

Poly[(μ_4 -pyridine-2,3-dicarboxylato)-lead(II)]Shie Fu Lush^a and Fwu Ming Shen^{b*}^aDepartment of General Education Center, Yuanpei University, HsinChu 30015, Taiwan, and ^bDepartment of Biotechnology, Yuanpei University, HsinChu 30015, Taiwan

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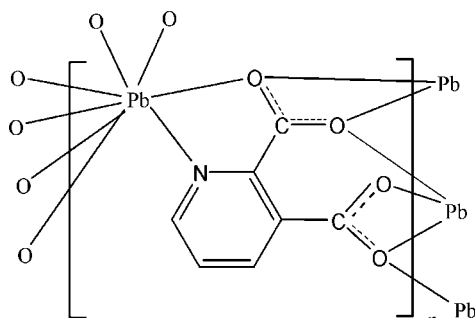
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Key indicators: single-crystal X-ray study; $T = 297$ K; mean $\sigma(\text{C}-\text{C}) = 0.016$ Å; R factor = 0.073; wR factor = 0.204; data-to-parameter ratio = 12.6.

In the title coordination polymer, $[\text{Pb}(\text{C}_7\text{H}_3\text{NO}_4)]_n$, the Pb^{II} ion is eight-coordinated in a distorted square-antiprismatic geometry formed by one pyridine N atom and seven carboxylate O atoms from four pyridine-2,3-dicarboxylate (pda) anions. In the pda anion, the dihedral angles between the pyridine ring and carboxylate groups are 19.5 (6) and 73.3 (6)°. The carboxylate groups of the pda anions bridge the Pb ions, forming a two-dimensional coordination polymer parallel to (100). Weak intermolecular C—H...O hydrogen bonding is present in the crystal structure.

Related literature

For the coordination modes of the pyridine-2,3-dicarboxylate anion, see: Aghabozorg *et al.* (2007); Baruah *et al.* (2007); Li *et al.* (2006). For the biological activity of pyridine-2,3-dicarboxylic acid, see: Xiang *et al.* (2006); Yang *et al.* (2006); Zhang *et al.* (2008). For the inert lone-pair effect, see: Liat *et al.* (1998). For longer Pb—O bonds, see: Mao *et al.* (2006); Yang *et al.* (2010).



Experimental

Crystal data

| | |
|--|-----------------------------------|
| $[\text{Pb}(\text{C}_7\text{H}_3\text{NO}_4)]$ | $V = 751.84$ (11) Å ³ |
| $M_r = 372.30$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 11.6943$ (9) Å | $\mu = 22.42$ mm ⁻¹ |
| $b = 4.5392$ (4) Å | $T = 297$ K |
| $c = 14.1636$ (12) Å | $0.54 \times 0.23 \times 0.04$ mm |
| $\beta = 90.046$ (2)° | |

Data collection

| | |
|--|--|
| Bruker SMART CCD area-detector diffractometer | 4013 measured reflections |
| Absorption correction: multi-scan (SADABS; Bruker, 2001) | 1484 independent reflections |
| $T_{\min} = 0.659$, $T_{\max} = 1.000$ | 1336 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.125$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.073$ | 118 parameters |
| $wR(F^2) = 0.204$ | H-atom parameters constrained |
| $S = 1.13$ | $\Delta\rho_{\text{max}} = 4.56$ e Å ⁻³ |
| 1484 reflections | $\Delta\rho_{\text{min}} = -5.06$ e Å ⁻³ |

Table 1

Selected bond lengths (Å).

| | | | |
|----------------------|-----------|----------------------|-----------|
| Pb—N | 2.651 (7) | Pb—O2 ⁱ | 2.566 (9) |
| Pb—O1 ⁱ | 2.816 (7) | Pb—O3 ⁱⁱⁱ | 2.397 (9) |
| Pb—O1 ⁱⁱⁱ | 2.911 (6) | Pb—O3 ⁱⁱ | 2.754 (9) |
| Pb—O2 | 2.592 (7) | Pb—O4 ⁱⁱ | 2.845 (7) |

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $x, -y - \frac{1}{2}, z + \frac{1}{2}$.

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{C5}-\text{H5A}\cdots\text{O3}^{\text{iii}}$ | 0.93 | 2.57 | 3.164 (13) | 122 |

Symmetry code: (iii) $x, -y - \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5124).

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supplementary materials

Acta Cryst. (2011). E67, m163-m164 [doi:10.1107/S1600536811000419]

Poly[(μ_4 -pyridine-2,3-dicarboxylato)lead(II)]

S. F. Lush and F. M. Shen

Comment

The pyridine-2,3-dicarboxylic acid (pdaH₂) is a typical chelated-form ligand. Its biological importance has been described in several literatures (Xiang *et al.*, 2006; Yang *et al.*, 2006; Zhang *et al.*, 2008). Pda shows diverse coordination modes, such as monodentate (Baruah *et al.*, 2007), μ_2 -bridging (Aghabozorg *et al.*, 2007), μ_3 -bridging (Li *et al.*, 2006). Here we describe the title compound in which the pda is a μ_4 -bridging ligand (Fig. 1).

The structure of a coordination polymer [Pb(C₇H₃NO₄)_n]_n, the lead ion is eight-coordinated with a distorted square-anti-prismatic geometry formed by one O-monodentate pda⁻² ligand, one N,O-bidentate pda⁻² ligand, one O,O'-bidentate pda⁻² ligand and one O,O',O''-tridentate pda⁻² ligand (Table 1). According to "inert-pair effect", the coordination number of Pb^{II} is variable, and the lengths of bonds to Pb^{II} vary in a wide range (Liat *et al.*, 1998). Longer distance is observed between Pb and O1 (2.911 (6) Å); some long Pb—O weak bonds have also been reported in reported lead complexes (Mao *et al.*, 2006; Yang *et al.*, 2010). The carboxylate group of pda⁻² ligand bridges four Pb^{II} ion forming a 2-D framework is constructed.

There are no classic intermolecular hydrogen-bonding in the title compound, but intermolecular C—H \cdots O weak interaction (Table 2) and ring \cdots metal interaction help to stabilize the crystal structure, the Cg3 (Pb/O1—O2—C6) \cdots Pb interaction is 3.877 Å (symmetry code: 1 - x, -1/2 + y, 3/2 - z).

Experimental

An aqueous solution (5 ml) containing Pb(NO₃)₂ (0.164 g, 0.50 mmol) and 1,2-bis(4-pyridyl)ethane (0.0934 g, 0.50 mmol) was added to an aqueous solution (5 ml) of pyridine-2,3-dicarboxylic acid (0.0838 g, 0.50 mmol), and the mixture was stirred for 30 minutes and then filtered. The solution was placed in a 23 ml Teflon-lined reactor, heated at 423 K for 3 days, then cooled slowly to room temperature. The colorless transparent single crystals of the title compound were obtained in 45.67% yield (based on Pb).

Refinement

H atoms were positioned geometrically with C—H = 0.93 (aromatic), and were refined using a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

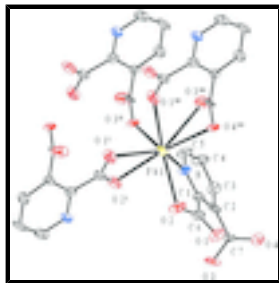


Fig. 1. View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. H atoms have been omitted for clarity. [Symmetry codes: (i) $-x + 1, y - 1/2, -z + 3/2$; (ii) $x, -y - 1/2, z + 1/2$; (iii) $x, -y + 1/2$].

Poly[(μ_4 -pyridine-2,3-dicarboxylato)lead(II)]

Crystal data

[Pb(C₇H₃NO₄)]

$M_r = 372.30$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.6943$ (9) Å

$b = 4.5392$ (4) Å

$c = 14.1636$ (12) Å

$\beta = 90.046$ (2)°

$V = 751.84$ (11) Å³

$Z = 4$

$F(000) = 664$

$D_x = 3.289$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2856 reflections

$\theta = 2.3$ – 26.0 °

$\mu = 22.42$ mm⁻¹

$T = 297$ K

Parallelepiped, colorless

$0.54 \times 0.23 \times 0.04$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

Detector resolution: 9 pixels mm⁻¹

ω scan

Absorption correction: multi-scan (SADABS; Bruker, 2001)

$T_{\min} = 0.659, T_{\max} = 1.000$

4013 measured reflections

1484 independent reflections

1336 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.125$

$\theta_{\max} = 26.0$ °, $\theta_{\min} = 1.7$ °

$h = -14 \rightarrow 10$

$k = -5 \rightarrow 5$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.073$

$wR(F^2) = 0.204$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

| | |
|------------------|--|
| $S = 1.13$ | $w = 1/[\sigma^2(F_o^2) + (0.1477P)^2]$ |
| 1484 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 118 parameters | $(\Delta/\sigma)_{\max} = 0.001$ |
| 0 restraints | $\Delta\rho_{\max} = 4.56 \text{ e } \text{Å}^{-3}$ |
| | $\Delta\rho_{\min} = -5.06 \text{ e } \text{Å}^{-3}$ |

Special details

Experimental. Elemental analysis: calculated for $C_7H_3NO_4Pb$: (Mr=372.29) C, 22.56; H, 0.81; N, 3.76%. Found: C, 22.47; H, 0.89; N, 3.85%. IR data (cm^{-1}): 3429(s), 1602(s), 1579(s), 1551(s), 1459(w), 1385(s), 1276(w), 1236(w), 1105(m), 871(m), 825(m), 779(m), 711(s), 700(m), 660(m), 603(w), 534(w), 443(w).

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|-------------|----------------------------------|
| Pb | 0.60762 (4) | 0.01636 (10) | 0.85892 (3) | 0.0213 (3) |
| O1 | 0.6007 (6) | 0.3400 (19) | 0.5591 (4) | 0.035 (2) |
| O2 | 0.5381 (7) | 0.120 (2) | 0.6883 (5) | 0.037 (3) |
| O3 | 0.6905 (9) | -0.061 (2) | 0.4106 (6) | 0.032 (3) |
| O4 | 0.8222 (6) | 0.2863 (17) | 0.4294 (4) | 0.038 (2) |
| N | 0.7437 (6) | -0.1315 (19) | 0.7170 (5) | 0.024 (2) |
| C1 | 0.7208 (12) | -0.0178 (18) | 0.6321 (9) | 0.021 (4) |
| C2 | 0.7951 (11) | -0.058 (3) | 0.5526 (9) | 0.021 (3) |
| C3 | 0.8932 (8) | -0.216 (2) | 0.5715 (6) | 0.027 (3) |
| C4 | 0.9192 (9) | -0.325 (3) | 0.6603 (7) | 0.038 (4) |
| C5 | 0.8383 (9) | -0.288 (3) | 0.7300 (6) | 0.036 (3) |
| C6 | 0.6141 (8) | 0.163 (3) | 0.6231 (6) | 0.023 (3) |
| C7 | 0.7687 (9) | 0.068 (2) | 0.4580 (7) | 0.019 (3) |
| H3A | 0.94410 | -0.25180 | 0.52240 | 0.0320* |
| H4A | 0.98820 | -0.41980 | 0.67230 | 0.0450* |
| H5A | 0.85060 | -0.37490 | 0.78850 | 0.0430* |

Atomic displacement parameters (Å^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|-------------|-------------|-------------|
| Pb | 0.0207 (5) | 0.0242 (5) | 0.0189 (5) | -0.0031 (1) | -0.0051 (3) | -0.0016 (1) |
| O1 | 0.035 (4) | 0.045 (5) | 0.026 (3) | 0.015 (4) | 0.007 (3) | 0.012 (3) |
| O2 | 0.027 (4) | 0.061 (6) | 0.024 (3) | 0.017 (5) | 0.001 (3) | 0.013 (4) |

supplementary materials

| | | | | | | |
|----|-----------|-----------|-----------|------------|------------|------------|
| O3 | 0.039 (5) | 0.031 (4) | 0.027 (4) | 0.006 (4) | -0.022 (4) | -0.013 (4) |
| O4 | 0.044 (4) | 0.041 (5) | 0.030 (3) | -0.007 (4) | -0.010 (3) | 0.009 (3) |
| N | 0.018 (4) | 0.032 (5) | 0.021 (3) | 0.004 (3) | -0.003 (3) | 0.004 (3) |
| C1 | 0.018 (7) | 0.028 (6) | 0.017 (6) | -0.002 (3) | 0.000 (5) | -0.001 (3) |
| C2 | 0.006 (5) | 0.037 (5) | 0.021 (5) | 0.002 (4) | -0.004 (4) | -0.010 (5) |
| C3 | 0.023 (5) | 0.032 (6) | 0.026 (4) | 0.006 (4) | 0.003 (4) | 0.003 (4) |
| C4 | 0.029 (6) | 0.054 (8) | 0.031 (5) | 0.018 (6) | -0.013 (4) | 0.010 (5) |
| C5 | 0.041 (6) | 0.046 (7) | 0.021 (4) | 0.009 (5) | -0.009 (4) | 0.011 (4) |
| C6 | 0.018 (5) | 0.031 (5) | 0.021 (4) | 0.006 (4) | -0.005 (3) | 0.002 (4) |
| C7 | 0.008 (5) | 0.030 (5) | 0.019 (4) | 0.006 (4) | -0.007 (4) | 0.004 (4) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|---------------------------------------|------------|--------------------------------------|------------|
| Pb—N | 2.651 (7) | N—C1 | 1.336 (14) |
| Pb—O1 ⁱ | 2.816 (7) | N—C5 | 1.327 (14) |
| Pb—O1 ⁱⁱ | 2.911 (6) | C1—C2 | 1.435 (18) |
| Pb—O2 | 2.592 (7) | C1—C6 | 1.499 (17) |
| Pb—O2 ⁱ | 2.566 (9) | C2—C3 | 1.379 (16) |
| Pb—O3 ⁱⁱⁱ | 2.397 (9) | C2—C7 | 1.489 (16) |
| Pb—O3 ⁱⁱ | 2.754 (9) | C3—C4 | 1.385 (14) |
| Pb—O4 ⁱⁱ | 2.845 (7) | C4—C5 | 1.378 (14) |
| O1—C6 | 1.221 (13) | C3—H3A | 0.9300 |
| O2—C6 | 1.297 (12) | C4—H4A | 0.9300 |
| O3—C7 | 1.276 (14) | C5—H5A | 0.9300 |
| O4—C7 | 1.240 (12) | | |
| O2—Pb—N | 61.8 (2) | Pb ^v —O1—C6 | 150.1 (7) |
| O1 ⁱ —Pb—O2 | 99.5 (2) | Pb ^{iv} —O1—Pb ^v | 111.3 (2) |
| O2—Pb—O2 ⁱ | 71.1 (3) | Pb—O2—C6 | 118.5 (6) |
| O2—Pb—O3 ⁱⁱⁱ | 124.6 (3) | Pb—O2—Pb ^{iv} | 125.3 (3) |
| O1 ⁱⁱ —Pb—O2 | 149.2 (2) | Pb ^{iv} —O2—C6 | 99.5 (7) |
| O2—Pb—O3 ⁱⁱ | 101.2 (3) | Pb ^{vi} —O3—C7 | 147.7 (8) |
| O2—Pb—O4 ⁱⁱ | 123.1 (2) | Pb ^v —O3—C7 | 88.8 (6) |
| O2—Pb—C7 ⁱⁱ | 121.1 (3) | Pb ^{vi} —O3—Pb ^v | 123.4 (4) |
| O1 ⁱ —Pb—N | 139.5 (2) | Pb ^v —O4—C7 | 85.5 (6) |
| O2 ⁱ —Pb—N | 91.4 (2) | Pb—N—C1 | 117.7 (7) |
| O3 ⁱⁱⁱ —Pb—N | 76.7 (3) | Pb—N—C5 | 122.1 (6) |
| O1 ⁱⁱ —Pb—N | 144.3 (2) | C1—N—C5 | 119.9 (9) |
| O3 ⁱⁱ —Pb—N | 102.6 (3) | N—C1—C2 | 122.4 (11) |
| O4 ⁱⁱ —Pb—N | 79.4 (2) | N—C1—C6 | 117.0 (10) |
| N—Pb—C7 ⁱⁱ | 97.8 (3) | C2—C1—C6 | 120.5 (10) |
| O1 ⁱ —Pb—O2 ⁱ | 48.2 (2) | C1—C2—C3 | 114.7 (11) |
| O1 ⁱ —Pb—O3 ⁱⁱⁱ | 88.8 (3) | C1—C2—C7 | 122.2 (11) |
| O1 ⁱ —Pb—O1 ⁱⁱ | 68.73 (19) | C3—C2—C7 | 123.1 (10) |
| O1 ⁱ —Pb—O3 ⁱⁱ | 116.7 (3) | C2—C3—C4 | 123.0 (10) |

| | | | |
|---|-------------|--|-------------|
| O1 ⁱ —Pb—O4 ⁱⁱ | 135.08 (17) | C3—C4—C5 | 117.2 (10) |
| O1 ⁱ —Pb—C7 ⁱⁱ | 121.8 (2) | N—C5—C4 | 122.6 (9) |
| O2 ⁱ —Pb—O3 ⁱⁱⁱ | 75.1 (3) | O1—C6—O2 | 122.7 (10) |
| O1 ⁱⁱ —Pb—O2 ⁱ | 113.1 (2) | O1—C6—C1 | 122.0 (9) |
| O2 ⁱ —Pb—O3 ⁱⁱ | 158.7 (3) | O2—C6—C1 | 115.3 (10) |
| O2 ⁱ —Pb—O4 ⁱⁱ | 153.8 (2) | O3—C7—O4 | 123.8 (9) |
| O2 ⁱ —Pb—C7 ⁱⁱ | 167.3 (2) | O3—C7—C2 | 116.4 (9) |
| O1 ⁱⁱ —Pb—O3 ⁱⁱⁱ | 84.7 (3) | Pb ^v —C7—O3 | 66.1 (6) |
| O3 ⁱⁱⁱ —Pb—O3 ⁱⁱ | 123.4 (3) | O4—C7—C2 | 119.8 (9) |
| O3 ⁱⁱⁱ —Pb—O4 ⁱⁱ | 78.9 (3) | Pb ^v —C7—O4 | 70.3 (5) |
| O3 ⁱⁱⁱ —Pb—C7 ⁱⁱ | 98.4 (3) | Pb ^v —C7—C2 | 142.1 (7) |
| O1 ⁱⁱ —Pb—O3 ⁱⁱ | 63.3 (2) | C2—C3—H3A | 119.00 |
| O1 ⁱⁱ —Pb—O4 ⁱⁱ | 67.22 (18) | C4—C3—H3A | 118.00 |
| O1 ⁱⁱ —Pb—C7 ⁱⁱ | 54.8 (2) | C3—C4—H4A | 121.00 |
| O3 ⁱⁱ —Pb—O4 ⁱⁱ | 46.7 (3) | C5—C4—H4A | 121.00 |
| O3 ⁱⁱ —Pb—C7 ⁱⁱ | 25.1 (3) | N—C5—H5A | 119.00 |
| O4 ⁱⁱ —Pb—C7 ⁱⁱ | 24.2 (2) | C4—C5—H5A | 119.00 |
| Pb ^{iv} —O1—C6 | 89.5 (6) | | |
| N—Pb—O2—C6 | -27.3 (8) | O2—Pb—O4 ⁱⁱ —C7 ⁱⁱ | 93.1 (6) |
| N—Pb—O2—Pb ^{iv} | -155.1 (5) | N—Pb—O4 ⁱⁱ —C7 ⁱⁱ | 138.9 (6) |
| O1 ⁱ —Pb—O2—C6 | -169.0 (9) | O2—Pb—C7 ⁱⁱ —O3 ⁱⁱ | 41.3 (7) |
| O1 ⁱ —Pb—O2—Pb ^{iv} | 63.2 (4) | O2—Pb—C7 ⁱⁱ —O4 ⁱⁱ | -102.4 (5) |
| O2 ⁱ —Pb—O2—C6 | -129.5 (9) | O2—Pb—C7 ⁱⁱ —C2 ⁱⁱ | 144.2 (11) |
| O2 ⁱ —Pb—O2—Pb ^{iv} | 102.7 (4) | N—Pb—C7 ⁱⁱ —O3 ⁱⁱ | 102.9 (6) |
| O3 ⁱⁱⁱ —Pb—O2—C6 | -73.9 (10) | N—Pb—C7 ⁱⁱ —O4 ⁱⁱ | -40.7 (6) |
| O3 ⁱⁱⁱ —Pb—O2—Pb ^{iv} | 158.3 (4) | N—Pb—C7 ⁱⁱ —C2 ⁱⁱ | -154.2 (12) |
| O1 ⁱⁱ —Pb—O2—C6 | 126.9 (9) | Pb ^v —O1—C6—O2 | -131.7 (11) |
| O1 ⁱⁱ —Pb—O2—Pb ^{iv} | -0.9 (7) | Pb ^{iv} —O1—C6—C1 | -175.5 (10) |
| O3 ⁱⁱ —Pb—O2—C6 | 71.1 (9) | Pb ^{iv} —O1—C6—O2 | 3.8 (11) |
| O3 ⁱⁱ —Pb—O2—Pb ^{iv} | -56.7 (4) | Pb ^v —O1—C6—C1 | 49.0 (19) |
| O4 ⁱⁱ —Pb—O2—C6 | 25.9 (10) | Pb ^{iv} —O2—C6—C1 | 175.1 (8) |
| O4 ⁱⁱ —Pb—O2—Pb ^{iv} | -101.9 (4) | Pb—O2—C6—C1 | 36.0 (13) |
| C7 ⁱⁱ —Pb—O2—C6 | 54.5 (10) | Pb—O2—C6—O1 | -143.4 (9) |
| C7 ⁱⁱ —Pb—O2—Pb ^{iv} | -73.3 (5) | Pb ^{iv} —O2—C6—O1 | -4.2 (12) |
| O2—Pb—N—C1 | 15.4 (7) | Pb ^{vi} —O3—C7—O4 | 136.7 (11) |
| O2—Pb—N—C5 | -171.4 (9) | Pb ^v —O3—C7—O4 | -42.2 (10) |
| O1 ⁱ —Pb—N—C1 | 85.5 (8) | Pb ^{vi} —O3—C7—C2 | -43.1 (19) |
| O1 ⁱ —Pb—N—C5 | -101.3 (8) | Pb ^v —O3—C7—C2 | 138.0 (9) |
| O2 ⁱ —Pb—N—C1 | 83.0 (7) | Pb ^{vi} —O3—C7—Pb ^v | 178.9 (15) |
| O2 ⁱ —Pb—N—C5 | -103.8 (9) | Pb ^v —O4—C7—O3 | 40.7 (10) |

supplementary materials

| | | | |
|--|------------|---------------------------|-------------|
| O3 ⁱⁱⁱ —Pb—N—C1 | 157.4 (8) | Pb ^v —O4—C7—C2 | -139.5 (9) |
| O3 ⁱⁱⁱ —Pb—N—C5 | -29.4 (9) | C5—N—C1—C2 | 0.1 (16) |
| O1 ⁱⁱ —Pb—N—C1 | -142.1 (7) | Pb—N—C1—C2 | 173.5 (8) |
| O1 ⁱⁱ —Pb—N—C5 | 31.1 (10) | Pb—N—C1—C6 | -5.1 (12) |
| O3 ⁱⁱ —Pb—N—C1 | -80.8 (7) | C1—N—C5—C4 | 4.1 (17) |
| O3 ⁱⁱ —Pb—N—C5 | 92.4 (9) | C5—N—C1—C6 | -178.5 (10) |
| O4 ⁱⁱ —Pb—N—C1 | -121.6 (7) | Pb—N—C5—C4 | -169.0 (9) |
| O4 ⁱⁱ —Pb—N—C5 | 51.6 (8) | C6—C1—C2—C3 | 176.8 (10) |
| C7 ⁱⁱ —Pb—N—C1 | -105.8 (7) | N—C1—C2—C3 | -1.7 (16) |
| C7 ⁱⁱ —Pb—N—C5 | 67.4 (9) | N—C1—C2—C7 | 179.7 (10) |
| O2—Pb—O1 ⁱ —C6 ⁱ | 51.7 (7) | C6—C1—C2—C7 | -1.9 (17) |
| O2—Pb—O1 ⁱ —Pb ^{vii} | -150.3 (3) | N—C1—C6—O1 | 159.4 (10) |
| N—Pb—O1 ⁱ —C6 ⁱ | -5.4 (8) | N—C1—C6—O2 | -20.0 (15) |
| N—Pb—O1 ⁱ —Pb ^{vii} | 152.5 (3) | C2—C1—C6—O1 | -19.2 (17) |
| O2—Pb—O2 ⁱ —Pb ⁱ | 14.6 (4) | C2—C1—C6—O2 | 161.5 (10) |
| O2—Pb—O2 ⁱ —C6 ⁱ | -120.6 (7) | C1—C2—C3—C4 | -0.7 (17) |
| N—Pb—O2 ⁱ —Pb ⁱ | -44.8 (4) | C7—C2—C3—C4 | 177.9 (11) |
| N—Pb—O2 ⁱ —C6 ⁱ | 179.9 (7) | C1—C2—C7—O3 | -74.7 (15) |
| O2—Pb—O3 ⁱⁱⁱ —C7 ⁱⁱⁱ | 136.3 (13) | C1—C2—C7—O4 | 105.5 (13) |
| N—Pb—O3 ⁱⁱⁱ —C7 ⁱⁱⁱ | 95.1 (14) | C1—C2—C7—Pb ^v | 10 (2) |
| O2—Pb—O1 ⁱⁱ —Pb ^{vii} | 72.2 (6) | C3—C2—C7—O3 | 106.7 (13) |
| O2—Pb—O1 ⁱⁱ —C6 ⁱⁱ | -59.0 (14) | C3—C2—C7—O4 | -73.0 (15) |
| N—Pb—O1 ⁱⁱ —Pb ^{vii} | -149.1 (3) | C3—C2—C7—Pb ^v | -168.9 (8) |
| N—Pb—O1 ⁱⁱ —C6 ⁱⁱ | 79.8 (14) | C2—C3—C4—C5 | 4.5 (18) |
| O2—Pb—O3 ⁱⁱ —C7 ⁱⁱ | -144.8 (6) | C3—C4—C5—N | -6.3 (18) |
| N—Pb—O3 ⁱⁱ —C7 ⁱⁱ | -81.6 (6) | | |

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$; (ii) $x, -y+1/2, z+1/2$; (iii) $x, -y-1/2, z+1/2$; (iv) $-x+1, y+1/2, -z+3/2$; (v) $x, -y+1/2, z-1/2$; (vi) $x, -y-1/2, z-1/2$; (vii) $-x+1, -y, -z+2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|-------|-------------|-------------|---------------|
| C5—H5A \cdots O3 ⁱⁱⁱ | 0.93 | 2.57 | 3.164 (13) | 122 |

Symmetry codes: (iii) $x, -y-1/2, z+1/2$.

Fig. 1

