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# Dilead(II) oxyhydroxide bromide

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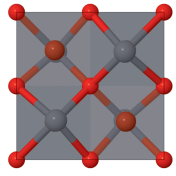
**Structural data:** full structural data are available from iucrdata.iucr.org

The crystal structure of the title compound, $Pb_2O(OH)Br$, has been determined by single-crystal X-ray diffraction. The compound crystallizes in the tetragonal space group $P4_2/mcm$. The structure consists of $[PbO_4Br_4]$ square antiprisms, which form square layers in the *ab* plane and are stacked alternately along the *c*-axis direction. The hydroxide site is assigned on the basis of bond-valence-sum calculations.

3D view



## Structure description

Lead oxyhalides comprise a diverse family of compounds with a variety of crystal structures. Among them, $Pb_2O(OH)Cl$ (Turner *et al.*, 2015), $Pb_2O(OH)I$ (Siidra *et al.*, 2013), and $Pb_3O_2(OH)Br$ (Krivovichev & Burns, 2001) are oxide–hydroxide halides related to the title compound. In this work, we report the crystal structure of $Pb_2O(OH)Br$ determined by single-crystal X-ray diffraction.

The title compound crystallizes in the tetragonal space group $P4_2/mcm$. We note that the crystal structure of $Pb_2O(OH)Br$ is different from the ones of $Pb_2O(OH)Cl$ (space group *Pnma*) and $Pb_2O(OH)I$ (space group *C*2/*m*). The asymmetric unit contains one Pb site, three O sites (O1, O2 and O3), and one Br site. The divalent Pb cation is coordinated in a square-antiprismatic mode by four O and four Br atoms (Fig. 1 and Table 1). The $O_4$ and $Br_4$ squares are parallel, rotated by 45° relative to each other, with the $Br_4$ square having an edge length approximately $\sqrt{2}$ times that of the $O_4$ square. The face-sharing square antiprisms form square layers in the *ab* plane (Fig. 2). These layers with opposite orientations are alternately stacked along the *c* axis. Consequently, a Br square layer and an O square layer are formed alternately along the *c* axis with 45° relative rotation. The Br atom is surrounded by eight Pb cations in a distorted cubic arrangement, while each of the O atoms is surrounded by four Pb cations in a distorted tetrahedral mode (Fig. 3).

Charge neutrality requires the presence of one H atom per formula unit, although the H atom could not be located from difference Fourier maps owing to the presence of the heavy Pb atom. Bond-valence-sum (BVS) calculations (Brown, 2002; Brese & O'Keeffe, 1991) were therefore carried out to identify the hydroxide site (Table 2). Consistent with

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Pb—Br$^{i}$ | 3.3741 (15) | Pb—O1 | 2.3101 (6) |
| Pb—Br | 3.3741 (15) | Pb—O2$^{iv}$ | 2.4291 (3) |
| Pb—Br$^{ii}$ | 3.6154 (15) | Pb—O2 | 2.4291 (3) |
| Pb—Br$^{iii}$ | 3.6154 (15) | Pb—O3*H* | 2.5426 (6) |

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

**Table 2**
Atomic sites (multiplicity and Wyckoff letter), site symmetries, and bond-valence sums (in valence units).

| | | |
|---|---|---|
| Pb (8 *o*) | *m* | 2.16 |
| Br (4 *j*) | *mm*2 | 0.84 |
| O1 (2 *d*) | $\bar{4}2m$ | 2.34 |
| O2 (4 *e*) | 222 | 1.70 |
| O3*H* (2 *b*) | $\bar{4}2m$ | 1.25 |

the longest Pb—O3 bond length, O3 exhibits the lowest BVS value, supporting its assignment as the hydroxide site. We note that the O2 site cannot be assigned as the hydroxide site because its multiplicity is twice that of the O1 and O3 sites. The off-centered coordination around the $Pb^{2+}$ cation reflects the stereochemical activity of its $6s^2$ electron lone pair.

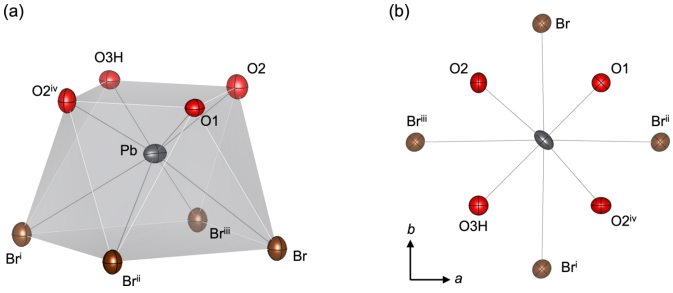



**Figure 1**
(*a*) Coordination environment of the Pb atom. Displacement ellipsoids are drawn at the 50% probability level; (*b*) the $[PbBr_4O_4]$ coordination polyhedron viewed along the *c* axis. [Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, -y+1, -z$; (iii) $-x, -y+1, -z$; (iv) $y, -x, -z+\frac{1}{2}$.]

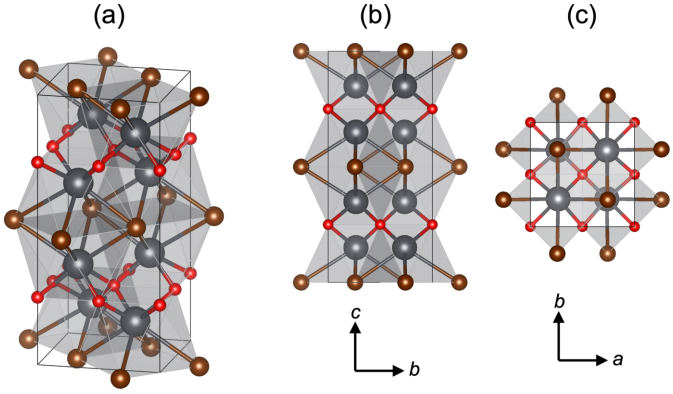



**Figure 2**
The crystal structure of $Pb_2O(OH)Br$ with polyhedral representation. (*a*) Perspective view, projections along (*b*) the *a* and (*c*) the *c* axes.

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $Pb_2O(OH)Br$ |
| $M_r$ | 526.29 |
| Crystal system, space group | Tetragonal, $P4_2/mcm$ |
| Temperature (K) | 296 |
| *a*, *c* (Å) | 5.8161 (3), 12.8870 (13) |
| *V* (Å$^3$) | 435.93 (6) |
| *Z* | 4 |
| Radiation type | Mo *Kα* |
| $\mu$ (mm$^{-1}$) | 86.13 |
| Crystal size (mm) | 0.10 × 0.08 × 0.01 |
| | |
| Data collection | |
| Diffractometer | XtaLAB AFC11 (RCD3): quarter-chi single |
| Absorption correction | Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023) |
| $T_{min}$, $T_{max}$ | 0.542, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 9139, 705, 460 |
| $R_{int}$ | 0.062 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.882 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.044, 0.131, 1.12 |
| No. of reflections | 705 |
| No. of parameters | 18 |
| H-atom treatment | H-atom parameters not defined |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 6.55, −4.22 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *VESTA* (Momma & Izumi, 2011), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

## Synthesis and crystallization

Single crystals of $Pb_2O(OH)Br$ were obtained serendipitously during attempts to synthesize the Br analogues of diaboleite, $Pb_2CuCl_2(OH)_4$, and cumengeite, $Pb_{21}Cu_{20}Cl_{42}(OH)_{40}{\cdot}6H_2O$, (Alun Humphreys *et al.*, 1980) under hydrothermal conditions. Pb wire (0.6 g, Nilaco), $CuBr_2$ (0.2 g, FUJIFILM Wako), and 5 wt% aqueous ammonia (8 ml) were placed in a polymer-lined stainless-steel autoclave and heated at 533 K for 24 h. Yellow, plate-like crystals grew on the surface of the Pb wire, and a suitable crystal was selected for the single-crystal X-ray diffraction measurements.

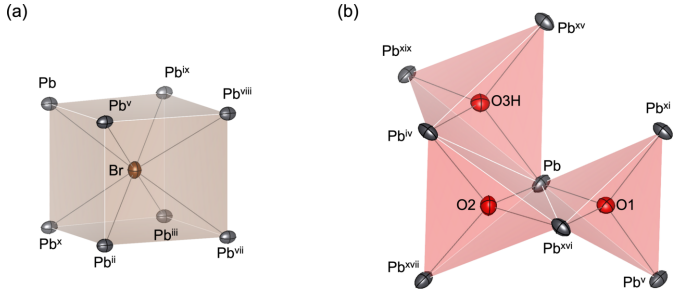



**Figure 3**
Coordination environments of the (*a*) Br and (*b*) O atoms. Ellipsoids are drawn at the 50% probability level. [Symmetry codes: (ii) $-x+1, -y+1, -z$; (iii) $-x, -y+1, -z$; (iv) $y, -x, -z+\frac{1}{2}$; (v) $-x+1, -y+1, z$; (vi) $y, -x+1, z-\frac{1}{2}$; (vii) $x, y+1, -z$; (viii) $x, y+1, z$; (ix) $-x, -y+1, z$; (*x*) $x, y, -z$; (xi) $y, -x+1, -z+\frac{1}{2}$; (xv) $-y, x, -z+\frac{1}{2}$; (xvi) $-y+1, x, -z+\frac{1}{2}$; (xvii) $-x+1, -y, z$; (xix) $-x, -y, z$.]

## Refinement

Details of data collection and structure refinement are given in Table 3. The largest residual electron-density peaks are located within 0.9 Å of the Pb atom and are attributed to Fourier termination errors associated with the heavy Pb atom.

## Acknowledgements

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# full crystallographic data

*IUCrData* (2026). **11**, x260790 [https://doi.org/10.1107/S241431462600790X]

## Dilead(II) oxyhydroxide bromide

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### Dilead(II) oxyhydroxide bromide

*Crystal data*

$Pb_2O(OH)Br$
$M_r$ = 526.29
Tetragonal, $P4_2/mcm$
$a$ = 5.8161 (3) Å
$c$ = 12.8870 (13) Å
$V$ = 435.93 (6) Å$^3$
$Z$ = 4
$F(000)$ = 860
$D_x$ = 8.019 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2463 reflections
$\theta$ = 3.1–34.3°
$\mu$ = 86.13 mm$^{-1}$
$T$ = 296 K
Plate, yellow
0.10 × 0.08 × 0.01 mm

*Data collection*

XtaLAB AFC11 (RCD3): quarter-chi single diffractometer
Radiation source: Rotating-anode X-ray tube, Rigaku (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2023)
$T_{min}$ = 0.542, $T_{max}$ = 1.000
9139 measured reflections
705 independent reflections
460 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.062
$\theta_{max}$ = 38.8°, $\theta_{min}$ = 3.2°
$h$ = −8→10
$k$ = −10→10
$l$ = −22→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.131
$S$ = 1.12
705 reflections
18 parameters
0 restraints
Primary atom site location: dual
H-atom parameters not defined
$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2 + 26.4764P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 6.55 e Å$^{-3}$
$\Delta\rho_{min}$ = −4.22 e Å$^{-3}$

*Special details*

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

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Pb | 0.26668 (8) | 0.26668 (8) | 0.15022 (4) | 0.01991 (19) |
| Br | 0.2582 (3) | 0.7418 (3) | 0.000000 | 0.0233 (4) |
| O1 | 0.500000 | 0.500000 | 0.250000 | 0.018 (7) |
| O2 | 0.000000 | 0.500000 | 0.250000 | 0.023 (8) |
| O3H | 0.000000 | 0.000000 | 0.250000 | 0.020 (7) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Pb | 0.0215 (2) | 0.0215 (2) | 0.0168 (3) | −0.0095 (3) | −0.00071 (12) | −0.00071 (12) |
| Br | 0.0204 (6) | 0.0204 (6) | 0.0290 (11) | 0.0022 (15) | 0.000 | 0.000 |
| O1 | 0.020 (10) | 0.020 (10) | 0.015 (16) | 0.000 | 0.000 | 0.000 |
| O2 | 0.016 (10) | 0.024 (12) | 0.03 (2) | 0.000 | 0.000 | 0.000 |
| O3H | 0.022 (11) | 0.022 (11) | 0.017 (17) | 0.000 | 0.000 | 0.000 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Pb—Br$^{i}$ | 3.3741 (15) | Pb—O1 | 2.3101 (6) |
| Pb—Br | 3.3741 (15) | Pb—O2$^{iv}$ | 2.4291 (3) |
| Pb—Br$^{ii}$ | 3.6154 (15) | Pb—O2 | 2.4291 (3) |
| Pb—Br$^{iii}$ | 3.6154 (15) | Pb—O3H | 2.5426 (6) |
| | | | |
| Br—Pb—Br$^{i}$ | 72.22 (6) | Pb—O1—Pb$^{ix}$ | 58.337 (6) |
| Br$^{iii}$—Pb—Br$^{ii}$ | 71.96 (6) | Pb$^{xiv}$—O1—Pb$^{xvii}$ | 94.056 (3) |
| Br—Pb—Br$^{iii}$ | 72.039 (8) | Pb$^{xv}$—O1—Pb$^{viii}$ | 94.056 (4) |
| Br$^{i}$—Pb—Br$^{ii}$ | 72.039 (8) | Pb—O2—Pb$^{xi}$ | 100.64 (3) |
| Br—Pb—Br$^{ii}$ | 112.590 (16) | Pb$^{xviii}$—O2—Pb$^{xix}$ | 51.065 (2) |
| Br$^{i}$—Pb—Br$^{iii}$ | 112.590 (16) | Pb$^{xviii}$—O2—Pb$^{xx}$ | 92.740 (7) |
| O1—Pb—Br | 81.191 (16) | Pb$^{viii}$—O2—Pb$^{iv}$ | 143.039 (7) |
| O1—Pb—Br$^{i}$ | 81.191 (16) | Pb$^{xvi}$—O2—Pb$^{viii}$ | 92.740 (7) |
| O1—Pb—Br$^{ii}$ | 143.33 (3) | Pb$^{ix}$—O2—Pb$^{xx}$ | 55.085 (16) |
| O1—Pb—Br$^{iii}$ | 143.33 (3) | Pb$^{xv}$—O2—Pb$^{iv}$ | 63.525 (18) |
| O1—Pb—O2 | 75.655 (14) | Pb—O2—Pb$^{xv}$ | 112.08 (3) |
| O1—Pb—O2$^{iv}$ | 75.655 (14) | Pb$^{xi}$—O2—Pb$^{viii}$ | 55.085 (16) |
| O1—Pb—O3H | 115.80 (2) | Pb$^{xviii}$—O2—Pb$^{viii}$ | 118.901 (6) |
| O2—Pb—Br$^{iii}$ | 75.63 (3) | Pb—O2—Pb$^{xx}$ | 148.277 (12) |
| O2$^{iv}$—Pb—Br$^{ii}$ | 75.63 (3) | Pb$^{xi}$—O2—Pb$^{xv}$ | 116.07 (2) |
| O2$^{iv}$—Pb—Br | 146.59 (3) | Pb$^{xix}$—O2—Pb$^{xx}$ | 143.039 (7) |
| O2—Pb—Br$^{i}$ | 146.59 (3) | Pb$^{xi}$—O2—Pb$^{iv}$ | 99.647 (17) |
| O2—Pb—Br$^{ii}$ | 138.29 (3) | Pb$^{v}$—O2—Pb$^{iv}$ | 92.740 (7) |
| O2$^{iv}$—Pb—Br$^{iii}$ | 138.29 (3) | Pb—O2—Pb$^{ix}$ | 116.07 (2) |
| O2—Pb—Br | 80.62 (3) | Pb$^{xxi}$—O2—Pb$^{xix}$ | 92.740 (7) |
| O2$^{iv}$—Pb—Br$^{i}$ | 80.62 (3) | Pb—O2—Pb$^{viii}$ | 99.647 (17) |
| O2$^{iv}$—Pb—O2 | 115.67 (2) | Pb$^{ix}$—O2—Pb$^{viii}$ | 63.525 (19) |
| O2—Pb—O3H | 71.552 (14) | Pb$^{xi}$—O2—Pb$^{ix}$ | 112.08 (3) |

| | | | |
|---|---|---|---|
| O2$^{iv}$—Pb—O3H | 71.552 (14) | Pb$^{xxi}$—O2—Pb$^{viii}$ | 95.585 (7) |
| O3H—Pb—Br$^{ii}$ | 75.368 (15) | Pb$^{xix}$—O2—Pb$^{viii}$ | 149.527 (14) |
| O3H—Pb—Br$^{iii}$ | 75.368 (15) | Pb$^{xv}$—O2—Pb$^{xx}$ | 99.647 (17) |
| O3H—Pb—Br | 141.34 (3) | Pb$^{xv}$—O2—Pb$^{ix}$ | 100.64 (3) |
| O3H—Pb—Br$^{i}$ | 141.34 (3) | Pb$^{v}$—O2—Pb$^{xx}$ | 95.585 (8) |
| Pb$^{v}$—Br—Pb$^{vi}$ | 124.37 (4) | Pb$^{xxi}$—O2—Pb$^{xx}$ | 51.065 (2) |
| Pb$^{i}$—Br—Pb$^{vii}$ | 72.180 (8) | Pb—O2—Pb$^{iv}$ | 55.085 (16) |
| Pb$^{viii}$—Br—Pb$^{ix}$ | 74.70 (4) | Pb—O2—Pb$^{xviii}$ | 106.011 (17) |
| Pb—Br—Pb$^{vi}$ | 124.37 (4) | Pb$^{xviii}$—O2—Pb$^{iv}$ | 95.585 (7) |
| Pb$^{x}$—Br—Pb$^{ix}$ | 107.961 (8) | Pb$^{xvi}$—O2—Pb$^{iv}$ | 51.065 (2) |
| Pb$^{iii}$—Br—Pb$^{vi}$ | 58.241 (14) | Pb$^{xix}$—O2—Pb$^{iv}$ | 48.630 (9) |
| Pb$^{x}$—Br—Pb$^{vii}$ | 112.591 (17) | Pb$^{xi}$—O2—Pb$^{xviii}$ | 153.350 (11) |
| Pb$^{ix}$—Br—Pb$^{vi}$ | 122.98 (3) | Pb$^{xi}$—O2—Pb$^{v}$ | 53.030 (17) |
| Pb—Br—Pb$^{viii}$ | 112.590 (16) | Pb$^{v}$—O2—Pb$^{xix}$ | 118.901 (6) |
| Pb$^{v}$—Br—Pb$^{xi}$ | 54.357 (14) | Pb$^{xv}$—O2—Pb$^{v}$ | 153.350 (11) |
| Pb$^{i}$—Br—Pb$^{ix}$ | 176.910 (14) | Pb$^{xv}$—O2—Pb$^{xviii}$ | 53.030 (17) |
| Pb—Br—Pb$^{xi}$ | 54.357 (14) | Pb$^{ix}$—O2—Pb$^{v}$ | 106.011 (17) |
| Pb$^{viii}$—Br—Pb$^{iii}$ | 108.04 (6) | Pb$^{xv}$—O2—Pb$^{viii}$ | 148.277 (12) |
| Pb$^{x}$—Br—Pb$^{viii}$ | 176.910 (15) | Pb$^{xviii}$—O2—Pb$^{v}$ | 147.940 (14) |
| Pb—Br—Pb$^{vii}$ | 176.910 (15) | Pb$^{ix}$—O2—Pb$^{xviii}$ | 55.377 (19) |
| Pb$^{v}$—Br—Pb$^{viii}$ | 72.179 (8) | Pb$^{xvi}$—O2—Pb$^{v}$ | 47.339 (10) |
| Pb$^{i}$—Br—Pb$^{viii}$ | 107.961 (8) | Pb$^{v}$—O2—Pb$^{viii}$ | 51.065 (2) |
| Pb$^{iii}$—Br—Pb$^{vii}$ | 74.70 (4) | Pb—O2—Pb$^{xxi}$ | 153.350 (11) |
| Pb$^{ix}$—Br—Pb$^{xi}$ | 58.241 (14) | Pb—O2—Pb$^{xvi}$ | 53.029 (17) |
| Pb$^{x}$—Br—Pb$^{xi}$ | 124.37 (4) | Pb$^{xi}$—O2—Pb$^{xxi}$ | 106.011 (17) |
| Pb$^{vii}$—Br—Pb$^{ix}$ | 108.04 (6) | Pb$^{xi}$—O2—Pb$^{xx}$ | 63.525 (19) |
| Pb$^{v}$—Br—Pb$^{iii}$ | 176.910 (15) | Pb$^{xv}$—O2—Pb$^{xxi}$ | 55.377 (19) |
| Pb$^{x}$—Br—Pb$^{vi}$ | 54.357 (14) | Pb$^{xi}$—O2—Pb$^{xvi}$ | 55.377 (19) |
| Pb$^{i}$—Br—Pb$^{xi}$ | 124.37 (4) | Pb$^{ix}$—O2—Pb$^{xxi}$ | 53.030 (17) |
| Pb$^{i}$—Br—Pb$^{vi}$ | 54.357 (14) | Pb$^{xvi}$—O2—Pb$^{xx}$ | 118.901 (6) |
| Pb—Br—Pb$^{iii}$ | 107.961 (8) | Pb$^{xviii}$—O2—Pb$^{xxi}$ | 47.339 (9) |
| Pb$^{viii}$—Br—Pb$^{vi}$ | 122.98 (3) | Pb$^{xv}$—O2—Pb$^{xvi}$ | 106.011 (17) |
| Pb$^{iii}$—Br—Pb$^{xi}$ | 122.98 (3) | Pb$^{xvi}$—O2—Pb$^{xxi}$ | 147.940 (14) |
| Pb$^{x}$—Br—Pb$^{v}$ | 107.78 (6) | Pb$^{viii}$—O2—Pb$^{xx}$ | 48.630 (8) |
| Pb$^{v}$—Br—Pb$^{ix}$ | 112.591 (17) | Pb$^{v}$—O2—Pb$^{xxi}$ | 146.116 (7) |
| Pb$^{x}$—Br—Pb$^{i}$ | 69.33 (4) | Pb$^{ix}$—O2—Pb$^{xvi}$ | 153.350 (11) |
| Pb$^{v}$—Br—Pb$^{vii}$ | 107.961 (8) | Pb—O2—Pb$^{xix}$ | 63.525 (19) |
| Pb$^{v}$—Br—Pb$^{i}$ | 70.02 (4) | Pb$^{ix}$—O2—Pb$^{iv}$ | 148.277 (12) |
| Pb$^{i}$—Br—Pb$^{iii}$ | 112.591 (17) | Pb$^{xi}$—O2—Pb$^{xix}$ | 148.277 (12) |
| Pb$^{x}$—Br—Pb | 70.02 (4) | Pb$^{xviii}$—O2—Pb$^{xvi}$ | 146.116 (7) |
| Pb—Br—Pb$^{ix}$ | 72.179 (8) | Pb$^{xv}$—O2—Pb$^{xix}$ | 55.085 (16) |
| Pb$^{v}$—Br—Pb | 69.33 (4) | Pb$^{xxi}$—O2—Pb$^{iv}$ | 118.901 (6) |
| Pb$^{x}$—Br—Pb$^{iii}$ | 72.180 (8) | Pb$^{ix}$—O2—Pb$^{xix}$ | 99.647 (17) |
| Pb$^{i}$—Br—Pb | 107.78 (6) | Pb—O2—Pb$^{v}$ | 55.377 (19) |
| Pb$^{viii}$—Br—Pb$^{vii}$ | 64.75 (3) | Pb$^{xvi}$—O2—Pb$^{xix}$ | 95.585 (7) |
| Pb$^{vi}$—Br—Pb$^{xi}$ | 178.24 (6) | Pb$^{xx}$—O2—Pb$^{iv}$ | 149.527 (14) |
| Pb$^{iii}$—Br—Pb$^{ix}$ | 64.75 (3) | Pb—O3H—Pb$^{xix}$ | 119.24 (3) |
| Pb$^{viii}$—Br—Pb$^{xi}$ | 58.241 (14) | Pb$^{ix}$—O3H—Pb$^{xvii}$ | 121.353 (13) |

| | | | |
|---|---|---|---|
| $Pb^{vii}$—Br—$Pb^{vi}$ | 58.241 (14) | $Pb^{xvi}$—O3H—$Pb^{ix}$ | 94.262 (4) |
| $Pb^{vii}$—Br—$Pb^{xi}$ | 122.98 (3) | $Pb^{xviii}$—O3H—$Pb^{xvii}$ | 148.360 (14) |
| Pb—O1—$Pb^{v}$ | 112.35 (3) | $Pb^{xi}$—O3H—$Pb^{xviii}$ | 94.262 (4) |
| $Pb^{iv}$—O1—$Pb^{viii}$ | 147.401 (13) | $Pb^{iv}$—O3H—$Pb^{ix}$ | 152.875 (9) |
| $Pb^{xii}$—O1—$Pb^{xiii}$ | 94.056 (4) | $Pb^{xv}$—O3H—$Pb^{xvii}$ | 152.875 (9) |
| $Pb^{xiii}$—O1—$Pb^{viii}$ | 116.547 (13) | Pb—O3H—$Pb^{xv}$ | 104.820 (12) |
| $Pb^{xiv}$—O1—$Pb^{xv}$ | 149.152 (13) | $Pb^{xix}$—O3H—$Pb^{xviii}$ | 60.734 (7) |
| $Pb^{xi}$—O1—$Pb^{xiii}$ | 148.721 (10) | $Pb^{xvi}$—O3H—$Pb^{xviii}$ | 141.606 (14) |
| $Pb^{xvi}$—O1—$Pb^{viii}$ | 148.721 (10) | Pb—O3H—$Pb^{ix}$ | 60.734 (7) |
| Pb—O1—$Pb^{xvi}$ | 108.051 (15) | $Pb^{xix}$—O3H—$Pb^{xv}$ | 104.820 (12) |
| $Pb^{v}$—O1—$Pb^{xv}$ | 148.721 (10) | $Pb^{ii}$—O3H—$Pb^{ix}$ | 148.360 (14) |
| $Pb^{xii}$—O1—$Pb^{xv}$ | 116.547 (13) | $Pb^{xix}$—O3H—$Pb^{xvii}$ | 102.305 (10) |
| Pb—O1—$Pb^{xiii}$ | 103.228 (10) | $Pb^{xxii}$—O3H—$Pb^{xvii}$ | 94.262 (4) |
| $Pb^{v}$—O1—$Pb^{xvi}$ | 108.051 (15) | Pb—O3H—$Pb^{iv}$ | 104.820 (12) |
| $Pb^{xvii}$—O1—$Pb^{xiii}$ | 53.957 (13) | $Pb^{xxiii}$—O3H—$Pb^{ii}$ | 94.262 (4) |
| $Pb^{v}$—O1—$Pb^{viii}$ | 58.337 (6) | Pb—O3H—$Pb^{xviii}$ | 102.305 (10) |
| $Pb^{ix}$—O1—$Pb^{viii}$ | 53.957 (13) | $Pb^{iv}$—O3H—$Pb^{xviii}$ | 152.875 (9) |
| Pb—O1—$Pb^{xi}$ | 108.051 (15) | $Pb^{xix}$—O3H—$Pb^{iv}$ | 104.820 (12) |
| $Pb^{iv}$—O1—$Pb^{xvii}$ | 45.493 (14) | $Pb^{xxiii}$—O3H—$Pb^{xviii}$ | 50.571 (15) |
| Pb—O1—$Pb^{xv}$ | 56.043 (19) | $Pb^{ii}$—O3H—$Pb^{xviii}$ | 121.353 (13) |
| $Pb^{xi}$—O1—$Pb^{xv}$ | 58.337 (6) | $Pb^{xv}$—O3H—$Pb^{ix}$ | 52.101 (18) |
| $Pb^{v}$—O1—$Pb^{xi}$ | 108.051 (15) | $Pb^{xv}$—O3H—$Pb^{iv}$ | 119.24 (3) |
| $Pb^{iv}$—O1—$Pb^{xv}$ | 53.957 (13) | $Pb^{xxii}$—O3H—$Pb^{ix}$ | 141.606 (14) |
| $Pb^{xvii}$—O1—$Pb^{xv}$ | 94.056 (4) | $Pb^{xxiii}$—O3H—$Pb^{ix}$ | 94.262 (4) |
| $Pb^{xvi}$—O1—$Pb^{xiii}$ | 56.044 (19) | Pb—O3H—$Pb^{xvii}$ | 60.734 (7) |
| $Pb^{xvi}$—O1—$Pb^{xi}$ | 112.35 (3) | Pb—O3H—$Pb^{xvi}$ | 52.101 (18) |
| $Pb^{ix}$—O1—$Pb^{xiii}$ | 149.152 (13) | $Pb^{xvi}$—O3H—$Pb^{xvii}$ | 50.571 (15) |
| $Pb^{iv}$—O1—$Pb^{xiii}$ | 94.056 (4) | $Pb^{xi}$—O3H—$Pb^{xvii}$ | 94.262 (4) |
| Pb—O1—$Pb^{viii}$ | 103.228 (10) | $Pb^{ii}$—O3H—$Pb^{xvii}$ | 48.014 (14) |
| Pb—O1—$Pb^{xii}$ | 148.721 (10) | $Pb^{xix}$—O3H—$Pb^{xvi}$ | 152.875 (9) |
| $Pb^{xii}$—O1—$Pb^{viii}$ | 45.493 (14) | $Pb^{xix}$—O3H—$Pb^{xxii}$ | 52.101 (18) |
| $Pb^{xiv}$—O1—$Pb^{viii}$ | 94.056 (4) | $Pb^{xxii}$—O3H—$Pb^{ii}$ | 50.571 (15) |
| $Pb^{xi}$—O1—$Pb^{xvii}$ | 148.721 (10) | $Pb^{xv}$—O3H—$Pb^{xxii}$ | 102.305 (10) |
| $Pb^{v}$—O1—$Pb^{xii}$ | 56.044 (19) | $Pb^{xv}$—O3H—$Pb^{xvi}$ | 102.305 (10) |
| $Pb^{v}$—O1—$Pb^{ix}$ | 103.228 (10) | $Pb^{iv}$—O3H—$Pb^{xxii}$ | 60.734 (7) |
| $Pb^{ix}$—O1—$Pb^{xvii}$ | 116.547 (13) | $Pb^{xv}$—O3H—$Pb^{xviii}$ | 52.101 (18) |
| $Pb^{xvi}$—O1—$Pb^{ix}$ | 148.721 (10) | $Pb^{xvi}$—O3H—$Pb^{xxii}$ | 121.353 (13) |
| $Pb^{xvi}$—O1—$Pb^{xii}$ | 103.228 (10) | $Pb^{iv}$—O3H—$Pb^{xvi}$ | 60.734 (7) |
| $Pb^{xi}$—O1—$Pb^{ix}$ | 56.044 (19) | $Pb^{xi}$—O3H—$Pb^{xxii}$ | 148.360 (14) |
| $Pb^{xvi}$—O1—$Pb^{xv}$ | 103.228 (10) | $Pb^{xxii}$—O3H—$Pb^{xviii}$ | 94.262 (4) |
| $Pb^{xii}$—O1—$Pb^{ix}$ | 94.056 (3) | Pb—O3H—$Pb^{xxiii}$ | 152.876 (9) |
| $Pb^{xi}$—O1—$Pb^{xii}$ | 58.337 (6) | Pb—O3H—$Pb^{xi}$ | 52.101 (18) |
| $Pb^{xiv}$—O1—$Pb^{ix}$ | 147.401 (13) | $Pb^{xix}$—O3H—$Pb^{xxiii}$ | 52.101 (18) |
| $Pb^{ix}$—O1—$Pb^{xv}$ | 45.493 (14) | $Pb^{xix}$—O3H—$Pb^{ix}$ | 102.305 (10) |
| Pb—O1—$Pb^{iv}$ | 56.043 (19) | $Pb^{xv}$—O3H—$Pb^{xxiii}$ | 60.734 (7) |
| Pb—O1—$Pb^{xiv}$ | 148.721 (10) | $Pb^{xix}$—O3H—$Pb^{xi}$ | 152.875 (9) |
| $Pb^{v}$—O1—$Pb^{iv}$ | 148.721 (10) | $Pb^{iv}$—O3H—$Pb^{xxiii}$ | 102.305 (10) |
| $Pb^{v}$—O1—$Pb^{xiii}$ | 58.337 (6) | $Pb^{xi}$—O3H—$Pb^{ix}$ | 50.571 (15) |

| | | | |
|---|---|---|---|
| $Pb^{xvi}$—O1—$Pb^{iv}$ | 58.337 (6) | $Pb^{xvi}$—O3H—$Pb^{xxiii}$ | 148.360 (14) |
| $Pb^{v}$—O1—$Pb^{xiv}$ | 56.044 (19) | $Pb^{xv}$—O3H—$Pb^{xi}$ | 60.734 (7) |
| $Pb^{xi}$—O1—$Pb^{iv}$ | 103.228 (10) | $Pb^{xi}$—O3H—$Pb^{xxiii}$ | 121.353 (13) |
| $Pb^{xiv}$—O1—$Pb^{xiii}$ | 45.493 (14) | $Pb^{xviii}$—O3H—$Pb^{ix}$ | 48.014 (14) |
| $Pb^{xii}$—O1—$Pb^{iv}$ | 149.152 (13) | $Pb^{xxii}$—O3H—$Pb^{xxiii}$ | 48.014 (14) |
| $Pb^{xvi}$—O1—$Pb^{xiv}$ | 58.337 (6) | $Pb^{iv}$—O3H—$Pb^{xi}$ | 102.305 (10) |
| $Pb^{xiv}$—O1—$Pb^{iv}$ | 116.547 (13) | Pb—O3H—$Pb^{ii}$ | 102.305 (10) |
| $Pb^{xv}$—O1—$Pb^{xiii}$ | 147.401 (13) | $Pb^{iv}$—O3H—$Pb^{xvii}$ | 52.101 (18) |
| $Pb^{ix}$—O1—$Pb^{iv}$ | 94.056 (4) | $Pb^{xix}$—O3H—$Pb^{ii}$ | 60.734 (7) |
| $Pb^{xi}$—O1—$Pb^{xiv}$ | 103.228 (10) | $Pb^{xvi}$—O3H—$Pb^{xi}$ | 48.014 (14) |
| Pb—O1—$Pb^{xvii}$ | 58.337 (6) | $Pb^{xv}$—O3H—$Pb^{ii}$ | 152.875 (9) |
| $Pb^{xi}$—O1—$Pb^{viii}$ | 56.044 (19) | $Pb^{xxiii}$—O3H—$Pb^{xvii}$ | 141.606 (14) |
| $Pb^{v}$—O1—$Pb^{xvii}$ | 103.228 (10) | $Pb^{iv}$—O3H—$Pb^{ii}$ | 52.101 (18) |
| $Pb^{xii}$—O1—$Pb^{xiv}$ | 53.957 (13) | Pb—O3H—$Pb^{xxii}$ | 152.876 (9) |
| $Pb^{xvi}$—O1—$Pb^{xvii}$ | 56.044 (19) | $Pb^{xvi}$—O3H—$Pb^{ii}$ | 94.262 (4) |
| $Pb^{xvii}$—O1—$Pb^{viii}$ | 149.152 (13) | $Pb^{xi}$—O3H—$Pb^{ii}$ | 141.606 (14) |
| $Pb^{xii}$—O1—$Pb^{xvii}$ | 147.401 (13) | | |

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, y-1, z$; (iii) $-x, -y+1, -z$; (iv) $y, -x, -z+1/2$; (v) $-x+1, -y+1, z$; (vi) $y, -x+1, z-1/2$; (vii) $x, y+1, -z$; (viii) $x, y+1, z$; (ix) $-x, -y+1, z$; (x) $x, y, -z$; (xi) $y, -x+1, -z+1/2$; (xii) $-y+1, x+1, -z+1/2$; (xiii) $x+1, y, z$; (xiv) $y+1, -x+1, -z+1/2$; (xv) $-y, x, -z+1/2$; (xvi) $-y+1, x, -z+1/2$; (xvii) $-x+1, -y, z$; (xviii) $x-1, y, z$; (xix) $-x, -y, z$; (xx) $-y, x+1, -z+1/2$; (xxi) $y-1, -x+1, -z+1/2$; (xxii) $-y, x-1, -z+1/2$; (xxiii) $y-1, -x, -z+1/2$.