  $NO_3$  via  $Pb \cdots O$  weak bond. As a result, the 3-connected Pb node in the (6,3) net is extended to a 4-connected node in the 3-D supramolecular framework ascribed to a 4-connected *sra* net with the  $(4^2.6^3.8)$  Schläfli symbol [37].

**3.2.2. Crystal structure of  $[Pb(oda)]$  (**2**).** In the asymmetric unit of **2**, there are three Pb's and three oda ligands (figure 3). Pb1 is a six-coordinate composed of three oxygens [Pb1–O9 = 2.619(7), Pb1–O8 = 2.614(8), and Pb1–O7 = 2.697(7) Å] from one oda and three oxygens [Pb1–O7A = 2.492(7), Pb1–O1 = 2.593(9), and Pb1–O11 = 2.521(9) Å]

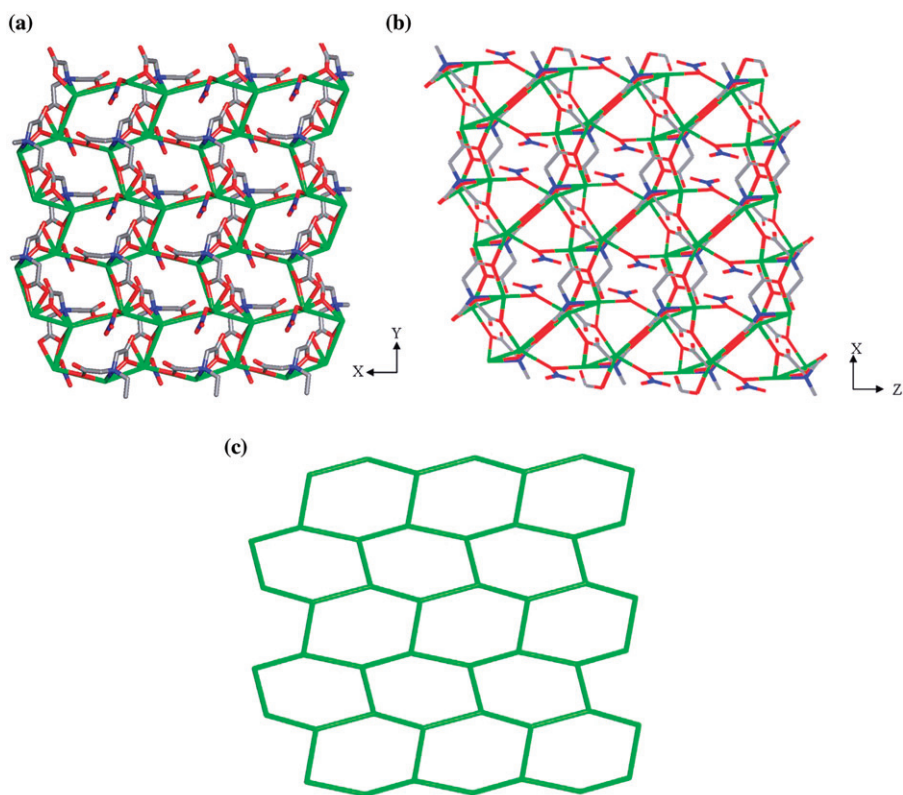
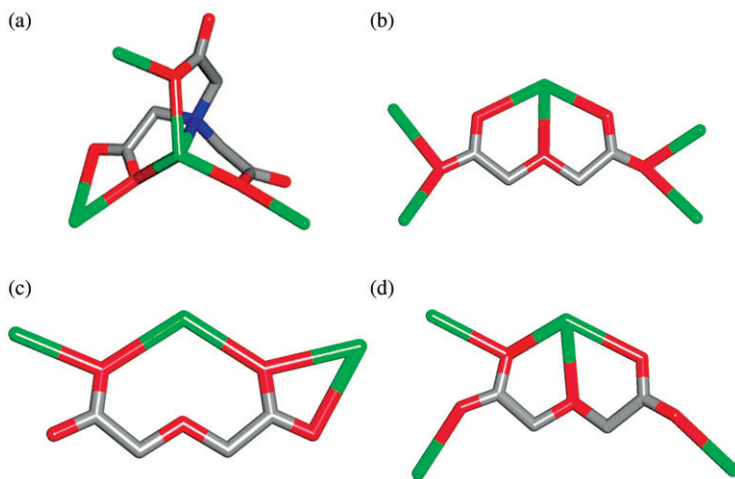


Figure 2. (a) View of the 2-D layer structure in  $xy$ -plane; (b) 3-D supramolecular framework of **1** formed through  $\text{Pb} \cdots \text{O}$  weak bonds in  $xz$ -plane; (c) schematic description of the 2-D honeycomb-like layer structure with (6,3) topology in the  $xy$ -plane.



Scheme 1. Coordination modes of nta and oda.

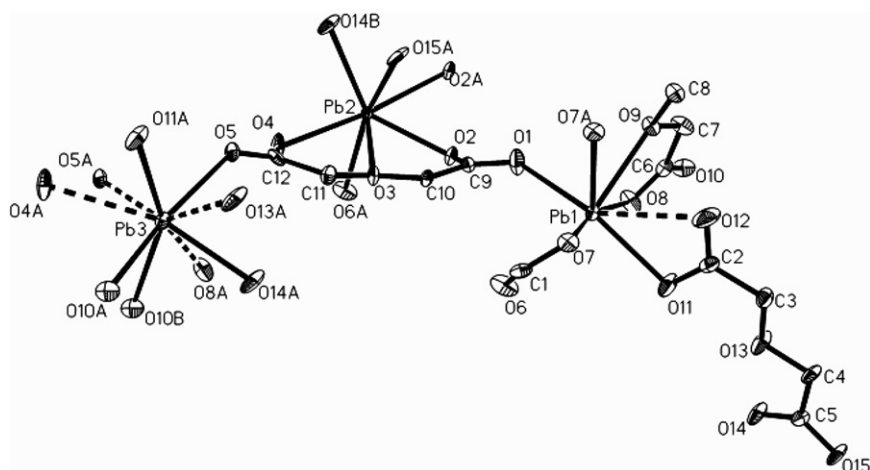


Figure 3. ORTEP of the coordination environments of Pb in **2** with 30% thermal ellipsoids. All hydrogens are omitted for clarity.

from the other three oda ligands. Pb2 is seven-coordinate in hemidirected geometry supplied by three oxygens [Pb2–O2 = 2.448(7), Pb2–O3 = 2.648(7), and Pb2–O4 = 2.659(8) Å] from one oda, two oxygens [Pb2–O2A = 2.660(7) and Pb2–O6A = 2.544(8) Å] from two different oda ligands and two oxygens [Pb2–O14B = 2.644(8) and Pb2–O15A = 2.643(9) Å] from one oda. Pb3 is five-coordinate with three oxygens [Pb3–O10A = 2.547(8), Pb3–O10B = 2.631(9), and Pb3–O5 = 2.585(7) Å] from three different oda ligands and two oxygens [Pb3–O11A = 2.599(9) and Pb3–O14A = 2.542(8) Å] from one oda. Oda shows three different  $\mu_3$ -,  $\mu_4$ -, and  $\mu_5$ -coordination modes (scheme 1b–d), respectively. In Pb(oda)(H<sub>2</sub>O), only  $\mu_5$ -coordination appeared (scheme 1b) [31], which leads to the different structure.

In **2**, basic [Pb(2)<sub>2</sub>O<sub>2</sub>] and [Pb(3)<sub>2</sub>O<sub>2</sub>] units are formed through Pb2 and Pb3 atoms linked with O2 and O10, respectively. The connection of [Pb<sub>2</sub>O<sub>2</sub>] through O5 contributes to the formation of 1-D Pb–O chains (figure 4a) in which the intrachain distances of Pb2...Pb2 and Pb3...Pb3 are 4.300 and 4.223 Å, respectively. Two adjacent chains are linked by O6 and O10 from oda to form a 2-D layer (figure 4b). The 2-D layer is further connected by Pb1-oda in a  $\mu_5$ -fashion (scheme 1c) to form a 3-D coordination polymer (figure 4c).

When the Pb–O bonding limit extends from 2.76 to 2.90 Å, five weak interactions [Pb1–O12 = 2.847(7), Pb3–O5A = 2.766(8), Pb3–O8A = 2.885(8), Pb3–O4A = 2.819(9), and Pb3–O13A = 2.800(7) Å] are found and the geometry of Pb3 changes from hemidirected to holodirected constructing a more complicated 3-D structure.

#### 4. Conclusion

Two lead compounds [Pb<sub>2</sub>(nta)]NO<sub>3</sub> (**1**) and [Pb(oda)] (**2**) have been obtained with nta and oda. In **1**, Pb's combine with nta ligands to give a 2-D honeycomb-like layer



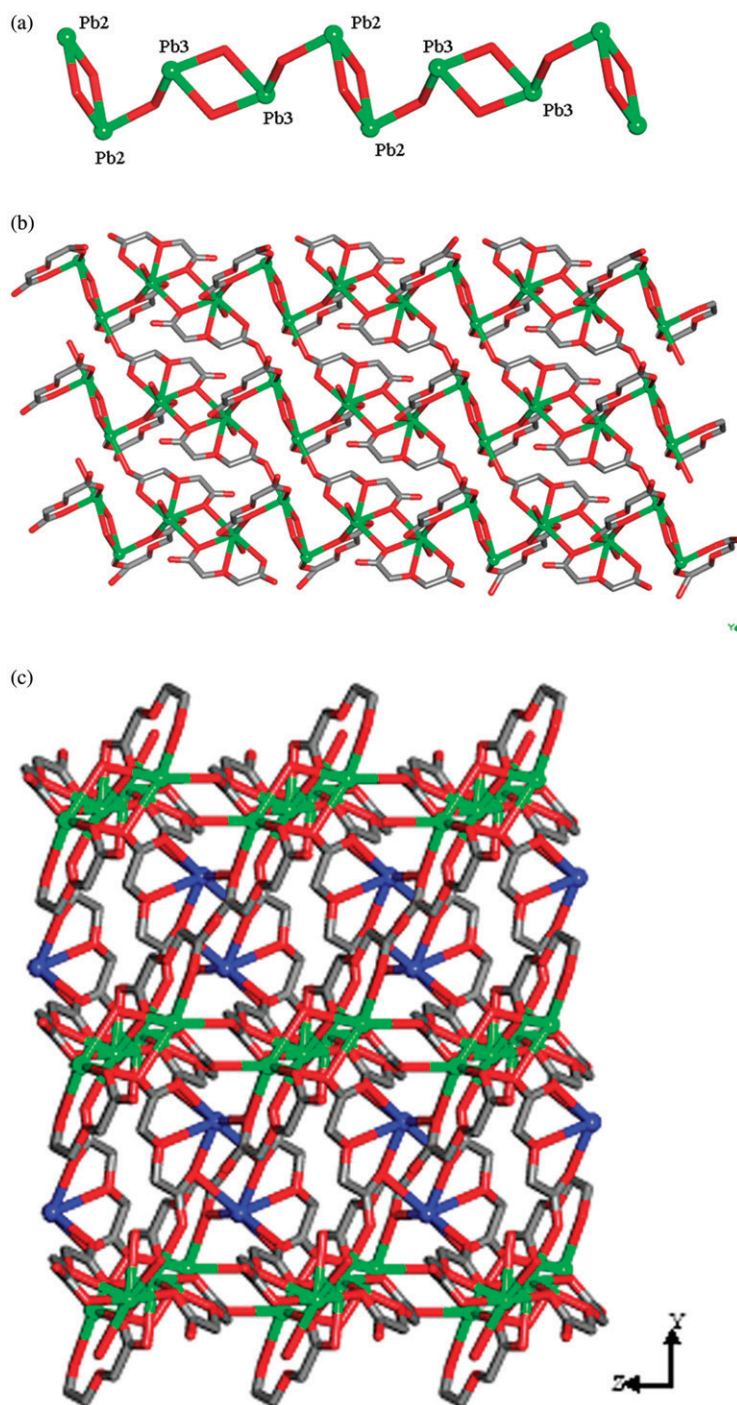


Figure 4. (a) View of the 1-D chain structure of **2**; (b) view of the 2-D layer structure containing the 1-D chain; (c) view of the 3-D structure of **2** in  $yz$ -plane [Pb1(blue), Pb2, and Pb3 (green)].



structure with (6,3) topology, which is further connected to create the 4-connected **sra** net with the (4<sup>2</sup>.6<sup>3</sup>.8) Schläfli symbol through weak Pb–O bonds. In **2**, oda ligands link two unique lead centers to generate Pb–O chains which are further extended to a 3-D structure *via* oda and the third unique lead center. For **1** and **2**, when the bonding limit of Pb–O extends from 2.76 to 2.90 Å, weak Pb–O bonds can be found which cause the transformation of geometry from hemidirected to holodirected for Pb<sub>2</sub> in **1** and Pb<sub>3</sub> in **2**. The weak Pb–O bonds result in a 3-D supramolecular framework for **1**.

### Supplementary material

CCDC-711125 and CCDC-731557 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge at [www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk) [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) +44-1223/336-033; E-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)].

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