



ISSN 1600-5368

Crystal structure of $\text{Pb}_3(\text{IO}_4(\text{OH})_2)_2$

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Received 16 April 2014

Accepted 25 April 2014

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

Keywords: crystal structure; lead; periodate; non-merohedral twinning

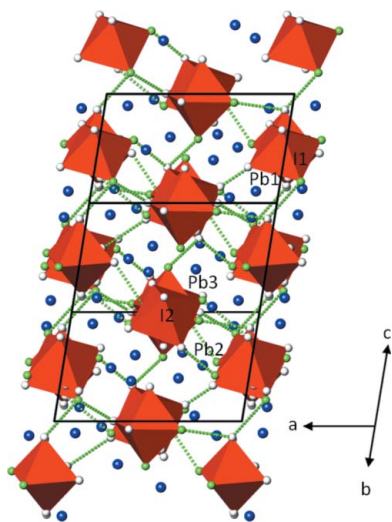
CCDC reference: 1004265

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The structure of the title compound, trilead(II) bis[dihydroxidotetraoxidoiodate(VII)], was determined from a crystal twinned by non-merohedry with two twin domains present [twin fraction 0.73 (1):0.27 (1)]. It contains three Pb^{2+} cations and two $\text{IO}_4(\text{OH})_2^{3-}$ anions in the asymmetric unit. Each of the Pb^{2+} cations is surrounded by eight O atoms (cut-off value = 3.1 Å) in the form of a distorted polyhedron. The octahedral $\text{IO}_4(\text{OH})_2^{3-}$ anions are arranged in rows extending parallel to [021], forming a distorted hexagonal rod packing. The cations and anions are linked into a framework structure. Although H-atom positions could not be located, $\text{O}\cdots\text{O}$ distances suggest medium-strength hydrogen-bonding interactions between the $\text{IO}_4(\text{OH})_2$ octahedra, further consolidating the crystal packing.

1. Chemical context

Lead and mercury can both exist in different oxidation states and each of the two elements exhibits a peculiar crystal chemistry. In the case of Pb^{2+} -containing compounds, the crystal chemistry is mainly dominated by the stereoactive $6s^2$ lone-pair of lead (Holloway & Melnik, 1997), whereas Hg^{2+} -containing compounds show a strong preference for a linear coordination of mercury (Breitinger, 2004). In this respect, it appears surprising that for some Pb^{2+} - and Hg^{2+} -containing compounds an isotopic relationship exists, *e.g.* for PbAs_2O_6 (Losilla *et al.*, 1995) and HgAs_2O_6 (Mormann & Jeitschko, 2000b; Weil, 2000), or for the mineral descloizite $\text{PbZn}(\text{VO}_4)\text{OH}$ (Hawthorne & Faggiani, 1979) and the synthetic phase $\text{HgZn}(\text{AsO}_4)\text{OH}$ (Weil, 2004). With this in mind, it seemed interesting to study the relation between phases in the systems $\text{Hg}^{\text{II}}\text{--I}^{\text{VII}}\text{--O--H}$ and $\text{Pb}^{\text{II}}\text{--I}^{\text{VII}}\text{--O--H}$. Whereas in the system $\text{Hg}^{\text{II}}\text{--I}^{\text{VII}}\text{--O--H}$ two compounds have been structurally characterized, *viz.* $\text{Hg}_3(\text{IO}_4(\text{OH})_2)_2$ (Mormann & Jeitschko, 2000a) and $\text{Hg}(\text{IO}_3(\text{OH})_3)$ (Mormann & Jeitschko, 2001), a phase in the system $\text{Pb}^{\text{II}}\text{--I}^{\text{VII}}\text{--O--H}$ has not yet been structurally determined, although several lead(II) periodate phases have been reported to exist. Willard & Thompson (1934) claimed to have obtained only one phase with composition $\text{Pb}_3\text{H}_4(\text{IO}_6)_2$ in the system $\text{Pb}^{\text{II}}\text{--I}^{\text{VII}}\text{--O--H}$. However, Drátovský & Matějčková (1965a,b) reported the existence of three phases with composition $\text{Pb}_3(\text{IO}_5)_2\cdot\text{H}_2\text{O}$, $\text{Pb}_2\text{I}_2\text{O}_9\cdot 3\text{H}_2\text{O}$ and $\text{Pb}_4\text{I}_2\text{O}_{11}\cdot 5\text{H}_2\text{O}$ in this system. To shed some light on the conflicting composition of the Pb:I 3:2 phase [$\text{Pb}_3\text{H}_4(\text{IO}_6)_2$ versus $\text{Pb}_3(\text{IO}_5)_2\cdot\text{H}_2\text{O}$ with a lower water content], the synthetic procedure described by Willard & Thompson (1934) was repeated for crystal growth of this lead periodate. The current structure determination of the obtained crystals showed the composition $\text{Pb}_3\text{H}_4(\text{IO}_6)_2$ as reported by Willard & Thompson (1934) to be correct. In a more reasonable crystal-chemical sense, the formula of these crystals should be rewritten as $\text{Pb}_3(\text{IO}_4(\text{OH})_2)_2$.



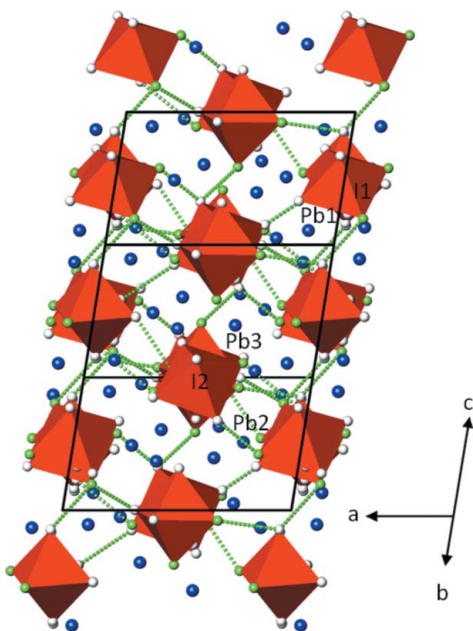


Figure 1

The crystal structure of $\text{Pb}_3(\text{IO}_4(\text{OH})_2)_2$ in a projection along $[021]$. Displacement ellipsoids are drawn at the 90% probability level. O atoms bearing the OH function are given in green, the other O atoms are white. Pb—O bonds are omitted for clarity; hydrogen-bonding interactions are shown as green dashed lines.

2. Structural commentary

Three Pb^{2+} cations and two $\text{IO}_4(\text{OH})_2^{3-}$ octahedra are present in the asymmetric unit. The anions form a slightly distorted hexagonal rod packing with the rods extending parallel to $[021]$. Cations and anions are linked through common oxygen atoms into a framework structure (Fig. 1).

Each of the Pb^{2+} cations exhibits a coordination number of eight if Pb—O interactions less than 3.1 Å are considered to be relevant. The resulting $[\text{PbO}_8]$ polyhedra are considerably distorted [Pb—O distances range from 2.433 (7) to 3.099 (8) Å]. The stereochemical activity of the electron lone pairs in each of the polyhedra appears not to be very pronounced (Fig. 2).

Compounds and structures containing the periodate anion have been reviewed some time ago by Levason (1997). The compiled I—O bond lengths are in good agreement with the two IO_6 octahedra of the title compound, having a mean I—O distance of 1.884 Å. Very similar mean values are found for comparable periodate compounds with large divalent cations, for example in $\text{BaI}_2\text{O}_6(\text{OH})_4 \cdot 2\text{H}_2\text{O}$ (one IO_6 octahedron, 1.895 Å; Häusel, 2008), in $\text{Ba}(\text{IO}_3(\text{OH})_3)$ (one IO_6 octahedron, 1.879 Å; Sasaki *et al.*, 1995), in $\text{Hg}_3(\text{IO}_4(\text{OH})_2)_2$ (two IO_6 octahedra, 1.888 Å; Mormann & Jeitschko, 2000a) or in $\text{Sr}(\text{IO}_2(\text{OH})_4)_2 \cdot 3\text{H}_2\text{O}$ (two IO_6 octahedra, 1.888 Å; Alexandrova & Häusel, 2004).

Results of bond-valence calculations (Brown, 2002), using the parameters of Brese & O'Keeffe (1991) for I—O bonds and of Krivovichev & Brown (2001) for Pb—O bonds, are reasonably close to the expected values (in valence units): Pb1 1.89, Pb2 1.73, Pb3 1.89, I1 6.78, I2 6.90, O1 1.95, O2 1.49, O3

Table 1

Selected bond lengths (Å).

I1—O6	1.845 (8)	I2—O11	1.820 (8)
I1—O3	1.860 (7)	I2—O9	1.850 (8)
I1—O2 ⁱ	1.861 (7)	I2—O8	1.855 (7)
I1—O1 ⁱⁱ	1.877 (7)	I2—O7	1.874 (8)
I1—O5 ⁱ	1.920 (8)	I2—O12	1.932 (9)
I1—O4 ⁱ	1.956 (8)	I2—O10	1.954 (8)

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

Table 2

Hydrogen-bond geometry (Å).

$D\cdots H\cdots A$	$D\cdots A$	$D-H\cdots A$	$D\cdots A$
O4 \cdots O7	2.849 (11)	O10 \cdots O11 ^{iv}	2.675 (11)
O4 \cdots O2 ⁱ	2.849 (11)	O12 \cdots O2 ^{iv}	2.852 (11)
O5 \cdots O11 ⁱⁱⁱ	2.634 (11)		

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

1.90, O4 1.15, O5 1.15, O6 1.92, O7 1.98, O8 1.95, O9 1.97, O10 1.09, O11 1.34, O12 1.12. The O atoms involved in hydrogen bonding are readily identifiable. The donor O atoms O4, O5, O10 and O12 exhibit the longest I—O bonds and the lowest bond-valence sums. Atom O11 has also a low bond-valence sum, explainable by its role as a twofold acceptor atom of medium-strength hydrogen-bonding interactions (Table 2) that additionally stabilize the packing of the structure (Fig. 1).

Comparison of the structures of $\text{Pb}_3(\text{IO}_4(\text{OH})_2)_2$ and of $\text{Hg}_3(\text{IO}_4(\text{OH})_2)_2$ [$P2_1/c$; $Z = 4$, $a = 8.5429$ (7), $b = 12.2051$ (8) Å, $c = 9.3549$ (8) Å, $\beta = 90.884$ (7)°] reveals some close similarities. A 'true' isotypic relationship (Lima-de-Faria *et al.*, 1990) is difficult to derive for the two structures.

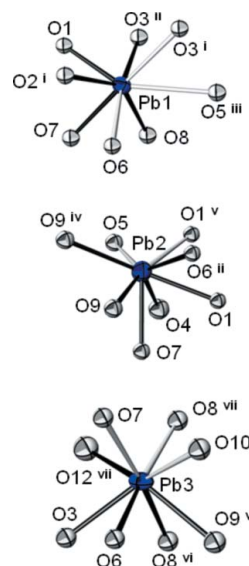


Figure 2

Coordination polyhedra of the three Pb^{2+} cations in the structure of $\text{Pb}_3(\text{IO}_4(\text{OH})_2)_2$. Bonds shorter than 2.7 Å are given by solid black lines, longer bonds between 2.7 and 3.1 Å as open black lines. Displacement ellipsoids are drawn at the 90% probability level. [Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $x, y - 1, z$; (iv) $-x + 1, -y + 1, -z$; (v) $-x, -y + 1, -z$; (vi) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (vii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (viii) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$.]

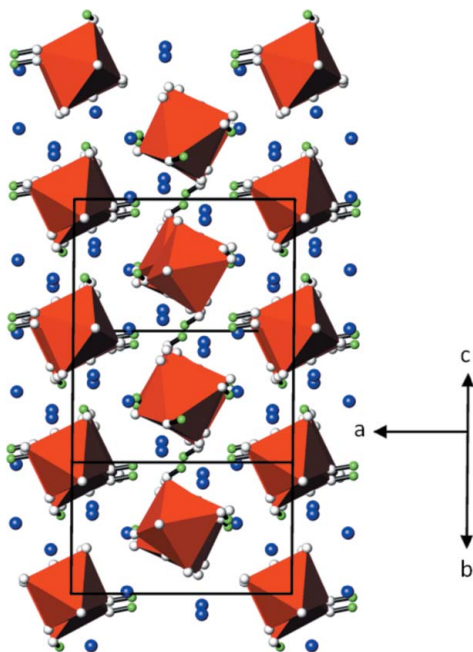


Figure 3
The crystal structure of $\text{Hg}_3(\text{IO}_4(\text{OH})_2)_2$ (Mormann & Jeitschko, 2000a) in a projection along [011]. Colour code as in Fig. 1. Hg—O and O—H...O interactions are omitted for clarity.

However, they are isopointal and show the same type of arrangement in terms of the crystal packing. In the mercury compound, the $\text{IO}_4(\text{OH})_2^{3-}$ octahedra are likewise hexagonally packed in rods (Fig. 3). The cations are situated in between this arrangement which is further consolidated by O—H...O hydrogen bonding.

3. Synthesis and crystallization

The preparation conditions described by Willard & Thompson (1934) were modified slightly. Instead of using NaIO_4 as the periodate source, periodic acid was employed.

1.25 g $\text{Pb}(\text{NO}_3)_2$ was dissolved in 25 ml water, acidified with a few drops of concentrated nitric acid and heated until boiling. Then the periodic acid solution (0.85 g in 25 ml water) was slowly added to the lead solution. The addition of the first portion of the periodic acid solution (ca. 3–4 ml) resulted in an off-white precipitate near the drop point that redissolved under stirring. After further addition, the precipitate remained and changed the colour in the still boiling solution from off-white to yellow–orange within half an hour. After filtration of the precipitate, a few colourless crystals of the title compound formed in the mother liquor on cooling. X-ray powder diffraction data of the polycrystalline precipitate are in very good agreement with simulated data based on the refinement of $\text{Pb}_3(\text{IO}_4(\text{OH})_2)_2$.

4. Refinement

All investigated crystals were twinned by non-merohedry. Intensity data of the measured crystal could be indexed to belong to two domains, with a refined twin domain ratio of

Table 3

Experimental details.

Crystal data	
Chemical formula	$\text{Pb}_3[\text{IO}_4(\text{OH})_2]_2$
M_r	1071.40
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	8.9653 (9), 9.2113 (9), 12.8052 (13)
β (°)	101.042 (2)
V (Å ³)	1037.90 (18)
Z	4
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	54.55
Crystal size (mm)	0.06 × 0.06 × 0.05
Data collection	
Diffractometer	Siemens SMART CCD
Absorption correction	Multi-scan (TWINABS; Bruker, 2008)
T_{\min}, T_{\max}	0.253, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3196, 3196, 2587
$(\sin \theta/\lambda)_{\max}$ (Å ⁻¹)	0.716
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.041, 0.087, 1.07
No. of reflections	3196
No. of parameters	94
H-atom treatment	H-atom parameters not refined
$\Delta\rho_{\max}, \Delta\rho_{\min}$ (e Å ⁻³)	2.88, -1.95

Computer programs: SMART (Bruker, 2008), SAINT-Plus (Bruker, 2008), SHELXS97 and SHELXL97 (Sheldrick, 2008), ATOMS for Windows (Dowty, 2006) and publCIF (Westrip, 2010).

0.73 (1):0.27 (1). Reflections originating from the minor component as well as overlapping reflections of the two domains (less than 10% of all measured reflections) were separated and excluded. The H atoms of the $\text{IO}_4(\text{OH})_2$ octahedra could not be located from difference maps and were therefore not considered in the final model. The O atoms were refined with isotropic displacement parameters. The remaining maximum and minimum electron densities are found 0.73 and 0.68 Å⁻³, respectively, from atom Pb2. Structure data were finally standardized with STRUCTURE-TIDY (Gelato & Parthé, 1987). It should be noted that the intensity statistics point to a pronounced C-centred basis cell (space group $C2/c$ with lattice parameters of $a \simeq 14.16$, $b \simeq 9.21$, $c \simeq 8.97$ Å, $\beta \simeq 117.4^\circ$) with weak superstructure reflections violating the C-centering.

Acknowledgements

The X-ray centre of the Vienna University of Technology is acknowledged for providing access to the single-crystal diffractometer.

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supporting information

Acta Cryst. (2014). E70, 14–17 [doi:10.1107/S1600536814009520]

Crystal structure of $\text{Pb}_3(\text{IO}_4(\text{OH})_2)_2$

Matthias Weil

Computing details

Data collection: *SMART* (Bruker, 2008); cell refinement: *SAINT-Plus* (Bruker, 2008); data reduction: *SAINT-Plus* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ATOMS for Windows* (Dowty, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Trilead(II) bis[dihydroxidotetraoxidoiodate(VII)]

Crystal data

$\text{Pb}_3[\text{IO}_4(\text{OH})_2]_2$
 $M_r = 1071.40$
 Monoclinic, $P2_1/c$
 Hall symbol: -P 2ybc
 $a = 8.9653$ (9) Å
 $b = 9.2113$ (9) Å
 $c = 12.8052$ (13) Å
 $\beta = 101.042$ (2)°
 $V = 1037.90$ (18) Å³
 $Z = 4$

$F(000) = 1808$
 $D_x = 6.857$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 3673 reflections
 $\theta = 3.2\text{--}30.5^\circ$
 $\mu = 54.55$ mm⁻¹
 $T = 296$ K
 Block, colourless
 $0.06 \times 0.06 \times 0.05$ mm

Data collection

Siemens SMART CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (*TWINABS*; Bruker, 2008)
 $T_{\min} = 0.253$, $T_{\max} = 0.746$

3196 measured reflections
 3196 independent reflections
 2587 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.000$
 $\theta_{\max} = 30.6^\circ$, $\theta_{\min} = 2.3^\circ$
 $h = -12 \rightarrow 12$
 $k = 0 \rightarrow 13$
 $l = 0 \rightarrow 18$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.087$
 $S = 1.07$
 3196 reflections
 94 parameters
 0 restraints

Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map
 H-atom parameters not refined
 $w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 17.8096P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 2.88$ e Å⁻³
 $\Delta\rho_{\min} = -1.95$ e Å⁻³

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.12192 (5)	0.13042 (4)	0.11904 (3)	0.01318 (10)
Pb2	0.25685 (5)	0.51903 (5)	0.01540 (4)	0.01842 (11)
Pb3	0.37507 (5)	0.36874 (5)	0.38134 (3)	0.01409 (10)
I1	0.00670 (7)	0.23313 (7)	0.36046 (5)	0.00838 (13)
I2	0.50186 (7)	0.24738 (6)	0.14267 (5)	0.00735 (13)
O1	0.0343 (9)	0.3332 (8)	0.0015 (6)	0.0110 (14)*
O2	0.0389 (9)	0.7922 (8)	0.2811 (6)	0.0125 (15)*
O3	0.1058 (9)	0.4046 (8)	0.4090 (6)	0.0122 (15)*
O4	0.1088 (10)	0.5549 (9)	0.1797 (6)	0.0178 (17)*
O5	0.1831 (9)	0.8100 (8)	0.1159 (6)	0.0129 (15)*
O6	0.1842 (9)	0.1429 (8)	0.3433 (6)	0.0117 (15)*
O7	0.3161 (9)	0.3260 (8)	0.1618 (6)	0.0121 (15)*
O8	0.4052 (9)	0.0832 (8)	0.0785 (6)	0.0121 (15)*
O9	0.4802 (9)	0.3391 (8)	0.0121 (6)	0.0144 (16)*
O10	0.5247 (9)	0.1474 (8)	0.2794 (6)	0.0145 (16)*
O11	0.6146 (10)	0.3912 (8)	0.2170 (6)	0.0158 (16)*
O12	0.6856 (10)	0.1562 (9)	0.1172 (6)	0.0182 (17)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0134 (2)	0.01402 (19)	0.01215 (19)	−0.00236 (15)	0.00243 (15)	0.00042 (14)
Pb2	0.0168 (2)	0.01424 (19)	0.0236 (2)	0.00084 (17)	0.00251 (16)	0.00040 (16)
Pb3	0.0153 (2)	0.0173 (2)	0.01036 (18)	−0.00366 (16)	0.00426 (15)	−0.00050 (14)
I1	0.0075 (3)	0.0104 (3)	0.0072 (3)	−0.0003 (2)	0.0014 (2)	0.0006 (2)
I2	0.0066 (3)	0.0078 (3)	0.0076 (3)	−0.0002 (2)	0.0011 (2)	−0.0005 (2)

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

Pb1—O1	2.433 (7)	Pb3—O9 ^{vi}	2.599 (8)
Pb1—O7	2.493 (8)	Pb3—O6	2.678 (8)
Pb1—O2 ⁱ	2.577 (8)	Pb3—O12 ^{vii}	2.704 (8)
Pb1—O3 ⁱⁱ	2.685 (7)	Pb3—O8 ^{vii}	2.767 (8)
Pb1—O8	2.723 (8)	Pb3—O7	2.787 (7)
Pb1—O6	2.821 (7)	Pb3—O10	2.886 (8)
Pb1—O3 ⁱ	2.888 (8)	I1—O6	1.845 (8)

Pb1—O5 ⁱⁱⁱ	3.004 (7)	I1—O3	1.860 (7)
Pb2—O7	2.564 (7)	I1—O2 ⁱ	1.861 (7)
Pb2—O9	2.606 (8)	I1—O1 ^{vi}	1.877 (7)
Pb2—O1	2.609 (7)	I1—O5 ⁱ	1.920 (8)
Pb2—O6 ⁱⁱ	2.638 (7)	I1—O4 ⁱ	1.956 (8)
Pb2—O4	2.714 (8)	I2—O11	1.820 (8)
Pb2—O9 ^{iv}	2.777 (8)	I2—O9	1.850 (8)
Pb2—O1 ^v	2.915 (7)	I2—O8	1.855 (7)
Pb2—O5	3.099 (8)	I2—O7	1.874 (8)
Pb3—O8 ^{vi}	2.527 (7)	I2—O12	1.932 (9)
Pb3—O3	2.528 (8)	I2—O10	1.954 (8)
O1—Pb1—O7	73.1 (2)	O7—I2—O10	90.5 (3)
O1—Pb1—O2 ⁱ	73.6 (2)	O12—I2—O10	90.0 (3)
O7—Pb1—O2 ⁱ	84.6 (2)	I1 ⁱⁱ —O1—Pb1	108.2 (3)
O1—Pb1—O3 ⁱⁱ	61.5 (2)	I1 ⁱⁱ —O1—Pb2	103.7 (3)
O7—Pb1—O3 ⁱⁱ	102.0 (2)	Pb1—O1—Pb2	108.0 (3)
O2 ⁱ —Pb1—O3 ⁱⁱ	129.7 (2)	I1 ⁱⁱ —O1—Pb2 ^v	97.4 (3)
O1—Pb1—O8	102.0 (2)	Pb1—O1—Pb2 ^v	125.6 (3)
O7—Pb1—O8	61.3 (2)	Pb2—O1—Pb2 ^v	111.1 (2)
O2 ⁱ —Pb1—O8	144.9 (2)	I1 ⁱⁱ —O1—Pb3 ⁱⁱ	57.6 (2)
O3 ⁱⁱ —Pb1—O8	70.3 (2)	Pb1—O1—Pb3 ⁱⁱ	73.14 (18)
O1—Pb1—O6	125.2 (2)	Pb2—O1—Pb3 ⁱⁱ	73.13 (17)
O7—Pb1—O6	75.7 (2)	Pb2 ^v —O1—Pb3 ⁱⁱ	154.3 (2)
O2 ⁱ —Pb1—O6	59.4 (2)	I1 ^{viii} —O2—Pb1 ^{viii}	106.0 (3)
O3 ⁱⁱ —Pb1—O6	170.7 (2)	I1 ^{viii} —O2—Pb2 ^{ix}	156.0 (4)
O8—Pb1—O6	101.0 (2)	Pb1 ^{viii} —O2—Pb2 ^{ix}	97.4 (2)
O1—Pb1—O3 ⁱ	109.8 (2)	I1 ^{viii} —O2—Pb1 ^x	75.1 (2)
O7—Pb1—O3 ⁱ	174.4 (2)	Pb1 ^{viii} —O2—Pb1 ^x	155.4 (3)
O2 ⁱ —Pb1—O3 ⁱ	91.7 (2)	Pb2 ^{ix} —O2—Pb1 ^x	86.03 (16)
O3 ⁱⁱ —Pb1—O3 ⁱ	83.5 (2)	I1 ^{viii} —O2—Pb3 ^{viii}	62.0 (2)
O8—Pb1—O3 ⁱ	121.6 (2)	Pb1 ^{viii} —O2—Pb3 ^{viii}	78.83 (19)
O6—Pb1—O3 ⁱ	98.8 (2)	Pb2 ^{ix} —O2—Pb3 ^{viii}	129.9 (2)
O1—Pb1—O5 ⁱⁱⁱ	141.7 (2)	Pb1 ^x —O2—Pb3 ^{viii}	80.39 (14)
O7—Pb1—O5 ⁱⁱⁱ	126.3 (2)	I1—O3—Pb3	104.5 (3)
O2 ⁱ —Pb1—O5 ⁱⁱⁱ	134.1 (2)	I1—O3—Pb1 ^{vi}	99.4 (3)
O3 ⁱⁱ —Pb1—O5 ⁱⁱⁱ	81.0 (2)	Pb3—O3—Pb1 ^{vi}	104.8 (3)
O8—Pb1—O5 ⁱⁱⁱ	70.2 (2)	I1—O3—Pb1 ^{viii}	106.8 (3)
O6—Pb1—O5 ⁱⁱⁱ	93.0 (2)	Pb3—O3—Pb1 ^{viii}	138.4 (3)
O3 ⁱ —Pb1—O5 ⁱⁱⁱ	54.4 (2)	Pb1 ^{vi} —O3—Pb1 ^{viii}	96.5 (2)
O7—Pb2—O9	61.2 (2)	I1 ^{viii} —O4—Pb2	102.2 (3)
O7—Pb2—O1	69.1 (2)	I1 ^{viii} —O4—Pb3	146.5 (3)
O9—Pb2—O1	99.3 (2)	Pb2—O4—Pb3	98.2 (2)
O7—Pb2—O6 ⁱⁱ	101.7 (2)	I1 ^{viii} —O4—Pb1 ^{viii}	71.6 (2)
O9—Pb2—O6 ⁱⁱ	72.2 (2)	Pb2—O4—Pb1 ^{viii}	173.4 (3)
O1—Pb2—O6 ⁱⁱ	60.6 (2)	Pb3—O4—Pb1 ^{viii}	88.39 (17)
O7—Pb2—O4	65.3 (2)	I1 ^{viii} —O4—Pb2 ^v	68.3 (2)
O9—Pb2—O4	125.5 (2)	Pb2—O4—Pb2 ^v	87.5 (2)

O1—Pb2—O4	69.6 (2)	Pb3—O4—Pb2 ^v	139.5 (2)
O6 ⁱⁱ —Pb2—O4	129.6 (2)	Pb1 ^{viii} —O4—Pb2 ^v	87.94 (18)
O7—Pb2—O9 ^{iv}	110.9 (2)	I1 ^{viii} —O4—Pb1	143.6 (3)
O9—Pb2—O9 ^{iv}	67.9 (3)	Pb2—O4—Pb1	72.12 (18)
O1—Pb2—O9 ^{iv}	163.1 (2)	Pb3—O4—Pb1	68.44 (14)
O6 ⁱⁱ —Pb2—O9 ^{iv}	103.9 (2)	Pb1 ^{viii} —O4—Pb1	111.3 (2)
O4—Pb2—O9 ^{iv}	126.5 (2)	Pb2 ^v —O4—Pb1	75.48 (15)
O7—Pb2—O1 ^v	115.8 (2)	I1 ^{viii} —O5—Pb1 ^x	101.0 (3)
O9—Pb2—O1 ^v	167.4 (2)	I1 ^{viii} —O5—Pb2	90.7 (3)
O1—Pb2—O1 ^v	68.9 (2)	Pb1 ^x —O5—Pb2	155.4 (3)
O6 ⁱⁱ —Pb2—O1 ^v	97.3 (2)	I1 ^{viii} —O5—Pb1 ^v	69.1 (2)
O4—Pb2—O1 ^v	55.7 (2)	Pb1 ^x —O5—Pb1 ^v	75.90 (16)
O9 ^{iv} —Pb2—O1 ^v	122.7 (2)	Pb2—O5—Pb1 ^v	88.49 (18)
O7—Pb2—O5	109.1 (2)	I1 ^{viii} —O5—Pb3 ^{vii}	163.1 (3)
O9—Pb2—O5	141.5 (2)	Pb1 ^x —O5—Pb3 ^{vii}	92.88 (19)
O1—Pb2—O5	112.0 (2)	Pb2—O5—Pb3 ^{vii}	80.25 (17)
O6 ⁱⁱ —Pb2—O5	142.9 (2)	Pb1 ^v —O5—Pb3 ^{vii}	124.4 (2)
O4—Pb2—O5	53.0 (2)	I1—O6—Pb2 ^{vi}	103.6 (3)
O9 ^{iv} —Pb2—O5	84.2 (2)	I1—O6—Pb3	99.4 (3)
O1 ^v —Pb2—O5	50.8 (2)	Pb2 ^{vi} —O6—Pb3	103.9 (3)
O8 ^{vi} —Pb3—O3	76.1 (3)	I1—O6—Pb1	97.6 (3)
O8 ^{vi} —Pb3—O9 ^{vi}	61.9 (2)	Pb2 ^{vi} —O6—Pb1	142.8 (3)
O3—Pb3—O9 ^{vi}	104.1 (2)	Pb3—O6—Pb1	102.2 (2)
O8 ^{vi} —Pb3—O6	105.1 (2)	I2—O7—Pb1	107.0 (3)
O3—Pb3—O6	62.2 (2)	I2—O7—Pb2	103.7 (3)
O9 ^{vi} —Pb3—O6	71.7 (2)	Pb1—O7—Pb2	107.6 (3)
O8 ^{vi} —Pb3—O12 ^{vii}	78.7 (2)	I2—O7—Pb3	100.6 (3)
O3—Pb3—O12 ^{vii}	70.9 (3)	Pb1—O7—Pb3	108.2 (3)
O9 ^{vi} —Pb3—O12 ^{vii}	140.0 (2)	Pb2—O7—Pb3	127.7 (3)
O6—Pb3—O12 ^{vii}	129.8 (2)	I2—O7—Pb3 ⁱⁱ	56.96 (19)
O8 ^{vi} —Pb3—O8 ^{vii}	75.7 (3)	Pb1—O7—Pb3 ⁱⁱ	72.30 (17)
O3—Pb3—O8 ^{vii}	123.1 (2)	Pb2—O7—Pb3 ⁱⁱ	73.10 (17)
O9 ^{vi} —Pb3—O8 ^{vii}	104.4 (2)	Pb3—O7—Pb3 ⁱⁱ	154.9 (3)
O6—Pb3—O8 ^{vii}	174.5 (2)	I2—O8—Pb3 ⁱⁱ	104.5 (3)
O12 ^{vii} —Pb3—O8 ^{vii}	55.7 (2)	I2—O8—Pb1	99.1 (3)
O8 ^{vi} —Pb3—O7	174.9 (2)	Pb3 ⁱⁱ —O8—Pb1	103.7 (3)
O3—Pb3—O7	99.1 (2)	I2—O8—Pb3 ^{xi}	104.1 (3)
O9 ^{vi} —Pb3—O7	121.4 (2)	Pb3 ⁱⁱ —O8—Pb3 ^{xi}	104.3 (3)
O6—Pb3—O7	73.5 (2)	Pb1—O8—Pb3 ^{xi}	137.3 (3)
O12 ^{vii} —Pb3—O7	98.4 (2)	I2—O9—Pb3 ⁱⁱ	102.0 (3)
O8 ^{vii} —Pb3—O7	106.2 (2)	I2—O9—Pb2	102.9 (3)
O8 ^{vi} —Pb3—O10	127.3 (2)	Pb3 ⁱⁱ —O9—Pb2	107.1 (3)
O3—Pb3—O10	134.0 (2)	I2—O9—Pb2 ^{iv}	112.6 (4)
O9 ^{vi} —Pb3—O10	68.2 (2)	Pb3 ⁱⁱ —O9—Pb2 ^{iv}	118.5 (3)
O6—Pb3—O10	72.9 (2)	Pb2—O9—Pb2 ^{iv}	112.1 (3)
O12 ^{vii} —Pb3—O10	143.8 (2)	I2—O10—Pb3	95.4 (3)
O8 ^{vii} —Pb3—O10	102.3 (2)	I2—O10—Pb2 ^{xi}	148.8 (4)
O7—Pb3—O10	57.2 (2)	Pb3—O10—Pb2 ^{xi}	98.9 (2)

O6—I1—O3	93.1 (3)	I2—O10—Pb3 ^{xi}	79.3 (2)
O6—I1—O2 ⁱ	92.8 (3)	Pb3—O10—Pb3 ^{xi}	167.0 (3)
O3—I1—O2 ⁱ	94.6 (3)	Pb2 ^{xi} —O10—Pb3 ^{xi}	91.50 (19)
O6—I1—O1 ^{vi}	90.6 (3)	I2—O10—Pb1	67.0 (2)
O3—I1—O1 ^{vi}	89.3 (3)	Pb3—O10—Pb1	78.32 (18)
O2 ⁱ —I1—O1 ^{vi}	174.7 (3)	Pb2 ^{xi} —O10—Pb1	143.2 (2)
O6—I1—O5 ⁱ	174.5 (3)	Pb3 ^{xi} —O10—Pb1	88.65 (17)
O3—I1—O5 ⁱ	90.9 (3)	I2—O11—Pb3	85.6 (3)
O2 ⁱ —I1—O5 ⁱ	90.5 (3)	I2—O11—Pb2 ^{iv}	88.1 (3)
O1 ^{vi} —I1—O5 ⁱ	85.8 (3)	Pb3—O11—Pb2 ^{iv}	157.4 (3)
O6—I1—O4 ⁱ	90.9 (3)	I2—O11—Pb1 ^{vii}	171.0 (4)
O3—I1—O4 ⁱ	174.5 (3)	Pb3—O11—Pb1 ^{vii}	95.8 (2)
O2 ⁱ —I1—O4 ⁱ	89.0 (3)	Pb2 ^{iv} —O11—Pb1 ^{vii}	93.74 (19)
O1 ^{vi} —I1—O4 ⁱ	86.9 (3)	I2—O11—Pb2	64.5 (2)
O5 ⁱ —I1—O4 ⁱ	84.8 (3)	Pb3—O11—Pb2	83.53 (18)
O11—I2—O9	95.3 (3)	Pb2 ^{iv} —O11—Pb2	74.20 (15)
O11—I2—O8	172.0 (3)	Pb1 ^{vii} —O11—Pb2	124.5 (2)
O9—I2—O8	90.7 (3)	I2—O12—Pb3 ^{xi}	104.2 (4)
O11—I2—O7	93.9 (4)	I2—O12—Pb2 ^{iv}	85.5 (3)
O9—I2—O7	90.0 (3)	Pb3 ^{xi} —O12—Pb2 ^{iv}	151.9 (3)
O8—I2—O7	91.2 (3)	I2—O12—Pb3 ⁱⁱ	68.4 (2)
O11—I2—O12	89.9 (4)	Pb3 ^{xi} —O12—Pb3 ⁱⁱ	79.9 (2)
O9—I2—O12	89.5 (4)	Pb2 ^{iv} —O12—Pb3 ⁱⁱ	79.42 (16)
O8—I2—O12	84.9 (3)	I2—O12—Pb1 ^{xii}	155.4 (4)
O7—I2—O12	176.1 (3)	Pb3 ^{xi} —O12—Pb1 ^{xii}	98.2 (2)
O11—I2—O10	85.5 (3)	Pb2 ^{iv} —O12—Pb1 ^{xii}	79.41 (17)
O9—I2—O10	179.1 (3)	Pb3 ⁱⁱ —O12—Pb1 ^{xii}	126.6 (2)
O8—I2—O10	88.4 (3)		

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

Hydrogen-bond geometry (\AA)

$D\cdots H\cdots A$	$D\cdots A$
O4 \cdots O7	2.849 (11)
O4 \cdots O2 ⁱ	2.849 (11)
O5 \cdots O11 ^{vii}	2.634 (11)
O10 \cdots O11 ^{xi}	2.675 (11)
O12 \cdots O2 ^{xi}	2.852 (11)

Symmetry codes: (i) $-x, y-1/2, -z+1/2$; (vii) $-x+1, y+1/2, -z+1/2$; (xi) $-x+1, y-1/2, -z+1/2$.