

Poly[bis(μ_3 -acetato- $\kappa^4 O, O':O:O'$)bis(μ_2 -acetato- $\kappa^3 O, O':O$)(μ_2 -2,5-dimethylbenzene-1,4-diol- $\kappa^2 O:O'$)dilead(II)]

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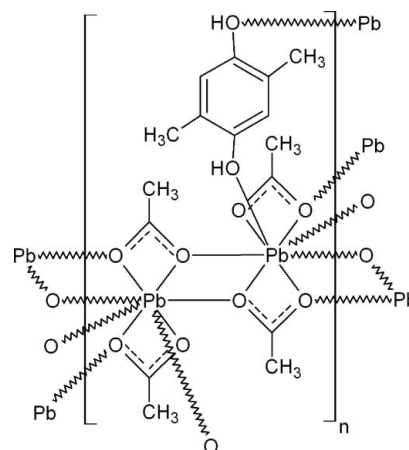
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(C-C) = 0.006$ Å; R factor = 0.017; wR factor = 0.043; data-to-parameter ratio = 18.4.

The title compound, $[\text{Pb}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_8\text{H}_{10}\text{O}_2)]_n$, has a polymeric structure, with acetatolead(II) chains and 2,5-dimethylbenzene-1,4-diol molecules forming bridges between two Pb^{II} ions from neighbouring chains. Each Pb^{II} centre is surrounded by eight O atoms; four belong to bidentate acetate ions, three to neighbouring bridging acetate groups and one to the 2,5-dimethylbenzene-1,4-diol molecule. The Pb^{II} ions are chelated symmetrically and asymmetrically by acetate ligands. The coordination environment of the Pb^{II} ion can be described as a hemidirected $\text{Pb}^{\text{II}}\text{O}_6$ core with the empty space around the metal ion filled by the stereochemically active $6s^2$ electron pair and two longer Pb—O contacts. The Pb—O distances are in the range of 2.355 (3)–2.994 (3) Å. Additionally, the crystal structure is stabilized by O—H...O hydrogen bonds.

Related literature

Other crystal structures containing polymeric lead(II) acetate were reported by: Rajaram & Rao (1982); Bryant *et al.* (1984); Harrowfield *et al.* (1996). Van der Waals radii of lead(II) and oxygen were presented by Bondi (1964). The synthesis of the title compound was carried out similarly to the method used for obtaining the bis(2,4-pentanedionato)lead(II) complex described by Lyczko *et al.* (2006). Hemi- and holodirected geometries of lead(II) complexes and the role of the $6s^2$ lone electron pair of the lead(II) ion were discussed by Shimoni-Livny *et al.* (1998).



Experimental

Crystal data

$[\text{Pb}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_8\text{H}_{10}\text{O}_2)]$
 $M_r = 788.72$
 Triclinic, $P\bar{1}$
 $a = 7.4905$ (11) Å
 $b = 7.5526$ (10) Å
 $c = 10.2522$ (15) Å
 $\alpha = 93.043$ (11)°
 $\beta = 100.787$ (12)°

$\gamma = 116.377$ (14)°
 $V = 504.28$ (12) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 16.72$ mm⁻¹
 $T = 100$ (2) K
 $0.35 \times 0.26 \times 0.14$ mm

Data collection

Kuma KM-4 four-circle CCD diffractometer
 Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2005)
 $T_{\text{min}} = 0.040$, $T_{\text{max}} = 0.227$

9065 measured reflections
 2432 independent reflections
 2317 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$
 $wR(F^2) = 0.043$
 $S = 1.07$
 2432 reflections

132 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.67$ e Å⁻³

Table 1

Selected bond lengths (Å).

Pb1—O3	2.355 (3)	Pb1—O4	2.700 (3)
Pb1—O1	2.493 (3)	Pb1—O2 ⁱⁱ	2.767 (3)
Pb1—O2	2.499 (3)	Pb1—O5	2.993 (3)
Pb1—O1 ⁱ	2.618 (2)	Pb1—O4 ⁱⁱⁱ	2.994 (3)

Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $-x, -y + 1, -z$; (iii) $-x, -y, -z$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O5}-\text{H5}\cdots\text{O3}^{\text{i}}$	0.84	1.87	2.668 (4)	159

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduc-

tion: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2114).

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supporting information

Acta Cryst. (2008). E64, m1341–m1342 [doi:10.1107/S1600536808030535]

Poly[bis(μ_3 -acetato- κ^4 O,O':O:O')bis(μ_2 -acetato- κ^3 O,O':O)(μ_2 -2,5-dimethylbenzene-1,4-diol- κ^2 O:O')dilead(II)]

Krzysztof Lyczko and Joanna Bak

S1. Comment

The aim of this work was to synthesize the complex between Pb^{II} ions and 3-methyl-2,4-pentanedione. Unexpectedly, under the reaction conditions (see *Experimental*), instead of the bis(3-methyl-2,4-pentanedionato)lead(II) complex, the title compound was obtained.

The structure of this compound consists of polymeric chains of lead(II) acetate linked *via* 2,5-dimethylbenzene-1,4-diol molecules, each bridging two Pb^{II} ions from neighbouring chains. As shown in Fig. 1 the title compound contains eight-coordinated Pb^{II} ions. Each Pb^{II} centre binds strongly four O atoms from bidentate acetate ions in a nearly pyramidal configuration with Pb—O bond lengths in the range of 2.355 (3)–2.700 (3) Å, and additionally two O atoms from neighbouring bridging acetate groups of the same chain at 2.618 (2) and 2.766 (3) Å. The Pb^{II} ions are chelated symmetrically [Pb—O = 2.493 (3) and 2.499 (3) Å] and asymmetrically [2.355 (3) and 2.700 (3) Å] by acetate ligands. The coordination sphere of the metal ion is completed by two additional O atoms at distances of about 2.99 Å, the first from the acetate group of the neighbouring chain and the second from the 2,5-dimethylbenzene-1,4-diol molecule. These two contacts are much longer than the Pb—O bonds in the chelate but shorter than the sum of the van der Waals radii of lead(II) and oxygen (3.44 Å; Bondi, 1964) indicating some weak interactions. The crystal structure is also stabilized by O—H \cdots O hydrogen bonds. The OH groups of the 2,5-dimethylbenzene-1,4-diol molecule form hydrogen bonds with a H \cdots O distance of 1.87 Å to O atoms from neighbouring CH₃COO⁻ ions (Fig. 2). The resulting coordination sphere of the Pb^{II} ion in the title compound has a hemidirected geometry (Shimoni-Livny *et al.*, 1998). It contains the Pb^{II}O₆ core with the empty space at one side of the metal ion filled by stereochemically active electron pair and two longer Pb—O contacts of 2.993 (3) and 2.994 (3) Å. The 6s² electron pair is located opposite to the shortest Pb—O bond length [2.355 (3) Å] and between two longest Pb—O contacts of about 2.99 Å.

Identical polymeric acetato-lead(II) chains are present in the lead(II) acetate trihydrate structure, where the metal ions are nine-coordinated (Pb^{II}O₉) with two bidentate acetate groups, three coordinated water molecules and two bridging distances to O atoms from the neighbouring acetate groups (Rajaram & Rao, 1982; Bryant *et al.*, 1984). In another polymeric structure of lead(II) acetate the Pb^{II} ions are eight-coordinated with the Pb^{II}O₆N₂ core containing in addition two N atoms from two 2-pyridylamine molecules (Harrowfield *et al.*, 1996).

S2. Experimental

The title compound was obtained unintentionally in an attempt to synthesize single crystals of the bis(3-methyl-2,4-pentanedionato)lead(II) complex. Small pieces of lead foil (0.26 g) were heated (~50 °C) for a few weeks in a mixture of 3-methyl-2,4-pentanedione (2 ml) and toluene (2 ml) under reflux. The synthesis was carried out similarly to that used for the preparation of crystals of the bis(2,4-pentanedionato)lead(II) complex (Lyczko *et al.*, 2006). After heating the reaction mixture was stored for a few months until it converted into an orange–brown solid. Colourless crystals of the title

compound were separated from the reaction mixture. Analysis, calculated for $C_{16}H_{22}O_{10}Pb_2$: C 24.36, H 2.81%; found: C 24.14, H 2.88%.

S3. Refinement

H atoms were placed in calculated positions (O—H H atoms allowed to rotate but not to tip) with C—H = 0.98 Å (CH_3), C—H = 0.95 Å (aromatic H atoms), O—H = 0.84 Å and were refined isotropic with $U_{iso}(H) = 1.5U_{eq}(C, O)$ and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms using a riding model.

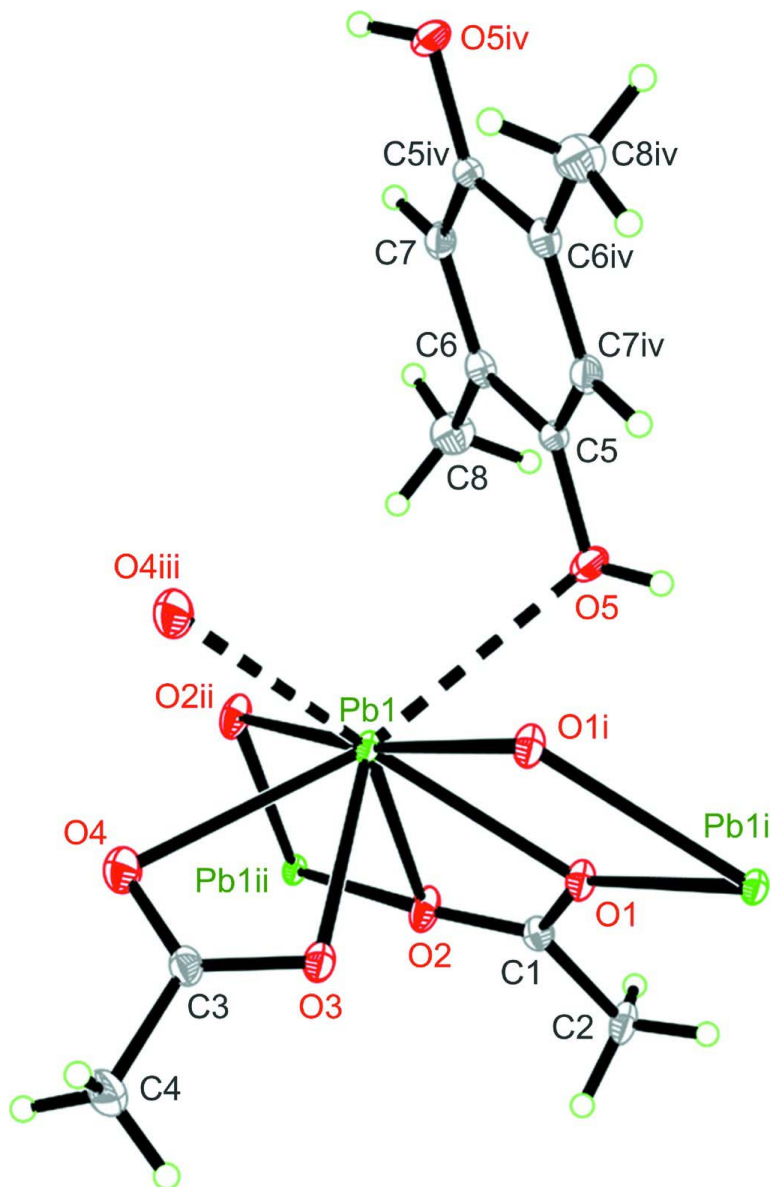


Figure 1

A fragment of the polymeric structure of the title compound with labelling showing the environment of the lead atom. Displacement ellipsoids of non-hydrogen atoms are drawn at the 50% probability level. [Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $-x, -y + 1, -z$; (iii) $-x, -y, -z$; (iv) $x + 1, -y + 1, -z + 1$].

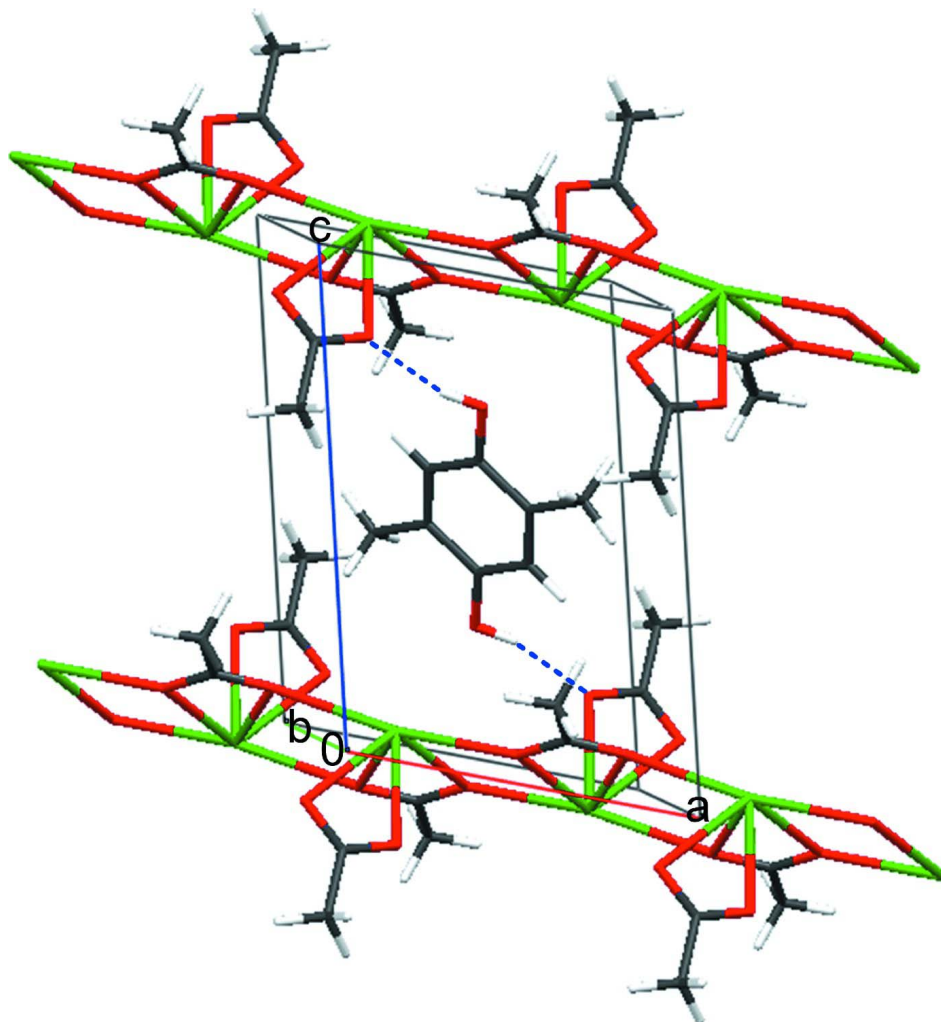


Figure 2

A packing diagram of the title compound. Hydrogen bonding is shown as blue dashed lines.

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Crystal data

[Pb₂(C₂H₃O₂)₄(C₈H₁₀O₂)]

$M_r = 788.72$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.4905$ (11) Å

$b = 7.5526$ (10) Å

$c = 10.2522$ (15) Å

$\alpha = 93.043$ (11)°

$\beta = 100.787$ (12)°

$\gamma = 116.377$ (14)°

$V = 504.28$ (12) Å³

$Z = 1$

$F(000) = 362$

$D_x = 2.597$ Mg m⁻³

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

Cell parameters from 9065 reflections

$\theta = 3.0$ – 28.7 °

$\mu = 16.72$ mm⁻¹

$T = 100$ K

Prismatic, colourless

$0.35 \times 0.26 \times 0.14$ mm

Data collection

Kuma KM-4 four-circle CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.6479 pixels mm⁻¹
 ω scans
Absorption correction: analytical
(*CrysAlis RED*; Oxford Diffraction, 2005)
 $T_{\min} = 0.040$, $T_{\max} = 0.227$

9065 measured reflections
2432 independent reflections
2317 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$
 $\theta_{\max} = 28.7^\circ$, $\theta_{\min} = 3.1^\circ$
 $h = -9 \rightarrow 9$
 $k = -10 \rightarrow 10$
 $l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.017$
 $wR(F^2) = 0.043$
 $S = 1.07$
2432 reflections
132 parameters
0 restraints
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
 $w = 1/[\sigma^2(F_o^2) + (0.0223P)^2 + 0.6288P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.30 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -1.67 \text{ e } \text{Å}^{-3}$
Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001x \text{Fc}^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0077 (5)

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.205617 (17)	0.362095 (17)	0.046792 (11)	0.00910 (6)
O1	0.4490 (4)	0.6463 (4)	-0.0457 (2)	0.0121 (5)
O2	0.1324 (4)	0.6005 (4)	-0.0858 (3)	0.0154 (5)
O3	0.1663 (4)	0.2264 (4)	-0.1751 (2)	0.0148 (5)
O4	-0.1191 (4)	0.0447 (4)	-0.1145 (3)	0.0214 (6)
C1	0.3154 (5)	0.6990 (5)	-0.0954 (3)	0.0111 (6)
C2	0.3723 (6)	0.8784 (6)	-0.1662 (4)	0.0155 (7)
H2A	0.5180	0.9365	-0.1668	0.023*
H2B	0.3466	0.9780	-0.1192	0.023*
H2C	0.2896	0.8380	-0.2589	0.023*
C3	-0.0132 (5)	0.0788 (6)	-0.1992 (4)	0.0135 (7)
C4	-0.0903 (6)	-0.0587 (6)	-0.3316 (4)	0.0212 (8)
H4A	-0.0509	-0.1658	-0.3216	0.032*
H4B	-0.0301	0.0174	-0.4002	0.032*

H4C	-0.2400	-0.1172	-0.3588	0.032*
C5	0.5070 (5)	0.5941 (5)	0.3871 (3)	0.0124 (7)
O5	0.5039 (4)	0.6836 (4)	0.2743 (3)	0.0166 (5)
H5	0.6216	0.7356	0.2591	0.025*
C6	0.3672 (5)	0.5861 (6)	0.4637 (4)	0.0129 (7)
C7	0.3647 (5)	0.4909 (5)	0.5766 (3)	0.0133 (7)
H7	0.2720	0.4841	0.6303	0.016*
C8	0.2300 (6)	0.6797 (7)	0.4230 (4)	0.0207 (8)
H8A	0.1526	0.6732	0.4914	0.031*
H8B	0.1344	0.6074	0.3366	0.031*
H8C	0.3133	0.8199	0.4145	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.00762 (8)	0.00740 (8)	0.01117 (8)	0.00219 (6)	0.00281 (5)	0.00244 (5)
O1	0.0087 (12)	0.0098 (12)	0.0179 (12)	0.0040 (10)	0.0032 (9)	0.0049 (10)
O2	0.0082 (12)	0.0150 (13)	0.0246 (14)	0.0054 (11)	0.0062 (10)	0.0077 (11)
O3	0.0130 (12)	0.0112 (12)	0.0157 (12)	0.0012 (11)	0.0048 (9)	0.0017 (10)
O4	0.0123 (13)	0.0247 (16)	0.0203 (14)	0.0021 (12)	0.0053 (10)	0.0018 (12)
C1	0.0119 (16)	0.0087 (16)	0.0123 (16)	0.0048 (14)	0.0025 (12)	0.0006 (13)
C2	0.0098 (17)	0.0120 (18)	0.0240 (19)	0.0034 (15)	0.0051 (14)	0.0091 (15)
C3	0.0107 (17)	0.0115 (17)	0.0151 (16)	0.0037 (15)	-0.0002 (13)	0.0028 (14)
C4	0.023 (2)	0.0139 (19)	0.0179 (18)	0.0035 (17)	0.0004 (15)	-0.0014 (15)
C5	0.0120 (16)	0.0105 (17)	0.0090 (15)	0.0010 (14)	0.0011 (12)	0.0005 (13)
O5	0.0146 (13)	0.0202 (14)	0.0150 (12)	0.0062 (12)	0.0061 (10)	0.0089 (11)
C6	0.0110 (17)	0.0105 (16)	0.0145 (16)	0.0036 (14)	0.0013 (12)	-0.0007 (13)
C7	0.0109 (16)	0.0128 (17)	0.0133 (16)	0.0029 (14)	0.0038 (12)	0.0001 (14)
C8	0.0190 (19)	0.025 (2)	0.0224 (19)	0.0138 (18)	0.0055 (15)	0.0065 (16)

Geometric parameters (Å, °)

Pb1—O3	2.355 (3)	C2—H2C	0.9800
Pb1—O1	2.493 (3)	C3—C4	1.509 (5)
Pb1—O2	2.499 (3)	C4—H4A	0.9800
Pb1—O1 ⁱ	2.618 (2)	C4—H4B	0.9800
Pb1—O4	2.700 (3)	C4—H4C	0.9800
Pb1—O2 ⁱⁱ	2.767 (3)	C5—O5	1.372 (4)
Pb1—O5	2.993 (3)	C5—C7 ^{iv}	1.384 (5)
Pb1—O4 ⁱⁱⁱ	2.994 (3)	C5—C6	1.405 (5)
O1—C1	1.267 (4)	O5—H5	0.8400
O1—Pb1 ⁱ	2.618 (2)	C6—C7	1.393 (5)
O2—C1	1.263 (4)	C6—C8	1.499 (5)
O3—C3	1.275 (4)	C7—C5 ^{iv}	1.384 (5)
O4—C3	1.245 (4)	C7—H7	0.9500
C1—C2	1.500 (5)	C8—H8A	0.9800
C2—H2A	0.9800	C8—H8B	0.9800
C2—H2B	0.9800	C8—H8C	0.9800

O3—Pb1—O1	75.58 (9)	O4—C3—O3	121.2 (3)
O3—Pb1—O2	77.94 (9)	O4—C3—C4	120.8 (3)
O1—Pb1—O2	52.08 (8)	O3—C3—C4	118.0 (3)
O3—Pb1—O1 ⁱ	75.04 (8)	C3—C4—H4A	109.5
O1—Pb1—O1 ⁱ	66.16 (9)	C3—C4—H4B	109.5
O2—Pb1—O1 ⁱ	116.77 (8)	H4A—C4—H4B	109.5
O3—Pb1—O4	50.93 (8)	C3—C4—H4C	109.5
O1—Pb1—O4	121.84 (8)	H4A—C4—H4C	109.5
O2—Pb1—O4	91.50 (9)	H4B—C4—H4C	109.5
O1 ⁱ —Pb1—O4	111.98 (8)	O5—C5—C7 ^{iv}	122.7 (3)
C1—O1—Pb1	94.0 (2)	O5—C5—C6	116.7 (3)
C1—O1—Pb1 ⁱ	149.3 (2)	C7 ^{iv} —C5—C6	120.6 (3)
Pb1—O1—Pb1 ⁱ	113.84 (9)	C5—O5—H5	109.5
C1—O2—Pb1	93.8 (2)	C7—C6—C5	117.4 (3)
C3—O3—Pb1	101.7 (2)	C7—C6—C8	122.9 (3)
C3—O4—Pb1	86.2 (2)	C5—C6—C8	119.8 (3)
O2—C1—O1	120.1 (3)	C5 ^{iv} —C7—C6	122.0 (3)
O2—C1—C2	119.4 (3)	C5 ^{iv} —C7—H7	119.0
O1—C1—C2	120.6 (3)	C6—C7—H7	119.0
C1—C2—H2A	109.5	C6—C8—H8A	109.5
C1—C2—H2B	109.5	C6—C8—H8B	109.5
H2A—C2—H2B	109.5	H8A—C8—H8B	109.5
C1—C2—H2C	109.5	C6—C8—H8C	109.5
H2A—C2—H2C	109.5	H8A—C8—H8C	109.5
H2B—C2—H2C	109.5	H8B—C8—H8C	109.5
O3—Pb1—O1—C1	86.8 (2)	O2—Pb1—O4—C3	74.2 (2)
O2—Pb1—O1—C1	1.04 (19)	O1 ⁱ —Pb1—O4—C3	-45.6 (2)
O1 ⁱ —Pb1—O1—C1	166.6 (3)	Pb1—O2—C1—O1	1.9 (3)
O4—Pb1—O1—C1	64.6 (2)	Pb1—O2—C1—C2	-178.1 (3)
O3—Pb1—O1—Pb1 ⁱ	-79.76 (11)	Pb1—O1—C1—O2	-1.9 (3)
O2—Pb1—O1—Pb1 ⁱ	-165.57 (16)	Pb1 ⁱ —O1—C1—O2	153.6 (3)
O1 ⁱ —Pb1—O1—Pb1 ⁱ	0.0	Pb1—O1—C1—C2	178.1 (3)
O4—Pb1—O1—Pb1 ⁱ	-101.97 (11)	Pb1 ⁱ —O1—C1—C2	-26.4 (6)
O3—Pb1—O2—C1	-82.0 (2)	Pb1—O4—C3—O3	-1.8 (3)
O1—Pb1—O2—C1	-1.05 (19)	Pb1—O4—C3—C4	175.7 (3)
O1 ⁱ —Pb1—O2—C1	-15.8 (2)	Pb1—O3—C3—O4	2.1 (4)
O4—Pb1—O2—C1	-131.5 (2)	Pb1—O3—C3—C4	-175.4 (3)
O1—Pb1—O3—C3	-156.6 (2)	O5—C5—C6—C7	178.3 (3)
O2—Pb1—O3—C3	-103.1 (2)	C7 ^{iv} —C5—C6—C7	0.3 (6)
O1 ⁱ —Pb1—O3—C3	134.7 (2)	O5—C5—C6—C8	-2.3 (5)
O4—Pb1—O3—C3	-1.1 (2)	C7 ^{iv} —C5—C6—C8	179.8 (4)
O3—Pb1—O4—C3	1.1 (2)	C5—C6—C7—C5 ^{iv}	-0.3 (6)
O1—Pb1—O4—C3	29.2 (2)	C8—C6—C7—C5 ^{iv}	-179.8 (4)

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x, -y+1, -z$; (iii) $-x, -y, -z$; (iv) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (Å, °)

<i>D—H···A</i>	<i>D—H</i>	<i>H···A</i>	<i>D···A</i>	<i>D—H···A</i>
O5—H5···O3 ⁱ	0.84	1.87	2.668 (4)	159

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