

catena-Poly[bis(μ_3 -3-aminobenzoato- κ^4 N:O:O,O')bis(μ_2 -3-aminobenzoato- κ^3 O,O':O)dilead(II)]

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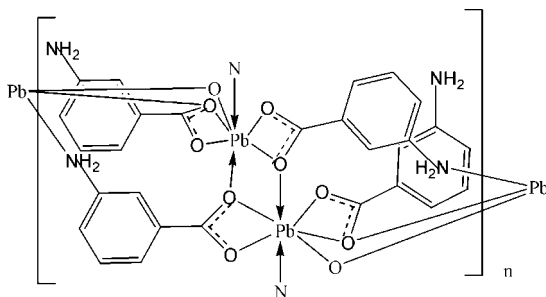
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.018$ Å; R factor = 0.065; wR factor = 0.169; data-to-parameter ratio = 13.0.

The Pb^{II} atom in the title compound, $[\text{Pb}_2(\text{C}_7\text{H}_6\text{NO}_2)_4]_n$, is chelated by two 3-aminobenzoato ligands in a distorted pentagonal-bipyramidal coordination geometry with five oxygen donors in the equatorial positions, one nitrogen donor and one oxygen donor in the axial positions. Two molecules are linked through a centre of inversion, forming a dinuclear entity. These entities are linked in a μ_3 -bridging mode through the amino N atom and two carboxylate O atoms into a chain along the b axis. Classical intermolecular N—H...O hydrogen bonding is observed in the structure. The supramolecular structure is consolidated by π - π stacking interactions with centroid-centroid distances between benzene rings of 3.837 (8) Å.

Related literature

For related structures, see: Tan *et al.* (2006); Wang *et al.* (2004, 2006); Wei *et al.* (2006).



Experimental

Crystal data

 $[\text{Pb}_2(\text{C}_7\text{H}_6\text{NO}_2)_4]$ $M_r = 958.92$

Triclinic, $P\bar{1}$
 $a = 6.8610$ (3) Å
 $b = 7.8943$ (3) Å
 $c = 13.9022$ (8) Å
 $\alpha = 76.030$ (2)°
 $\beta = 88.103$ (2)°
 $\gamma = 70.154$ (2)°

$V = 686.33$ (6) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 12.31$ mm⁻¹
 $T = 295$ K
 $0.18 \times 0.16 \times 0.02$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (SCALEPACK; Otwinowski & Minor, 1997)
 $T_{\min} = 0.215$, $T_{\max} = 0.791$

5451 measured reflections
 2470 independent reflections
 2218 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.103$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$
 $wR(F^2) = 0.169$
 $S = 1.05$
 2470 reflections
 190 parameters

12 restraints
 H-atom parameters constrained
 $\Delta\rho_{\max} = 3.23$ e Å⁻³
 $\Delta\rho_{\min} = -5.12$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O4}^i$	0.86	2.52	3.037 (12)	119
$\text{N2}-\text{H2B}\cdots\text{O1}^i$	0.86	2.32	2.936 (12)	129

Symmetry code: (i) $-x + 2, -y - 1, -z$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); 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: RK2229).

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supplementary materials

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F. M. Shen and S. F. Lush

Comment

In the last few years, much research on metal–organic framework has mostly focused on coordination polymer with rigid organic ligands containing either N– or O–atom donors, such as 3–aminobenzoic acid. Up to now, only few examples of coordination polymer with 3–aminobenzoic acid have been reported (Wang *et al.*, 2004, 2006; Tan *et al.*, 2006; Wei *et al.*, 2006.), and the polymeric complexes of non–transition metal with 3–aminobenzoic acid have not yet been found.

Herein we report the syntheses and crystal structure of Pb(II) coordination polymer with μ_3 –bridged 3–aminobenzoic acid (Fig.1). The title compound is a one–dimensional coordination polymer based on infinite μ_3 –bridging chains along *b* axial direction. The Pb···Pb distances are 4.118 (3)Å and 4.491 (3)Å. The Pb^{II} ion is a pentagonal–bipyramidal coordination environment and is coordinated by five carboxyl oxygen atoms comprise the equatorial plane, one nitrogen and one oxygen atom occupies the axial positions. The classic N—H···O hydrogen–bondings are observed in the structure (full details and symmetry codes shown as Table 1).

The supramolecular structure is also consolidated by π – π stacking interaction. The distance between *Cg*1 (C2–C7)···*Cg*2ⁱⁱ (C9–C14) is 3.842 (8)Å and dihedral angle between two benzene rings is 4.6 (7)°. Symmetry code: (ii) 1–*x*, –*y*, –*y*.

Experimental

A solution of 3–aminobenzoic acid (0.0683 g, 0.50 mmol) in 5 ml deionized water was slowly added to a stirring solution of Pb(NO₃)₂·6H₂O (0.1670 g, 0.50 mmol) and 1,2–bis(4–pyridyl)ethane (0.0926 g, 0.50 mmol) in 5 ml deionized water. The mixture solution was seal in 25 ml stainless steel reactor with a teflon liner, heat to 423 K for 24 hr, and then slowly cooled to room temperature. The pink single crystals of the title compound were obtained in 56.45% yield. (Based on Pb).

Refinement

H atoms were positioned geometrically N—H = 0.86Å and C—H = 0.93Å (aromatic), and were refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C},\text{N})$.

Figures

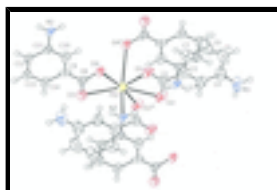


Fig. 1. View of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

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Crystal data

[Pb ₂ (C ₇ H ₆ NO ₂) ₄]	$Z = 1$
$M_r = 958.92$	$F(000) = 448$
Triclinic, $P\bar{1}$	$D_x = 2.320 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 6.8610 (3) \text{ \AA}$	Cell parameters from 12232 reflections
$b = 7.8943 (3) \text{ \AA}$	$\theta = 2.0\text{--}25.4^\circ$
$c = 13.9022 (8) \text{ \AA}$	$\mu = 12.31 \text{ mm}^{-1}$
$\alpha = 76.030 (2)^\circ$	$T = 295 \text{ K}$
$\beta = 88.103 (2)^\circ$	Prism, pink
$\gamma = 70.154 (2)^\circ$	$0.18 \times 0.16 \times 0.02 \text{ mm}$
$V = 686.33 (6) \text{ \AA}^3$	

Data collection

Nonius KappaCCD diffractometer	2470 independent reflections
Radiation source: fine-focus sealed tube graphite	2218 reflections with $I > 2\sigma(I)$
Detector resolution: 9 pixels mm^{-1}	$R_{\text{int}} = 0.103$
CCD rotation images, thick slices scans	$\theta_{\text{max}} = 25.3^\circ$, $\theta_{\text{min}} = 2.8^\circ$
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	$h = -7 \rightarrow 8$
$T_{\text{min}} = 0.215$, $T_{\text{max}} = 0.791$	$k = -7 \rightarrow 9$
5451 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.065$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.169$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.1239P)^2 + 1.1995P]$
2470 reflections	where $P = (F_o^2 + 2F_c^2)/3$
190 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
12 restraints	$\Delta\rho_{\text{max}} = 3.23 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -5.12 \text{ e \AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness 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 threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *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.

The residual peaks show two relatively high peaks of $3.19 \text{ e}\text{\AA}^{-3}$ at 0.405, 0.645, 0.123 and $3.13 \text{ e}\text{\AA}^{-3}$ at 0.566, 0.080, 0.038, and the distances to the nearest Pb atoms are 1.26Å and 1.50Å, respectively.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.57722 (5)	0.23064 (4)	-0.04961 (3)	0.0375 (1)
O1	0.7342 (13)	0.0825 (10)	0.1159 (6)	0.051 (3)
O2	0.6749 (13)	0.3823 (11)	0.0817 (6)	0.051 (3)
O3	0.6695 (17)	0.0912 (11)	-0.2133 (6)	0.058 (3)
O4	0.7229 (12)	-0.0859 (9)	-0.0620 (5)	0.040 (2)
N1	0.655 (3)	0.555 (2)	0.4054 (11)	0.092 (6)
N2	0.9270 (14)	-0.7459 (12)	-0.0976 (7)	0.038 (3)
C1	0.7141 (15)	0.2286 (15)	0.1435 (8)	0.037 (3)
C2	0.7253 (16)	0.2209 (16)	0.2521 (9)	0.043 (3)
C3	0.6951 (19)	0.3817 (19)	0.2811 (9)	0.054 (4)
C4	0.696 (2)	0.383 (2)	0.3805 (10)	0.065 (4)
C5	0.733 (2)	0.212 (3)	0.4500 (10)	0.076 (5)
C6	0.768 (3)	0.050 (3)	0.4203 (11)	0.079 (5)
C7	0.766 (2)	0.056 (2)	0.3199 (10)	0.057 (4)
C8	0.7220 (17)	-0.0662 (14)	-0.1556 (8)	0.039 (3)
C9	0.7796 (14)	-0.2349 (13)	-0.1927 (8)	0.035 (3)
C10	0.8350 (14)	-0.4092 (14)	-0.1314 (8)	0.035 (3)
C11	0.8749 (15)	-0.5654 (13)	-0.1674 (7)	0.029 (3)
C12	0.8668 (17)	-0.5505 (15)	-0.2690 (8)	0.037 (3)
C13	0.8113 (19)	-0.3757 (16)	-0.3344 (8)	0.043 (3)
C14	0.7646 (16)	-0.2166 (14)	-0.2971 (8)	0.040 (3)
H1A	0.63110	0.65490	0.35920	0.1100*
H1B	0.65500	0.55860	0.46660	0.1100*
H2A	0.93270	-0.75440	-0.03490	0.0460*
H2B	0.95220	-0.84430	-0.11910	0.0460*
H3	0.67370	0.49200	0.23310	0.0660*
H5	0.73270	0.20860	0.51740	0.0920*
H6	0.79390	-0.06200	0.46730	0.0940*
H7	0.79250	-0.05300	0.29910	0.0690*
H10	0.84610	-0.42310	-0.06320	0.0420*

supplementary materials

H12	0.89830	-0.65640	-0.29310	0.0450*
H13	0.80500	-0.36400	-0.40250	0.0510*
H14	0.72400	-0.09940	-0.34050	0.0480*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0414 (2)	0.0231 (2)	0.0487 (3)	-0.0120 (2)	0.0023 (2)	-0.0088 (2)
O1	0.056 (5)	0.034 (4)	0.065 (5)	-0.015 (4)	0.007 (4)	-0.019 (4)
O2	0.061 (5)	0.035 (4)	0.059 (5)	-0.020 (4)	-0.004 (4)	-0.010 (4)
O3	0.094 (6)	0.034 (4)	0.051 (5)	-0.028 (4)	0.010 (4)	-0.013 (4)
O4	0.058 (5)	0.023 (3)	0.043 (4)	-0.013 (3)	0.007 (3)	-0.015 (3)
N1	0.113 (11)	0.118 (11)	0.067 (7)	-0.049 (10)	0.027 (7)	-0.054 (8)
N2	0.046 (5)	0.026 (4)	0.048 (5)	-0.016 (4)	0.005 (4)	-0.014 (4)
C1	0.027 (4)	0.038 (5)	0.046 (5)	-0.009 (4)	0.010 (4)	-0.014 (4)
C2	0.029 (5)	0.046 (6)	0.051 (6)	-0.009 (4)	0.004 (4)	-0.015 (4)
C3	0.050 (6)	0.063 (7)	0.055 (6)	-0.022 (6)	-0.003 (5)	-0.019 (6)
C4	0.044 (7)	0.091 (8)	0.066 (7)	-0.015 (7)	0.012 (6)	-0.041 (6)
C5	0.065 (8)	0.123 (11)	0.044 (6)	-0.032 (9)	0.013 (6)	-0.027 (6)
C6	0.081 (10)	0.080 (9)	0.053 (7)	-0.010 (9)	0.003 (7)	-0.001 (7)
C7	0.048 (7)	0.053 (7)	0.059 (7)	-0.008 (6)	0.008 (6)	-0.006 (6)
C8	0.046 (5)	0.023 (5)	0.053 (6)	-0.018 (4)	0.006 (5)	-0.012 (4)
C9	0.025 (4)	0.022 (4)	0.056 (6)	-0.011 (4)	0.007 (4)	0.000 (4)
C10	0.021 (4)	0.030 (5)	0.049 (5)	0.003 (4)	-0.002 (4)	-0.018 (4)
C11	0.027 (4)	0.024 (4)	0.042 (5)	-0.013 (4)	0.007 (4)	-0.013 (4)
C12	0.048 (6)	0.036 (5)	0.041 (5)	-0.024 (4)	0.012 (4)	-0.021 (4)
C13	0.058 (6)	0.040 (6)	0.038 (5)	-0.024 (5)	0.005 (5)	-0.014 (4)
C14	0.044 (5)	0.029 (5)	0.050 (6)	-0.016 (4)	0.003 (5)	-0.010 (4)

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

Pb1—O1	2.415 (8)	C3—C4	1.385 (18)
Pb1—O2	2.626 (8)	C4—C5	1.41 (2)
Pb1—O3	2.726 (8)	C5—C6	1.38 (3)
Pb1—O4	2.403 (7)	C6—C7	1.39 (2)
Pb1—N2 ⁱ	2.525 (10)	C8—C9	1.469 (15)
Pb1—O4 ⁱⁱ	2.913 (8)	C9—C10	1.365 (15)
Pb1—O2 ⁱⁱⁱ	2.887 (8)	C9—C14	1.427 (15)
O1—C1	1.264 (14)	C10—C11	1.380 (15)
O2—C1	1.254 (14)	C11—C12	1.390 (14)
O3—C8	1.246 (14)	C12—C13	1.392 (16)
O4—C8	1.273 (13)	C13—C14	1.407 (16)
N1—C4	1.42 (2)	C3—H3	0.9300
N2—C11	1.452 (13)	C5—H5	0.9300
N1—H1B	0.8600	C6—H6	0.9300
N1—H1A	0.8600	C7—H7	0.9300
N2—H2B	0.8600	C10—H10	0.9300
N2—H2A	0.8600	C12—H12	0.9300

C1—C2	1.500 (16)	C13—H13	0.9300
C2—C7	1.356 (19)	C14—H14	0.9300
C2—C3	1.371 (19)		
Pb1...C3 ⁱⁱⁱ	3.870 (13)	C3...C13 ⁱⁱ	3.542 (19)
Pb1...C10 ⁱⁱ	3.854 (11)	C3...C14 ^{vi}	3.482 (18)
Pb1...H3 ⁱⁱⁱ	3.0500	C4...C13 ^{vi}	3.40 (2)
Pb1...H10 ⁱⁱ	3.3300	C4...C13 ⁱⁱ	3.58 (2)
O1...O2	2.193 (11)	C7...C14 ⁱⁱ	3.422 (19)
O1...O4	3.092 (11)	C8...Pb1 ⁱⁱ	3.698 (11)
O1...C2	2.394 (15)	C9...C1 ⁱⁱ	3.419 (15)
O1...N2 ^{iv}	2.936 (12)	C9...C1 ^{vi}	3.582 (15)
O1...C8 ⁱⁱ	3.200 (15)	C9...C2 ^{vi}	3.501 (16)
O1...O4 ⁱⁱ	3.238 (13)	C9...C2 ⁱⁱ	3.547 (16)
O2...N2 ⁱ	3.158 (13)	C10...O2 ^v	3.369 (14)
O2...C2	2.378 (15)	C10...Pb1 ⁱⁱ	3.854 (11)
O2...O1	2.193 (11)	C10...C1 ⁱⁱ	3.540 (15)
O2...O2 ⁱⁱⁱ	3.109 (12)	C13...C3 ⁱⁱ	3.542 (19)
O2...C10 ⁱ	3.369 (14)	C13...C3 ^{vi}	3.473 (19)
O3...C9	2.370 (13)	C13...C4 ^{vi}	3.40 (2)
O3...O4	2.190 (11)	C13...C4 ⁱⁱ	3.58 (2)
O3...N2 ⁱ	3.177 (14)	C14...C3 ^{vi}	3.482 (18)
O4...O1	3.092 (11)	C14...C2 ^{vi}	3.563 (17)
O4...C9	2.348 (12)	C14...C2 ⁱⁱ	3.409 (17)
O4...O1 ⁱⁱ	3.238 (13)	C14...C7 ⁱⁱ	3.422 (19)
O4...N2 ^{iv}	3.037 (12)	C1...H2A ⁱ	2.8500
O4...O3	2.190 (11)	C5...H1B ^{vii}	3.0400
O1...H7	2.5000	C8...H2B ⁱ	2.8600
O1...H2B ^{iv}	2.3200	H1A...H3	2.3700
O1...H2A ⁱ	2.7600	H1A...O3 ⁱⁱⁱ	2.8100
O2...H10 ⁱ	2.7200	H1B...H5	2.5500
O2...H2A ⁱ	2.5100	H1B...H13 ^{viii}	2.4100
O2...H3	2.4600	H1B...C5 ^{vii}	3.0400
O3...H2B ⁱ	2.6300	H2A...H10	2.4100
O3...H1A ⁱⁱⁱ	2.8100	H2A...O4 ^{iv}	2.5200
O3...H14	2.5300	H2B...H12	2.4800
O4...H10	2.5100	H2B...O1 ^{iv}	2.3200
O4...H2B ⁱ	2.8200	H3...O2	2.4600
O4...H2A ^{iv}	2.5200	H3...H1A	2.3700
N2...O2 ^v	3.158 (13)	H3...Pb1 ⁱⁱⁱ	3.0500
N2...O3 ^v	3.177 (14)	H5...H1B	2.5500
N2...O1 ^{iv}	2.936 (12)	H7...O1	2.5000

supplementary materials

N2...O4 ^{iv}	3.037 (12)	H7...H12 ^{iv}	2.5600
C1...C10 ⁱⁱ	3.540 (15)	H10...O2 ^v	2.7200
C1...C9 ⁱⁱ	3.419 (15)	H10...O4	2.5100
C1...C9 ^{vi}	3.582 (15)	H10...H2A	2.4100
C2...C14 ^{vi}	3.563 (17)	H10...Pb1 ⁱⁱ	3.3300
C2...C9 ⁱⁱ	3.547 (16)	H10...H10 ^{iv}	2.5500
C2...C9 ^{vi}	3.501 (16)	H12...H2B	2.4800
C2...C14 ⁱⁱ	3.409 (17)	H12...H7 ^{iv}	2.5600
C3...C13 ^{vi}	3.473 (19)	H13...H1B ^{ix}	2.4100
C3...Pb1 ⁱⁱⁱ	3.870 (13)	H14...O3	2.5300
O1—Pb1—O2	51.4 (3)	H2A—N2—H2B	120.00
O1—Pb1—O3	126.6 (3)	Pb1 ^v —N2—H2B	88.00
O1—Pb1—O4	79.9 (2)	Pb1—C1—O1	56.6 (6)
O1—Pb1—C1	25.9 (3)	O1—C1—O2	121.1 (10)
O1—Pb1—C8	103.8 (3)	Pb1—C1—O2	66.2 (6)
O1—Pb1—N2 ⁱ	86.3 (3)	Pb1—C1—C2	163.1 (8)
O1—Pb1—O4 ⁱⁱ	74.2 (3)	O1—C1—C2	119.7 (10)
O1—Pb1—O2 ⁱⁱⁱ	115.3 (2)	O2—C1—C2	119.1 (10)
O2—Pb1—O3	149.9 (3)	C3—C2—C7	121.1 (12)
O2—Pb1—O4	128.1 (3)	C1—C2—C3	119.1 (11)
O2—Pb1—C1	25.9 (3)	C1—C2—C7	119.8 (11)
O2—Pb1—C8	145.9 (3)	C2—C3—C4	121.3 (13)
O2—Pb1—N2 ⁱ	75.6 (3)	N1—C4—C5	124.5 (14)
O2—Pb1—O4 ⁱⁱ	98.7 (2)	C3—C4—C5	117.1 (14)
O2—Pb1—O2 ⁱⁱⁱ	68.5 (3)	N1—C4—C3	118.4 (13)
O3—Pb1—O4	50.1 (2)	C4—C5—C6	121.4 (13)
O3—Pb1—C1	147.2 (3)	C5—C6—C7	119.4 (16)
O3—Pb1—C8	25.0 (3)	C2—C7—C6	119.8 (15)
O3—Pb1—N2 ⁱ	74.3 (3)	Pb1—C8—O3	67.9 (6)
O3—Pb1—O4 ⁱⁱ	109.5 (3)	Pb1—C8—O4	53.2 (5)
O2 ⁱⁱⁱ —Pb1—O3	117.1 (2)	Pb1—C8—C9	169.2 (8)
O4—Pb1—C1	105.3 (3)	O3—C8—O4	120.9 (10)
O4—Pb1—C8	25.1 (3)	O3—C8—C9	121.5 (10)
O4—Pb1—N2 ⁱ	85.9 (3)	O4—C8—C9	117.7 (9)
O4—Pb1—O4 ⁱⁱ	81.3 (2)	C8—C9—C10	122.9 (10)
O2 ⁱⁱⁱ —Pb1—O4	163.4 (2)	C8—C9—C14	118.9 (9)
C1—Pb1—C8	128.2 (3)	C10—C9—C14	118.1 (9)
N2 ⁱ —Pb1—C1	83.5 (3)	C9—C10—C11	122.1 (10)
O4 ⁱⁱ —Pb1—C1	83.0 (3)	N2—C11—C12	120.4 (9)
O2 ⁱⁱⁱ —Pb1—C1	90.5 (3)	C10—C11—C12	120.5 (10)
N2 ⁱ —Pb1—C8	80.2 (3)	N2—C11—C10	119.0 (9)
O4 ⁱⁱ —Pb1—C8	94.8 (3)	C11—C12—C13	119.4 (10)
O2 ⁱⁱⁱ —Pb1—C8	140.9 (3)	C12—C13—C14	119.8 (10)

O4 ⁱⁱ —Pb1—N2 ⁱ	158.2 (3)	C9—C14—C13	120.1 (10)
O2 ⁱⁱⁱ —Pb1—N2 ⁱ	101.2 (3)	C2—C3—H3	119.00
O2 ⁱⁱⁱ —Pb1—O4 ⁱⁱ	96.0 (2)	C4—C3—H3	119.00
Pb1—O1—C1	97.5 (7)	C4—C5—H5	119.00
Pb1—O2—C1	87.9 (7)	C6—C5—H5	120.00
Pb1—O2—Pb1 ⁱⁱⁱ	111.5 (3)	C5—C6—H6	120.00
Pb1 ⁱⁱⁱ —O2—C1	142.0 (7)	C7—C6—H6	120.00
Pb1—O3—C8	87.1 (6)	C2—C7—H7	120.00
Pb1—O4—C8	101.8 (6)	C6—C7—H7	120.00
Pb1—O4—Pb1 ⁱⁱ	98.7 (3)	C9—C10—H10	119.00
Pb1 ⁱⁱ —O4—C8	118.8 (7)	C11—C10—H10	119.00
Pb1 ^v —N2—C11	103.4 (7)	C11—C12—H12	120.00
H1A—N1—H1B	120.00	C13—C12—H12	120.00
C4—N1—H1A	120.00	C12—C13—H13	120.00
C4—N1—H1B	120.00	C14—C13—H13	120.00
Pb1 ^v —N2—H2A	78.00	C9—C14—H14	120.00
C11—N2—H2A	120.00	C13—C14—H14	120.00
C11—N2—H2B	120.00		
O2—Pb1—O1—C1	8.3 (6)	O2—Pb1—N2 ⁱ —C11 ⁱ	-90.7 (6)
O3—Pb1—O1—C1	150.1 (7)	O3—Pb1—N2 ⁱ —C11 ⁱ	88.7 (6)
O4—Pb1—O1—C1	169.3 (7)	O4—Pb1—N2 ⁱ —C11 ⁱ	138.3 (6)
C8—Pb1—O1—C1	161.8 (7)	C1—Pb1—N2 ⁱ —C11 ⁱ	-115.8 (6)
N2 ⁱ —Pb1—O1—C1	82.8 (7)	C8—Pb1—N2 ⁱ —C11 ⁱ	113.6 (6)
O4 ⁱⁱ —Pb1—O1—C1	-107.0 (7)	O1—Pb1—O4 ⁱⁱ —Pb1 ⁱⁱ	-81.9 (3)
O2 ⁱⁱⁱ —Pb1—O1—C1	-17.8 (8)	O1—Pb1—O4 ⁱⁱ —C8 ⁱⁱ	26.8 (7)
O1—Pb1—O2—C1	-8.3 (6)	O2—Pb1—O4 ⁱⁱ —Pb1 ⁱⁱ	-127.4 (3)
O1—Pb1—O2—Pb1 ⁱⁱⁱ	-154.7 (5)	O2—Pb1—O4 ⁱⁱ —C8 ⁱⁱ	-18.8 (8)
O3—Pb1—O2—C1	-106.4 (8)	O3—Pb1—O4 ⁱⁱ —Pb1 ⁱⁱ	42.0 (3)
O3—Pb1—O2—Pb1 ⁱⁱⁱ	107.2 (5)	O3—Pb1—O4 ⁱⁱ —C8 ⁱⁱ	150.6 (7)
O4—Pb1—O2—C1	-32.3 (8)	O4—Pb1—O4 ⁱⁱ —Pb1 ⁱⁱ	0.0 (2)
O4—Pb1—O2—Pb1 ⁱⁱⁱ	-178.7 (3)	O4—Pb1—O4 ⁱⁱ —C8 ⁱⁱ	108.6 (7)
C1—Pb1—O2—Pb1 ⁱⁱⁱ	-146.4 (8)	C1—Pb1—O4 ⁱⁱ —Pb1 ⁱⁱ	-106.8 (3)
C8—Pb1—O2—C1	-58.8 (9)	C1—Pb1—O4 ⁱⁱ —C8 ⁱⁱ	1.9 (7)
C8—Pb1—O2—Pb1 ⁱⁱⁱ	154.7 (4)	C8—Pb1—O4 ⁱⁱ —Pb1 ⁱⁱ	21.2 (3)
N2 ⁱ —Pb1—O2—C1	-105.1 (7)	C8—Pb1—O4 ⁱⁱ —C8 ⁱⁱ	129.8 (7)
N2 ⁱ —Pb1—O2—Pb1 ⁱⁱⁱ	108.5 (4)	O1—Pb1—O2 ⁱⁱⁱ —Pb1 ⁱⁱⁱ	21.7 (4)
O4 ⁱⁱ —Pb1—O2—C1	53.4 (7)	O1—Pb1—O2 ⁱⁱⁱ —C1 ⁱⁱⁱ	137.8 (12)
O4 ⁱⁱ —Pb1—O2—Pb1 ⁱⁱⁱ	-93.1 (3)	O2—Pb1—O2 ⁱⁱⁱ —Pb1 ⁱⁱⁱ	0.0 (3)
O2 ⁱⁱⁱ —Pb1—O2—C1	146.4 (7)	O2—Pb1—O2 ⁱⁱⁱ —C1 ⁱⁱⁱ	116.1 (13)
O2 ⁱⁱⁱ —Pb1—O2—Pb1 ⁱⁱⁱ	0.0 (3)	O3—Pb1—O2 ⁱⁱⁱ —Pb1 ⁱⁱⁱ	-147.5 (3)
O1—Pb1—O3—C8	27.6 (9)	O3—Pb1—O2 ⁱⁱⁱ —C1 ⁱⁱⁱ	-31.4 (13)
O2—Pb1—O3—C8	101.9 (9)	C1—Pb1—O2 ⁱⁱⁱ —Pb1 ⁱⁱⁱ	14.0 (4)

supplementary materials

O4—Pb1—O3—C8	2.7 (7)	C1—Pb1—O2 ⁱⁱⁱ —C1 ⁱⁱⁱ	130.1 (12)
C1—Pb1—O3—C8	51.3 (11)	C8—Pb1—O2 ⁱⁱⁱ —Pb1 ⁱⁱⁱ	-157.7 (4)
N2 ⁱ —Pb1—O3—C8	100.6 (8)	C8—Pb1—O2 ⁱⁱⁱ —C1 ⁱⁱⁱ	-41.6 (14)
O4 ⁱⁱ —Pb1—O3—C8	-56.9 (8)	Pb1—O1—C1—O2	-16.0 (12)
O2 ⁱⁱⁱ —Pb1—O3—C8	-164.6 (7)	Pb1—O1—C1—C2	160.6 (9)
O1—Pb1—O4—C8	-162.6 (8)	Pb1—O2—C1—O1	14.5 (11)
O1—Pb1—O4—Pb1 ⁱⁱ	75.4 (3)	Pb1—O2—C1—C2	-162.0 (9)
O2—Pb1—O4—C8	-143.8 (7)	Pb1 ⁱⁱⁱ —O2—C1—Pb1	123.3 (11)
O2—Pb1—O4—Pb1 ⁱⁱ	94.2 (3)	Pb1 ⁱⁱⁱ —O2—C1—O1	137.8 (10)
O3—Pb1—O4—C8	-2.7 (7)	Pb1 ⁱⁱⁱ —O2—C1—C2	-38.8 (18)
O3—Pb1—O4—Pb1 ⁱⁱ	-124.7 (5)	Pb1—O3—C8—O4	-4.6 (12)
C1—Pb1—O4—C8	-157.8 (7)	Pb1—O3—C8—C9	173.8 (11)
C1—Pb1—O4—Pb1 ⁱⁱ	80.2 (3)	Pb1—O4—C8—O3	5.3 (14)
C8—Pb1—O4—Pb1 ⁱⁱ	-122.0 (8)	Pb1—O4—C8—C9	-173.1 (8)
N2 ⁱ —Pb1—O4—C8	-75.7 (7)	Pb1 ⁱⁱ —O4—C8—Pb1	106.9 (6)
N2 ⁱ —Pb1—O4—Pb1 ⁱⁱ	162.3 (3)	Pb1 ⁱⁱ —O4—C8—O3	112.2 (12)
O4 ⁱⁱ —Pb1—O4—C8	122.0 (7)	Pb1 ⁱⁱ —O4—C8—C9	-66.2 (12)
O4 ⁱⁱ —Pb1—O4—Pb1 ⁱⁱ	0.0 (2)	Pb1 ^v —N2—C11—C12	-96.2 (10)
O1—Pb1—C1—O2	165.1 (11)	Pb1 ^v —N2—C11—C10	84.2 (10)
O2—Pb1—C1—O1	-165.1 (11)	O1—C1—C2—C7	2.9 (17)
O3—Pb1—C1—O1	-47.5 (10)	O1—C1—C2—C3	-177.5 (12)
O3—Pb1—C1—O2	117.6 (7)	O2—C1—C2—C7	179.5 (12)
O4—Pb1—C1—O1	-11.0 (7)	O2—C1—C2—C3	-0.9 (17)
O4—Pb1—C1—O2	154.1 (6)	C7—C2—C3—C4	-3(2)
C8—Pb1—C1—O1	-22.7 (8)	C1—C2—C3—C4	177.5 (12)
C8—Pb1—C1—O2	142.4 (6)	C1—C2—C7—C6	-177.4 (15)
N2 ⁱ —Pb1—C1—O1	-94.8 (7)	C3—C2—C7—C6	3(2)
N2 ⁱ —Pb1—C1—O2	70.3 (7)	C2—C3—C4—N1	-177.3 (15)
O4 ⁱⁱ —Pb1—C1—O1	68.0 (7)	C2—C3—C4—C5	1(2)
O4 ⁱⁱ —Pb1—C1—O2	-126.9 (7)	N1—C4—C5—C6	178.7 (18)
O2 ⁱⁱⁱ —Pb1—C1—O1	163.9 (7)	C3—C4—C5—C6	0(2)
O2 ⁱⁱⁱ —Pb1—C1—O2	-31.0 (7)	C4—C5—C6—C7	0(3)
O1—Pb1—C8—O3	-157.5 (8)	C5—C6—C7—C2	-2(3)
O1—Pb1—C8—O4	17.6 (8)	O4—C8—C9—C10	-0.6 (17)
O2—Pb1—C8—O3	-119.1 (8)	O3—C8—C9—C10	-179.1 (12)
O2—Pb1—C8—O4	56.0 (10)	O3—C8—C9—C14	-3.2 (17)
O3—Pb1—C8—O4	175.1 (13)	O4—C8—C9—C14	175.2 (10)
O4—Pb1—C8—O3	-175.1 (13)	C14—C9—C10—C11	-0.4 (16)
C1—Pb1—C8—O3	-147.5 (8)	C8—C9—C14—C13	-177.6 (11)
C1—Pb1—C8—O4	27.6 (9)	C8—C9—C10—C11	175.5 (10)
N2 ⁱ —Pb1—C8—O3	-73.8 (8)	C10—C9—C14—C13	-1.6 (16)
N2 ⁱ —Pb1—C8—O4	101.3 (7)	C9—C10—C11—C12	2.3 (17)
O4 ⁱⁱ —Pb1—C8—O3	127.6 (8)	C9—C10—C11—N2	-178.2 (10)

O4 ⁱⁱ —Pb1—C8—O4	-57.3 (7)	N2—C11—C12—C13	178.4 (11)
O2 ⁱⁱⁱ —Pb1—C8—O3	22.0 (10)	C10—C11—C12—C13	-2.1 (18)
O2 ⁱⁱⁱ —Pb1—C8—O4	-162.9 (6)	C11—C12—C13—C14	0.1 (19)
O1—Pb1—N2 ⁱ —C11 ⁱ	-141.7 (6)	C12—C13—C14—C9	1.7 (18)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A \cdots O4 ^{iv}	0.86	2.52	3.037 (12)	119
N2—H2B \cdots O1 ^{iv}	0.86	2.32	2.936 (12)	129

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

Fig. 1

