Crystal structure of Pb₃(IO₄(OH)₂)₂

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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 Pb²⁺ cations and two IO₄(OH)₂³⁻ anions in the asymmetric unit. Each of the Pb²⁺ cations is surrounded by eight O atoms (cut-off value = 3.1 Å) in the form of a distorted polyhedron. The octahedral IO₄(OH)₂³⁻ 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, O···O distances suggest medium-strength hydrogen-bonding interactions between the IO₄(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 Pb²⁺-containing compounds, the crystal chemistry is mainly dominated by the stereoactive 6s² lone-pair of lead (Holloway & Melnik, 1997), whereas Hg²⁺-containing compounds show a strong preference for a linear coordination of mercury (Breitinger, 2004). In this respect, it appears surprising that for some Pb²⁺- and Hg²⁺-containing compounds an isotypic relationship exists, e.g. for PbAs₂O₆ (Losilla et al., 1995) and HgAs₂O₆ (Mormann & Jeitschko, 2000b; Weil, 2000), or for the mineral descoelite PbZn(VO₄)OH (Hawthorne & Faggiani, 1979) and the synthetic phase HgZn(AsO₄)OH (Weil, 2004). With this in mind, it seemed interesting to study the relation between phases in the systems HgII–IVII–O–H and PbII–IVII–O–H. Whereas in the system HgII–IVII–O–H two compounds have been structurally characterized, viz. Hg₃(IO₄(OH))₂ (Mormann & Jeitschko, 2000a) and Hg(IO₃(OH))₃ (Mormann & Jeitschko, 2001), a phase in the system PbII–IVII–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 Pb₃H₄(IO₆)₂ in the system PbII–IVII–O–H. However, Drátovský & Matějčková (1965a,b) reported the existence of three phases with composition Pb₅(IO₅)₂·H₂O, Pb₂I₂O₇·3H₂O and Pb₂I₂O₁₁·5H₂O in this system. To shed some light on the conflicting composition of the PbI₃·2H₂O phase [Pb₃H₄(IO₆)₂ versus Pb₅(IO₅)₂·H₂O 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 Pb₃H₄(IO₆)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 Pb₃(IO₄(OH))₂²⁻.
for example in BaI2O6(OH)4 comparable periodate compounds with large divalent cations, two IO6 octahedra of the title compound, having a mean I—O bond lengths are in good agreement with the parameter of Brese & O’Keeffe (1991) for I—O bonds, are very similar mean values are found for IO6 octahedra, 1.888 Å; Mormann & Jeitschko, 2000.\[1.895 Å; Ha¨useler 2008\] in Hg3(IO3(OH)3) (one IO6 octaha & Ha¨useler, 2004). Two IO6 octahedra are present in the asymmetric unit. The anions form a slightly distorted hexagonal rod packing with the rods extending parallel to the [021] direction. Each of the IO6 octahedra exhibit a coordination number of 8 if Pb—O interactions less than 3.1 Å are considered to be relevant. The resulting [PbO8] 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).

2. Structural commentary

Three Pb2+ cations and two IO4(OH)2− 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 Pb2+ cations exhibits a coordination number of 6 if 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.

**Figure 1**
The crystal structure of Pb3[IO4(OH)2]2 in a projection along [021]. Displacement ellipsoids are drawn at the 90% probability level. O atoms are shown as green dashed lines.

**Table 1**

| Bond Lengths (Å) |
|------------------|
| Pb1—O6           | 1.845 (8) |
| Pb1—O3           | 1.860 (7) |
| Pb1—O2           | 1.861 (7) |
| Pb1—O11          | 1.877 (7) |
| Pb1—O5           | 1.920 (8) |
| Pb1—O4           | 1.956 (8) |
| O2—O10           | 2.675 (11) |
| O2—O12           | 2.852 (11) |
| O11—O10          | 2.634 (11) |

**Table 2**

| Hydrogen-bond geometry (Å) |
|----------------------------|
| D—H···A | D···A |
|---------|-------|
| O4···O7  | 2.849 (11) | O10···O11\(\text{w}\)  | 2.675 (11) |
| O4···O2\(\text{w}\)  | 2.849 (11) | O12···O2\(\text{w}\)  | 2.852 (11) |

**Figure 2**

Coordination polyhedra of the three Pb2+ cations in the structure of Pb3[IO4(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) \(-x, y + \frac{1}{2}, z - \frac{1}{2}\); (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) \(-x, y + \frac{1}{2}, -z + \frac{1}{2}\)]

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Each of the Pb2+ cations exhibits a coordination number of eight if Pb—O interactions less than 3.1 Å are considered to be relevant. The resulting [PbO8] 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 peridote anion have been reviewed some time ago by Levason (1997). The compiled I—O bond lengths are in good agreement with the two IO6 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 BaI2O6(OH)4·2H2O (one IO6 octahedron, 1.895 Å; Häuseler, 2008), in Ba(IO4(OH)3) (one IO6 octahedron, 1.879 Å; Sasaki et al., 1995), in Hg3(IO4(OH)2)2 (two IO6 octahedra, 1.888 Å; Mormann & Jeitschko, 2000a) or in Sr(IO4(OH)2)2·3H2O (two IO6 octahedra, 1.888 Å; Alexandrova & Häuseler, 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 1.90, O4 1.15, O5 1.15, O6 1.92, O7 1.98, O8 1.95, O 9 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 Pb3[IO4(OH)2]2 and of Hg3[IO4(OH)2]2 [P21/c; Z = 4, \(a = 8.5429\) (7), \(b = 12.2051\) (8) Å, \(c = 9.3549\) (8) Å, \(\beta = 90.884\) (7)\(\text{°}\)] reveals some close similarities. A ‘true’ isotypic relationship (Lima-de-Faria et al., 1990) is difficult to derive for the two structures.
However, they are isopointal and show the same type of arrangement in terms of the crystal packing. In the mercury compound, the IO$_4$(OH)$_2$ 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 Pb(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 part 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 to 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 Pb$_3$(IO$_4$(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 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 IO$_4$(OH)$_2$ octahedra could not be located from difference maps and were therefore not considered in the final model. The O atoms were separated and excluded. The H-atom treatment H-atom parameters not refined

Table 3

| Property | Value |
|----------|-------|
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 296 |
| $a$, $b$, $c$ (Å) | 8.9653 (9), 9.2113 (9), 12.8052 (13) |
| $V$ (Å$^3$) | 1037.90 (18) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 54.55 |
| Crystal size (mm) | 0.06 $\times$ 0.06 $\times$ 0.05 |

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

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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

\( \text{Pb}_3[\text{IO}_4(\text{OH})_2] \)

- \( M_r = 1071.40 \)
- 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) Å\(^3\)
- \( Z = 4 \)
- \( F(000) = 1808 \)
- \( D_x = 6.857 \text{ Mg m}^{-3} \)
- \( \lambda = 0.71073 \) Å
- Mo \( K\alpha \) radiation
- Cell parameters from 3673 reflections
- \( \theta = 3.2−30.5^\circ \)
- \( \mu = 54.55 \text{ mm}^{-1} \)
- \( T = 296 \text{ K} \)
- Block, colourless
- \( 0.06 \times 0.06 \times 0.05 \text{ mm} \)

Data collection

Siemens SMART CCD diffractometer
- Radiation source: fine-focus sealed tube
- Graphite monochromator
- \( \omega \) scans
- Absorption correction: multi-scan (TWINABS; Bruker, 2008)
- \( T_{\text{min}} = 0.253, T_{\text{max}} = 0.746 \)
- 3196 measured reflections
- 3196 independent reflections
- 2587 reflections with \( I > 2\sigma(I) \)
- \( R_{\text{int}} = 0.000 \)
- \( \theta_{\text{max}} = 30.6^\circ, \theta_{\text{min}} = 2.3^\circ \)
- \( h = -12\rightarrow12 \)
- \( k = 0\rightarrow13 \)
- \( l = 0\rightarrow18 \)

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

\[ w = 1/\left[\sigma(F_o^2) + (0.0319P)^2 + 17.8096P\right] \]
where \( P = (F_o^2 + 2F_c^2)/3 \)
\( (\Delta/\sigma)_{\text{max}} < 0.001 \)
\( \Delta
\rho_{\text{max}} = 2.88 \text{ e Å}^{-3} \)
\( \Delta
\rho_{\text{min}} = -1.95 \text{ e Å}^{-3} \)
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 ($\tilde{A}^2$)

|    | $x$     | $y$     | $z$     | $U_{iso}$/$U_{eq}$ |
|----|---------|---------|---------|-------------------|
| Pb1| 0.12192 | 0.13042 | 0.11904 | 0.01318 (10)      |
| Pb2| 0.25685 | 0.51903 | 0.01540 | 0.01842 (11)      |
| Pb3| 0.37507 | 0.36874 | 0.38134 | 0.01409 (10)      |
| I1 | 0.00670 | 0.23313 | 0.36046 | 0.00838 (13)      |
| I2 | 0.50186 | 0.24738 | 0.14267 | 0.00735 (13)      |
| O1 | 0.0343  | 0.3332  | 0.0015  | 0.0110 (14)*      |
| O2 | 0.0389  | 0.7922  | 0.2811  | 0.0125 (15)*      |
| O3 | 0.0908  | 0.4046  | 0.4090  | 0.0122 (15)*      |
| O4 | 0.1088  | 0.5549  | 0.1797  | 0.0178 (17)*      |
| O5 | 0.1831  | 0.8100  | 0.1159  | 0.0129 (15)*      |
| O6 | 0.1842  | 0.1429  | 0.3433  | 0.0117 (15)*      |
| O7 | 0.3161  | 0.3260  | 0.1618  | 0.0121 (15)*      |
| O8 | 0.4502  | 0.0832  | 0.0785  | 0.0121 (15)*      |
| O9 | 0.4802  | 0.3391  | 0.0121  | 0.0144 (16)*      |
| O10| 0.5297  | 0.1474  | 0.2794  | 0.0145 (16)*      |
| O11| 0.6146  | 0.3912  | 0.2170  | 0.0158 (16)*      |
| O12| 0.6856  | 0.1562  | 0.1172  | 0.0182 (17)*      |

Atomic displacement parameters ($\tilde{A}^2$)

|    | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|----|----------|----------|----------|----------|----------|----------|
| 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) | -0.00003 (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 ($\tilde{A}$, °)

| Pb1—O1     | 2.433 (7) | Pb3—O9   | 2.599 (8) |
| Pb1—O7     | 2.493 (8) | Pb3—O6   | 2.678 (8) |
| Pb1—O2     | 2.577 (8) | Pb3—O12  | 2.704 (8) |
| Pb1—O3     | 2.685 (7) | Pb3—O8   | 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     | 2.888 (8) | I1—O6    | 1.845 (8) |
Pb1—O5\textsuperscript{iii} 3.004 (7) I1—O3 1.860 (7)
Pb2—O7 2.564 (7) I1—O2\textsuperscript{i} 1.861 (7)
Pb2—O9 2.606 (8) I1—O1\textsuperscript{vi} 1.877 (7)
Pb2—O1 2.609 (7) I1—O5\textsuperscript{i} 1.920 (8)
Pb2—O6\textsuperscript{ii} 2.638 (7) I1—O4\textsuperscript{i} 1.956 (8)
Pb2—O4 2.714 (8) I2—O11 1.820 (8)
Pb2—O9\textsuperscript{iv} 2.777 (8) I2—O9 1.850 (8)
Pb2—O1\textsuperscript{v} 2.915 (7) I2—O8 1.855 (7)
Pb2—O5 3.099 (8) I2—O7 1.874 (8)
Pb3—O8\textsuperscript{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\textsuperscript{i} 73.6 (2) O12—I2—O10 90.0 (3)
O7—Pb1—O2\textsuperscript{i} 84.6 (2) I1\textsuperscript{ii}—O1—Pb1 108.2 (3)
O1—Pb1—O3\textsuperscript{ii} 61.5 (2) I1\textsuperscript{ii}—O1—Pb2 103.7 (3)
O7—Pb1—O3\textsuperscript{ii} 102.0 (2) Pb1—O1—Pb2 108.0 (3)
O2\textsuperscript{i}—Pb1—O3\textsuperscript{ii} 129.7 (2) I1\textsuperscript{ii}—O1—Pb2\textsuperscript{v} 97.4 (3)
O1—Pb1—O8 102.0 (2) Pb1—O1—Pb2\textsuperscript{v} 125.6 (3)
O7—Pb1—O8 61.3 (2) Pb2—O1—Pb2\textsuperscript{v} 111.1 (2)
O2\textsuperscript{i}—Pb1—O8 144.9 (2) I1\textsuperscript{ii}—O1—Pb3\textsuperscript{ii} 57.6 (2)
O3\textsuperscript{ii}—Pb1—O8 70.3 (2) Pb1—O1—Pb3\textsuperscript{ii} 73.14 (18)
O1—Pb1—O6 125.2 (2) Pb2—O1—Pb3\textsuperscript{ii} 73.13 (17)
O7—Pb1—O6 75.7 (2) Pb2\textsuperscript{v}—O1—Pb3\textsuperscript{ii} 154.3 (2)
O2\textsuperscript{i}—Pb1—O6 59.4 (2) I1\textsuperscript{iii}—O2—Pb1\textsuperscript{iv} 106.0 (3)
O3\textsuperscript{ii}—Pb1—O6 170.7 (2) I1\textsuperscript{iii}—O2—Pb2\textsuperscript{iv} 156.0 (4)
O8—Pb1—O6 101.0 (2) Pb1\textsuperscript{iv}—O2—Pb2\textsuperscript{iv} 97.4 (2)
O1—Pb1—O3\textsuperscript{i} 109.8 (2) I1\textsuperscript{iii}—O2—Pb1\textsuperscript{i} 75.1 (2)
O7—Pb1—O3\textsuperscript{i} 174.4 (2) Pb1\textsuperscript{iv}—O2—Pb1\textsuperscript{i} 155.4 (3)
O2\textsuperscript{i}—Pb1—O3\textsuperscript{i} 91.7 (2) Pb2\textsuperscript{v}—O2—Pb1\textsuperscript{i} 86.03 (16)
O3\textsuperscript{ii}—Pb1—O3\textsuperscript{i} 83.5 (2) I1\textsuperscript{iii}—O2—Pb3\textsuperscript{iii} 62.0 (2)
O8—Pb1—O3\textsuperscript{i} 121.6 (2) Pb1\textsuperscript{iv}—O2—Pb3\textsuperscript{iii} 78.83 (19)
O6—Pb1—O3\textsuperscript{i} 98.8 (2) Pb2\textsuperscript{v}—O2—Pb3\textsuperscript{iii} 129.9 (2)
O1—Pb1—O5\textsuperscript{iii} 141.7 (2) Pb1\textsuperscript{iv}—O2—Pb3\textsuperscript{iii} 80.39 (14)
O7—Pb1—O5\textsuperscript{iii} 126.3 (2) I1—O3—Pb3 104.5 (3)
O2—Pb1—O5\textsuperscript{iii} 134.1 (2) I1—O3—Pb1\textsuperscript{vi} 99.4 (3)
O3\textsuperscript{ii}—Pb1—O5\textsuperscript{iii} 81.0 (2) Pb3—O3—Pb1\textsuperscript{vi} 104.8 (3)
O8—Pb1—O5\textsuperscript{iii} 70.2 (2) I1—O3—Pb1\textsuperscript{vi} 106.8 (3)
O6—Pb1—O5\textsuperscript{iii} 93.0 (2) Pb3—O3—Pb1\textsuperscript{vi} 138.4 (3)
O3\textsuperscript{i}—Pb1—O5\textsuperscript{iii} 54.4 (2) Pb1\textsuperscript{v}—O3—Pb1\textsuperscript{viii} 96.5 (2)
O7—Pb2—O9 61.2 (2) I1\textsuperscript{vii}—O4—Pb2 102.2 (3)
O7—Pb2—O1 69.1 (2) I1\textsuperscript{vii}—O4—Pb3 146.5 (3)
O9—Pb2—O1 99.3 (2) Pb2—O4—Pb3 98.2 (2)
O7—Pb2—O6\textsuperscript{ii} 101.7 (2) I1\textsuperscript{vii}—O4—Pb1\textsuperscript{viii} 71.6 (2)
O9—Pb2—O6\textsuperscript{ii} 72.2 (2) Pb2—O4—Pb1\textsuperscript{viii} 173.4 (3)
O1—Pb2—O6\textsuperscript{ii} 60.6 (2) Pb3—O4—Pb1\textsuperscript{viii} 88.39 (17)
O7—Pb2—O4 65.3 (2) I1\textsuperscript{vii}—O4—Pb2\textsuperscript{v} 68.3 (2)
O9—Pb2—O4 125.5 (2) Pb2—O4—Pb2\textsuperscript{v} 87.5 (2)
O1—Pb2—O4 69.6 (2)  Pb3—O4—Pb2v 139.5 (2)
O6ii—Pb2—O4 129.6 (2)  Pb1vi—O4—Pb2v 87.94 (18)
O7—Pb2—O9iv 110.9 (2)  I1viii—O4—Pb1 143.6 (3)
O9—Pb2—O9iv 67.9 (3)  Pb2—O4—Pb1 72.12 (18)
O1—Pb2—O9iv 163.1 (2)  Pb3—O4—Pb1 68.44 (14)
O6ii—Pb2—O9iv 103.9 (2)  Pb1iv—O4—Pb1 111.3 (2)
O4—Pb2—O9iv 126.5 (2)  Pb2v—O4—Pb1 75.48 (15)
O7—Pb2—O1v 115.8 (2)  I1viii—O5—Pb1x 101.0 (3)
O9—Pb2—O1v 167.4 (2)  I1viii—O5—Pb2 90.7 (3)
O1—Pb2—O1v 68.9 (2)  Pb1i—O5—Pb2 155.4 (3)
O6ii—Pb2—O1v 97.3 (2)  I1viii—O5—Pb1v 69.1 (2)
O4—Pb2—O1v 55.7 (2)  Pb1i—O5—Pb1v 75.90 (16)
O9iv—Pb2—O1v 122.7 (2)  Pb2—O5—Pb1v 88.49 (18)
O7—Pb2—O5 109.1 (2)  I1viii—O5—Pb3vii 163.1 (3)
O9—Pb2—O5 141.5 (2)  Pb1i—O5—Pb3vii 92.88 (19)
O1—Pb2—O5 112.0 (2)  Pb2—O5—Pb3vii 80.25 (17)
O6ii—Pb2—O5 142.9 (2)  Pb1—O5—Pb3vii 124.4 (2)
O4—Pb2—O5 53.0 (2)  I1—O6—Pb2i 103.6 (3)
O9iv—Pb2—O5 84.2 (2)  I1—O6—Pb3 99.4 (3)
O1v—Pb2—O5 50.8 (2)  Pb2v—O6—Pb3 103.9 (3)
O8vi—Pb3—O3 76.1 (3)  I1—O6—Pb1 97.6 (3)
O8vi—Pb3—O9vi 61.9 (2)  Pb2v—O6—Pb1 142.8 (3)
O3—Pb3—O9vi 104.1 (2)  Pb3—O6—Pb1 102.2 (2)
O8vi—Pb3—O6 105.1 (2)  I2—O7—Pb1 107.0 (3)
O3—Pb3—O6 62.2 (2)  I2—O7—Pb2 103.7 (3)
O9vi—Pb3—O6 71.7 (2)  Pb1—O7—Pb2 107.6 (3)
O8vi—Pb3—O12vii 78.7 (2)  I2—O7—Pb3 100.6 (3)
O3—Pb3—O12vii 70.9 (3)  Pb1—O7—Pb3 108.2 (3)
O9vi—Pb3—O12vii 140.0 (2)  Pb2—O7—Pb3 127.7 (3)
O6—Pb3—O12vii 129.8 (2)  I2—O7—Pb3 56.96 (19)
O8vi—Pb3—O8vii 75.7 (3)  Pb1—O7—Pb3 72.30 (17)
O3—Pb3—O8vii 123.1 (2)  Pb2—O7—Pb3i 73.10 (17)
O9vi—Pb3—O8vii 104.4 (2)  Pb3—O7—Pb3i 154.9 (3)
O6—Pb3—O8vii 174.5 (2)  I2—O8—Pb3i 104.5 (3)
O12vii—Pb3—O8vii 55.7 (2)  I2—O8—Pb1 99.1 (3)
O8vi—Pb3—O7 174.9 (2)  Pb3v—O8—Pb1 103.7 (3)
O3—Pb3—O7 99.1 (2)  I2—O8—Pb3i 104.1 (3)
O9vi—Pb3—O7 121.4 (2)  Pb3v—O8—Pb3iv 104.3 (3)
O6—Pb3—O7 73.5 (2)  Pb1—O8—Pb3iv 137.3 (3)
O12vii—Pb3—O7 98.4 (2)  I2—O9—Pb3iv 102.0 (3)
O8vi—Pb3—O7 106.2 (2)  I2—O9—Pb2 102.9 (3)
O8vi—Pb3—O10 127.3 (2)  Pb3iv—O9—Pb2 107.1 (3)
O3—Pb3—O10 134.0 (2)  I2—O9—Pb2iv 112.6 (4)
O9vi—Pb3—O10 68.2 (2)  Pb3iv—O9—Pb2iv 118.5 (3)
O6—Pb3—O10 72.9 (2)  Pb2—O9—Pb2iv 112.1 (3)
O12vii—Pb3—O10 143.8 (2)  I2—O10—Pb3 95.4 (3)
O8vi—Pb3—O10 102.3 (2)  I2—O10—Pb2iv 148.8 (4)
O7—Pb3—O10 57.2 (2)  Pb3—O10—Pb2iv 98.9 (2)
Hydrogen-bond geometry (Å)

| D—H···A     | D···A     |
|-------------|-----------|
| O4···O7     | 2.849 (11)|
| O4···O2i    | 2.849 (11)|
| O5···O11vi  | 2.634 (11)|
| O10···O11vi | 2.675 (11)|
| O12···O2xi  | 2.852 (11)|

Symmetry codes: (i) −x, y + 1/2, −z + 1/2; (ii) x, −y + 1/2, z; (iii) x, −y + 1/2, 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; (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.