Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

The mineral marrucciite: monoclinic $Hg_3Pb_{16}Sb_{18}S_{46}$

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Received 20 September 2007; accepted 12 October 2007

Key indicators: single-crystal X-ray study; T = 292 K; mean σ(Sb–S) = 0.003 Å; disorder in main residue; R factor = 0.024; wR factor = 0.045; data-to-parameter ratio = 19.3.

Recently, a new mineral species, monoclinic marrucciite, mercury lead antimony sulfide, $Hg_3Pb_{16}Sb_{18}S_{46}$ [Orlandi *et al.* (2007). *Eur. J. Mineral.* **19**, 267–279], was discovered in the Fe—Ba deposit of Buca della Vena, Apuan Alps (Italy). In that report, the crystal structure was refined to R = 0.096. Our new discovery of crystals of this sulfosalt in the Gelnica ore district, situated in the Spišsko-gemerské rudohorie mountain range, Slovak Republic, has now allowed a substantially more precise determination of the crystal structure (R = 0.024). The monoclinic unit cell contains 19 independent cation positions (including two mixed Sb^{III}/Pb^{II} positions) and 23 independent S positions.

Related literature

For the description and relation of the crystal structure of marrucciite to other mineral species and related compounds, see Orlandi *et al.* (2007).

Experimental

Crystal data

$Hg_3Pb_{16}S_{46}Sb_{18}$
M_r = 7583.1
Monoclinic, $C2/m$
a = 48.124 (11) Å
b = 4.1083 (2) Å
c = 23.990 (5) Å
β = 118.76 (2)°
V = 4158.2 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 44.67 mm^{-1}
T = 292 K
0.04 × 0.02 × 0.00 mm

Data collection

Oxford Diffraction XCalibur CCD diffractometer
Absorption correction: analytical Clark & Reid (1995)
T_{min} = 0.198, T_{max} = 0.805
26009 measured reflections
4942 independent reflections
3000 reflections with $I > 3\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.045
S = 0.85
4942 reflections
256 parameters
$\Delta\rho_{max}$ = 1.20 e Å^{-3}
$\Delta\rho_{min}$ = −1.09 e Å^{-3}

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2004); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *JANA2000* (Petricek *et al.*, 2000); molecular graphics: *DIAMOND* 3 (Brandenburg & Putz, 2005); software used to prepare material for publication: *JANA2000*.

This research was supported by an internal project of the Czech Geological Survey (No. 3323), by the Grant Agency of the Czech Republic (No. 202/05/0421), by a project of the Ministry of Culture of the Czech Republic (No. MK00002327201), and by a VEGA grant (No. 1/2028/05).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2073).

References

Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Version 3. Crystal Impact GbR, Bonn, Germany.
Burla, M. C., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). *J. Appl. Cryst.* **36**, 1103.
Clark, R. C. & Reid, J. S. (1995). *Acta Cryst.* A**51**, 887–897.
Orlandi, P., Moëlo, Y., Campostrini, I. & Meerschaut, A. (2007). *Eur. J. Mineral.* **19**, 267–279.
Oxford Diffraction (2004). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd., Abingdon, Oxfordshire, England.
Petříček, V., Dušek, M. & Palatinus, L. (2000). *JANA2000*. Institute of Physics, Praha, Czech Republic.

  doi:10.1107/S1600536806040980

supplementary materials

Acta Cryst. (2007). E**63**, i190 [**doi:10.1107/S1600536806040980**]

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Comment

The structure model proposed by Orlandi *et al.* (2007) is, in basic outline, confirmed; only minor deviations were found. The most striking difference is the absence of 2*b* superstructure diffraction spots, which were observed in the marrucciite from Bucca della Vena. Consequently, no split positions with half-occupancy occur in the structure model proposed in the present paper. Moreover, we present anisotropic displacement parameters for all atoms of the marrucciite structure.

Fig. 1 depicts the projection of the crystal structure of the title compound along *b*. The asymmetric unit contains two Hg^{II} positions, seven pure Pb^{II} positions, eight pure Sb^{III} positions, two mixed Pb^{II}/Sb^{III} positions [refined to $Pb_{0.612\,(5)}Sb_{0.388\,(5)}$ and $Pb_{0.388\,(6)}Sb_{0.612\,(6)}$] and 23 S positions. One of the main characteristics of the structure of this compound is the ordering of Sb atoms to form so-called lone electron pair micelles (Fig. 1, left part) where the stereochemically active lone pairs of electrons on Sb are accommodated.

Experimental

Black needle fragments of the mineral marrucciite were found embedded in a quartz-siderite vein in the Gelnica ore district. They were separated from quartz by means of hydrofluoric acid. Of many fibrous crystals that were examined, only a few were suitable for X-ray single-crystal structure determination.

Refinement

For the disordered sites Pb8/Sb8 and Pb9/Sb9, atoms on the same site were constrained to have identical atomic coordinates and anisotropic displacement parameters, and the site occupancy factores at each site were constrained to sum to unity. No other constraints were apllied. The highest residual peak in the final difference Fourier map was 0.7 Å from Sb1 and the deepest hole 0.44 Å from Sb1.

Figures

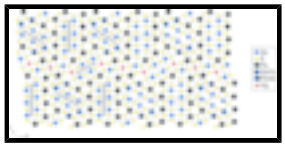

Fig. 1. A projection along *b* of the crystal structure of marrucciite, monoclinic $Hg_3Pb_{16}Sb_{18}S_{48}$. Grey areas (left side of figure) highlight lone electron pair micelles.

(I)

Crystal data

$Hg_3Pb_{16}S_{46}Sb_{18}$ $F_{000} = 6412$

M_r = 7583.1	D_x = 6.055 Mg m^{-3}
Monoclinic, *C*2/*m*	Mo *K*α radiation λ = 0.71069 Å
Hall symbol: -C 2y	Cell parameters from 9276 reflections
a = 48.124 (11) Å	θ = 2.5–26.5º
b = 4.1083 (2) Å	μ = 44.67 mm^{-1}
c = 23.990 (5) Å	*T* = 292 K
β = 118.76 (2)º	Prism, black
V = 4158.2 (15) Å^3	0.04 × 0.02 × 0.002 mm
Z = 2	

Data collection

Oxford Diffraction XCalibur CCD diffractometer	4942 independent reflections
Radiation source: X-ray tube	3000 reflections with $I > 3\sigma(I)$
Monochromator: graphite	R_{int} = 0.052
Detector resolution: 8.3438 pixels mm^{-1}	θ_{max} = 26.6º
T = 292 K	θ_{min} = 2.5º
Rotation method data acquisition using ω scans	*h* = −59→60
Absorption correction: analytical Clark & Reid (1995)	*k* = −5→5
T_{min} = 0.198, T_{max} = 0.805	*l* = −30→30
26009 measured reflections	

Refinement

Refinement on F^2	Weighting scheme based on measured s.u.'s *w* = 1/ ($\sigma^2(I)$ + 0.0004I^2)
$R[F^2 > 2\sigma(F^2)]$ = 0.024	$(\Delta/\sigma)_{max}$ = 0.031
$wR(F^2)$ = 0.045	$\Delta\rho_{max}$ = 1.20 e Å^{-3}
S = 0.85	$\Delta\rho_{min}$ = −1.09 e Å^{-3}
4942 reflections	Extinction correction: none
256 parameters	

Special details

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 are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > n*sigma(F^2) is used only for calculating *R*-factors *etc*. and is not relevant to the choice of reflections for refinement.

The program used for refinement, Jana2000, uses the weighting scheme based on the experimental expectations, see _refine_ls_weighting_details, that does not force S to be one. Therefore the values of S are usually larger then the ones from the *SHELX* program.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Sb1	0.42917 (2)	0	0.01409 (5)	0.0410 (5)	
Sb2	0.29529 (2)	0	0.04272 (5)	0.0326 (4)	
S1	0.04720 (8)	0	0.04514 (15)	0.0315 (15)	
S2	0.34675 (8)	0	0.04430 (13)	0.0183 (14)	
Pb1	0.160743 (13)	0	0.05141 (2)	0.0257 (2)	
S3	0.22457 (8)	0	0.04890 (17)	0.0483 (19)	
S4	0.90931 (7)	0	0.05559 (14)	0.0235 (14)	
Sb3	0.52171 (2)	0	0.08609 (5)	0.0467 (5)	
S5	0.61989 (7)	0	0.08618 (14)	0.0190 (13)	
S6	0.81662 (9)	0	0.13629 (17)	0.053 (2)	
S7	−0.00105 (8)	0	0.13619 (15)	0.0269 (14)	
Sb4	0.74970 (2)	0	0.15156 (4)	0.0228 (4)	
Pb2	0.880439 (12)	0	0.14750 (2)	0.0219 (2)	
S8	0.69971 (7)	0	0.15478 (14)	0.0193 (13)	
Pb3	0.110919 (12)	0	0.16376 (2)	0.0242 (2)	
Sb9	0.464070 (18)	0	0.18486 (4)	0.0415 (4)	0.612 (6)
Pb9	0.46407	0	0.184862	0.0415 (4)	0.388 (6)
S9	0.57042 (7)	0	0.18714 (14)	0.0190 (13)	
S10	0.27134 (8)	0	0.23034 (15)	0.0310 (15)	
Sb5	0.33940 (2)	0	0.24093 (4)	0.0229 (4)	
S11	0.39219 (7)	0	0.24290 (14)	0.0187 (13)	
S12	0.95232 (8)	0	0.24303 (14)	0.0253 (15)	
Pb8	0.052297 (17)	0	0.25068 (3)	0.0319 (4)	0.612 (5)
Sb8	0.052297	0	0.250683	0.0319 (4)	0.388 (5)
S13	0.66387 (7)	0	0.24881 (13)	0.0178 (13)	
Pb4	0.215046 (13)	0	0.26212 (2)	0.0271 (2)	
S14	0.52026 (8)	0	0.27747 (14)	0.0235 (14)	
S15	0.85629 (10)	0	0.32046 (17)	0.052 (2)	
S16	0.12719 (9)	0	0.33026 (16)	0.0437 (18)	
S17	0.76523 (7)	0	0.34565 (13)	0.0194 (13)	
Pb5	0.931045 (13)	0	0.34166 (2)	0.0258 (2)	
Sb6	0.67121 (2)	0	0.35664 (4)	0.0245 (4)	
Pb6	0.313833 (12)	0	0.37726 (2)	0.0259 (2)	
Pb7	0.026006 (13)	0	0.38264 (2)	0.0255 (2)	
S18	0.48083 (7)	0	0.38906 (13)	0.0182 (13)	
S19	0.21658 (8)	0	0.39248 (16)	0.0443 (18)	
S20	0.57670 (8)	0	0.41998 (14)	0.0233 (14)	
Hg1	0.129886 (14)	0	0.43129 (3)	0.0323 (3)	
Sb7	0.76574 (2)	0	0.44928 (4)	0.0293 (5)	
S21	0.86164 (7)	0	0.46313 (13)	0.0178 (13)	
S22	0.94500 (7)	0	0.47995 (14)	0.0188 (13)	
S23	0.30691 (8)	0	0.48875 (15)	0.0297 (15)	
Hg2	0.5	0	0.5	0.0271 (3)	
Sb10	0.59584 (3)	0.0842 (3)	0.53354 (5)	0.0196 (6)	0.5

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.0181 (5)	0.0697 (8)	0.0277 (5)	0	0.0050 (4)	0
Sb2	0.0193 (5)	0.0187 (5)	0.0557 (6)	0	0.0149 (5)	0
S1	0.0209 (19)	0.055 (3)	0.0199 (17)	0	0.0105 (15)	0
S2	0.0292 (19)	0.0127 (17)	0.0171 (16)	0	0.0145 (14)	0
Pb1	0.0381 (3)	0.0180 (3)	0.0249 (3)	0	0.0185 (3)	0
S3	0.017 (2)	0.098 (4)	0.026 (2)	0	0.0077 (16)	0
S4	0.0183 (18)	0.034 (2)	0.0193 (16)	0	0.0097 (14)	0
Sb3	0.0328 (6)	0.0650 (8)	0.0242 (5)	0	−0.0009 (5)	0
S5	0.0168 (17)	0.0203 (18)	0.0176 (16)	0	0.0063 (13)	0
S6	0.018 (2)	0.113 (4)	0.032 (2)	0	0.0153 (17)	0
S7	0.0181 (18)	0.040 (2)	0.0193 (17)	0	0.0068 (14)	0
Sb4	0.0172 (5)	0.0269 (5)	0.0241 (5)	0	0.0098 (4)	0
Pb2	0.0259 (3)	0.0175 (3)	0.0232 (3)	0	0.0127 (2)	0
S8	0.0218 (18)	0.0180 (18)	0.0168 (16)	0	0.0082 (14)	0
Pb3	0.0262 (3)	0.0226 (3)	0.0280 (3)	0	0.0165 (2)	0
Sb9	0.0259 (5)	0.0376 (6)	0.0552 (6)	0	0.0149 (4)	0
Pb9	0.0259 (5)	0.0376 (6)	0.0552 (6)	0	0.0149 (4)	0
S9	0.0142 (17)	0.0216 (19)	0.0180 (16)	0	0.0053 (13)	0
S10	0.0231 (19)	0.050 (3)	0.0181 (17)	0	0.0085 (15)	0
Sb5	0.0252 (5)	0.0250 (5)	0.0225 (5)	0	0.0146 (4)	0
S11	0.0184 (17)	0.0153 (18)	0.0189 (16)	0	0.0062 (14)	0
S12	0.028 (2)	0.034 (2)	0.0205 (17)	0	0.0165 (15)	0
Pb8	0.0472 (5)	0.0259 (4)	0.0341 (4)	0	0.0287 (4)	0
Sb8	0.0472 (5)	0.0259 (4)	0.0341 (4)	0	0.0287 (4)	0
S13	0.0179 (17)	0.0193 (18)	0.0142 (15)	0	0.0062 (13)	0
Pb4	0.0285 (3)	0.0205 (3)	0.0245 (3)	0	0.0065 (2)	0
S14	0.0219 (19)	0.031 (2)	0.0168 (16)	0	0.0088 (14)	0
S15	0.041 (3)	0.090 (4)	0.027 (2)	0	0.0183 (19)	0
S16	0.028 (2)	0.086 (3)	0.0161 (17)	0	0.0094 (16)	0
S17	0.0217 (18)	0.0160 (18)	0.0154 (16)	0	0.0048 (14)	0
Pb5	0.0339 (3)	0.0169 (3)	0.0244 (3)	0	0.0123 (2)	0
Sb6	0.0391 (6)	0.0181 (5)	0.0153 (4)	0	0.0123 (4)	0
Pb6	0.0244 (3)	0.0251 (3)	0.0266 (3)	0	0.0109 (2)	0
Pb7	0.0322 (3)	0.0251 (3)	0.0234 (3)	0	0.0168 (2)	0
S18	0.0179 (17)	0.0174 (18)	0.0181 (16)	0	0.0077 (14)	0
S19	0.019 (2)	0.090 (4)	0.0230 (19)	0	0.0097 (16)	0
S20	0.0218 (19)	0.024 (2)	0.0158 (16)	0	0.0025 (14)	0
Hg1	0.0443 (4)	0.0367 (4)	0.0230 (3)	0	0.0219 (3)	0
Sb7	0.0457 (6)	0.0193 (5)	0.0383 (5)	0	0.0326 (5)	0
S21	0.0189 (17)	0.0179 (18)	0.0141 (15)	0	0.0059 (13)	0
S22	0.0169 (17)	0.0227 (19)	0.0176 (16)	0	0.0090 (14)	0
S23	0.0227 (19)	0.045 (2)	0.0176 (17)	0	0.0063 (15)	0
Hg2	0.0288 (4)	0.0328 (5)	0.0176 (4)	0	0.0096 (3)	0
Sb10	0.0213 (6)	0.0203 (12)	0.0162 (5)	0.0021 (5)	0.0082 (4)	0.0009 (5)

Geometric parameters (Å, °)

Hg1—S21^{i}	2.364 (3)	Sb3—S7^{iv}	2.851 (3)
Hg1—S16	2.364 (4)	Sb3—S7^{v}	2.851 (3)
Hg2—S18	2.360 (3)	Sb3—S9	2.430 (3)
Pb1^{ii}—S2	2.971 (3)	Sb4^{vi}—S3	2.980 (3)
Pb1^{iii}—S2	2.971 (3)	Sb4^{vii}—S3	2.980 (3)
Pb2—S6	2.951 (5)	Sb4—S8	2.444 (4)
Pb2—S11^{iv}	2.921 (2)	Sb4—S10^{iv}	2.6424 (19)
Pb2—S11^{v}	2.921 (2)	Sb4—S10^{v}	2.6424 (19)
Pb3^{iv}—S5	2.940 (3)	Sb5—S11	2.519 (4)
Pb3^{v}—S5	2.940 (3)	Sb5—S15^{vi}	2.651 (2)
Pb5—S12	2.996 (4)	Sb5—S15^{vii}	2.651 (2)
Pb5—S18^{iv}	2.938 (2)	Sb6—S13	2.437 (4)
Pb5—S18^{v}	2.938 (2)	Sb6^{vi}—S16	2.796 (3)
Pb6^{iv}—S17	2.926 (2)	S16—Sb6^{vii}	2.796 (3)
Pb6^{v}—S17	2.926 (2)	Sb6—S19^{iv}	2.814 (3)
Pb6—S23	2.850 (4)	Sb6—S19^{v}	2.814 (3)
Pb7—S20^{vi}	2.979 (3)	Sb7—S17	2.474 (4)
Pb7—S20^{vii}	2.979 (3)	Sb7^{vi}—S19	2.928 (2)
Pb7—S22^{i}	2.896 (3)	Sb7^{vii}—S19	2.928 (2)
Pb8—S7	2.711 (3)	Sb7—S23^{iv}	2.690 (2)
Pb8^{iv}—S9	2.930 (3)	Sb7—S23^{v}	2.690 (2)
Pb8^{v}—S9	2.930 (3)	Sb8^{iv}—S9	2.930 (3)
Pb8—S14^{vi}	2.822 (3)	Sb8^{v}—S9	2.930 (3)
Pb8—S14^{vii}	2.822 (3)	Sb8—S14^{vi}	2.822 (3)
Pb9—S12^{vi}	2.692 (3)	Sb8—S14^{vii}	2.822 (3)
Pb9—S12^{vii}	2.692 (3)	Sb8—S7	2.711 (3)
Pb9—S14	2.543 (3)	Sb9—S12^{vi}	2.692 (3)
Sb1—S4^{vi}	2.653 (3)	Sb9—S12^{vii}	2.692 (3)
Sb1—S4^{vii}	2.653 (3)	Sb9—S14	2.543 (3)
Sb1—S5^{viii}	2.428 (3)	Sb10^{ix}—S21	2.637 (3)
Sb2—S2	2.459 (4)	Sb10^{x}—S21	2.637 (3)
Sb2—S3^{iii}	2.818 (3)	Sb10^{ix}—S22	2.505 (3)
Sb2—S3^{ii}	2.818 (3)	Sb10^{x}—S22	2.505 (3)
Sb2—S6^{vi}	2.845 (3)	Sb10—S20	2.445 (3)
Sb2—S6^{vii}	2.845 (3)	Sb10^{xi}—S20	2.445 (3)
Sb3^{vi}—S1	2.802 (3)	Sb10—Sb10^{xi}	0.6919 (15)
Sb3^{vii}—S1	2.802 (3)		
S4^{vi}—Sb1—S4^{vii}	101.46 (11)	S9^{vi}—Pb8—S14^{vi}	86.57 (9)

S4^{vi}—Sb1—S5^{viii}	91.01 (9)	S9^{vi}—Pb8—S14^{vii}	163.93 (7)
S4^{vii}—Sb1—S4^{vi}	101.46 (11)	S9^{vii}—Pb8—S9^{vi}	89.03 (8)
S4^{vii}—Sb1—S5^{viii}	91.01 (9)	S9^{vii}—Pb8—S14^{vi}	163.93 (7)
S2—Sb2—S3^{iii}	88.47 (11)	S9^{vii}—Pb8—S14^{vii}	86.57 (9)
S2—Sb2—S3^{ii}	88.47 (11)	S14^{vi}—Pb8—S14^{vii}	93.43 (9)
S2—Sb2—S6^{vi}	90.53 (11)	S14^{vii}—Pb8—S14^{vi}	93.43 (9)
S2—Sb2—S6^{vii}	90.53 (11)	S7—Sb8—S9^{vi}	81.18 (8)
S3^{iii}—Sb2—S3^{ii}	93.58 (8)	S7—Sb8—S9^{vii}	81.18 (8)
S3^{iii}—Sb2—S6^{vi}	86.98 (8)	S7—Sb8—S14^{vi}	82.88 (8)
S3^{iii}—Sb2—S6^{vii}	178.84 (11)	S7—Sb8—S14^{vii}	82.88 (8)
S3^{ii}—Sb2—S3^{iii}	93.58 (8)	S9^{vi}—Sb8—S9^{vii}	89.03 (8)
S3^{ii}—Sb2—S6^{vi}	178.84 (11)	S9^{vi}—Sb8—S14^{vi}	86.57 (9)
S3^{ii}—Sb2—S6^{vii}	86.98 (8)	S9^{vi}—Sb8—S14^{vii}	163.93 (7)
S6^{vi}—Sb2—S6^{vii}	92.44 (8)	S9^{vii}—Sb8—S9^{vi}	89.03 (8)
S6^{vii}—Sb2—S6^{vi}	92.44 (8)	S9^{vii}—Sb8—S14^{vi}	163.93 (7)
Sb3^{vi}—S1—Sb3^{vii}	94.31 (14)	S9^{vii}—Sb8—S14^{vii}	86.57 (9)
Sb3^{vii}—S1—Sb3^{vi}	94.31 (14)	S14^{vi}—Sb8—S14^{vii}	93.43 (9)
Sb2—S2—Pb1^{iii}	103.96 (8)	S14^{vii}—Sb8—S14^{vi}	93.43 (9)
Sb2—S2—Pb1^{ii}	103.96 (8)	Sb9—S14—Pb8^{iv}	105.60 (8)
Pb1^{iii}—S2—Pb1^{ii}	87.49 (9)	Sb9—S14—Pb8^{v}	105.60 (8)
Pb1^{ii}—S2—Pb1^{iii}	87.49 (9)	Sb9—S14—Sb8^{iv}	105.60 (8)
S2^{iii}—Pb1—S2^{ii}	87.49 (7)	Sb9—S14—Sb8^{v}	105.60 (8)
S2^{ii}—Pb1—S2^{iii}	87.49 (7)	Pb9—S14—Pb8^{iv}	105.60 (8)
Sb2^{iii}—S3—Sb2^{ii}	93.58 (11)	Pb9—S14—Pb8^{v}	105.60 (8)
Sb2^{iii}—S3—Sb4^{vi}	89.578 (19)	Pb9—S14—Sb8^{iv}	105.60 (8)
Sb2^{iii}—S3—Sb4^{vii}	175.88 (10)	Pb9—S14—Sb8^{v}	105.60 (8)
Sb2^{ii}—S3—Sb2^{iii}	93.58 (11)	Pb8^{iv}—S14—Pb8^{v}	93.43 (13)
Sb2^{ii}—S3—Sb4^{vi}	175.88 (10)	Pb8^{iv}—S14—Sb8^{v}	93.43 (13)
Sb2^{ii}—S3—Sb4^{vii}	89.578 (19)	Pb8^{v}—S14—Pb8^{iv}	93.43 (13)
Sb4^{vi}—S3—Sb4^{vii}	87.15 (9)	Pb8^{v}—S14—Sb8^{iv}	93.43 (13)
Sb4^{vii}—S3—Sb4^{vi}	87.15 (9)	Sb8^{iv}—S14—Sb8^{v}	93.43 (12)
Sb1^{iv}—S4—Sb1^{v}	101.46 (15)	Sb8^{v}—S14—Sb8^{iv}	93.43 (12)
Sb1^{v}—S4—Sb1^{iv}	101.46 (15)	Sb5^{iv}—S15—Sb5^{v}	101.58 (13)
S1^{iv}—Sb3—S1^{v}	94.31 (10)	Sb5^{v}—S15—Sb5^{iv}	101.58 (13)
S1^{iv}—Sb3—S7^{iv}	86.64 (9)	Sb6^{vi}—S16—Sb6^{vii}	94.55 (12)
S1^{iv}—Sb3—S7^{v}	176.19 (8)	Sb6^{vi}—S16—Hg1	96.08 (8)
S1^{iv}—Sb3—S9	88.55 (9)	Sb6^{vii}—S16—Sb6^{vi}	94.55 (12)
S1^{v}—Sb3—S1^{iv}	94.31 (10)	Sb6^{vii}—S16—Hg1	96.08 (8)
S1^{v}—Sb3—S7^{iv}	176.19 (8)	Pb6^{iv}—S17—Pb6^{v}	89.19 (9)
S1^{v}—Sb3—S7^{v}	86.64 (9)	Pb6^{iv}—S17—Sb7	96.85 (7)
S1^{v}—Sb3—S9	88.55 (9)	Pb6^{v}—S17—Pb6^{iv}	89.19 (9)

S7^{iv}—Sb3—S7^{v}	92.18 (10)	Pb6^{v}—S17—Sb7	96.85 (7)
S7^{iv}—Sb3—S9	87.78 (9)	S12—Pb5—S18^{iv}	77.34 (8)
S7^{v}—Sb3—S7^{iv}	92.18 (10)	S12—Pb5—S18^{v}	77.34 (8)
S7^{v}—Sb3—S9	87.78 (9)	S18^{iv}—Pb5—S18^{v}	88.73 (6)
Sb1^{viii}—S5—Pb3^{iv}	104.96 (10)	S18^{v}—Pb5—S18^{iv}	88.73 (6)
Sb1^{viii}—S5—Pb3^{v}	104.96 (10)	S13—Sb6—S16^{iv}	93.02 (10)
Pb3^{iv}—S5—Pb3^{v}	88.64 (11)	S13—Sb6—S16^{v}	93.02 (10)
Pb3^{v}—S5—Pb3^{iv}	88.64 (11)	S13—Sb6—S19^{iv}	91.10 (10)
Sb2^{iv}—S6—Sb2^{v}	92.44 (11)	S13—Sb6—S19^{v}	91.10 (10)
Sb2^{iv}—S6—Pb2	92.10 (13)	S16^{iv}—Sb6—S16^{v}	94.55 (9)
Sb2^{v}—S6—Sb2^{iv}	92.44 (11)	S16^{iv}—Sb6—S19^{iv}	85.69 (8)
Sb2^{v}—S6—Pb2	92.10 (13)	S16^{iv}—Sb6—S19^{v}	175.85 (12)
Sb3^{vi}—S7—Sb3^{vii}	92.18 (13)	S16^{v}—Sb6—S16^{iv}	94.55 (9)
Sb3^{vi}—S7—Pb8	93.36 (9)	S16^{v}—Sb6—S19^{iv}	175.85 (12)
Sb3^{vi}—S7—Sb8	93.36 (9)	S16^{v}—Sb6—S19^{v}	85.69 (8)
Sb3^{vii}—S7—Sb3^{vi}	92.18 (13)	S19^{iv}—Sb6—S19^{v}	93.76 (8)
Sb3^{vii}—S7—Pb8	93.36 (9)	S19^{v}—Sb6—S19^{iv}	93.76 (8)
Sb3^{vii}—S7—Sb8	93.36 (9)	S17^{vi}—Pb6—S17^{vii}	89.19 (7)
S3^{iv}—Sb4—S3^{v}	87.15 (7)	S17^{vi}—Pb6—S23	78.66 (8)
S3^{iv}—Sb4—S8	90.69 (10)	S17^{vii}—Pb6—S17^{vi}	89.19 (7)
S3^{iv}—Sb4—S10^{iv}	85.35 (7)	S17^{vii}—Pb6—S23	78.66 (8)
S3^{iv}—Sb4—S10^{v}	172.09 (10)	S20^{vi}—Pb7—S20^{vii}	87.20 (7)
S3^{v}—Sb4—S3^{iv}	87.15 (7)	S20^{vi}—Pb7—S22^{i}	77.37 (8)
S3^{v}—Sb4—S8	90.69 (10)	S20^{vii}—Pb7—S20^{vi}	87.20 (7)
S3^{v}—Sb4—S10^{iv}	172.09 (10)	S20^{vii}—Pb7—S22^{i}	77.37 (8)
S3^{v}—Sb4—S10^{v}	85.35 (7)	Pb5^{vi}—S18—Pb5^{vii}	88.73 (8)
S8—Sb4—S10^{iv}	91.87 (11)	Pb5^{vi}—S18—Hg2	103.89 (11)
S8—Sb4—S10^{v}	91.87 (11)	Pb5^{vii}—S18—Pb5^{vi}	88.73 (8)
S10^{iv}—Sb4—S10^{v}	102.04 (8)	Pb5^{vii}—S18—Hg2	103.89 (11)
S10^{v}—Sb4—S10^{iv}	102.04 (8)	Sb6^{vi}—S19—Sb6^{vii}	93.76 (11)
S6—Pb2—S11^{iv}	82.66 (9)	Sb6^{vi}—S19—Sb7^{vi}	87.97 (2)
S6—Pb2—S11^{v}	82.66 (9)	Sb6^{vi}—S19—Sb7^{vii}	171.40 (16)
S11^{iv}—Pb2—S11^{v}	89.38 (7)	Sb6^{vii}—S19—Sb6^{vi}	93.76 (11)
S11^{v}—Pb2—S11^{iv}	89.38 (7)	Sb6^{vii}—S19—Sb7^{vi}	171.40 (16)
S5^{vi}—Pb3—S5^{vii}	88.64 (8)	Sb6^{vii}—S19—Sb7^{vii}	87.97 (2)
S5^{vii}—Pb3—S5^{vi}	88.64 (8)	Sb7^{vi}—S19—Sb7^{vii}	89.11 (9)
S12^{vi}—Sb9—S12^{vii}	99.48 (10)	Sb7^{vii}—S19—Sb7^{vi}	89.11 (9)
S12^{vi}—Sb9—S14	86.98 (9)	Pb7^{iv}—S20—Pb7^{v}	87.20 (9)
S12^{vii}—Sb9—S12^{vi}	99.48 (10)	Pb7^{iv}—S20—Sb10	104.34 (13)
S12^{vii}—Sb9—S14	86.98 (9)	Pb7^{iv}—S20—Sb10^{xi}	93.01 (10)
S12^{vi}—Pb9—S12^{vii}	99.48 (10)	Pb7^{v}—S20—Pb7^{iv}	87.20 (9)

S12^{vi}—Pb9—S14	86.98 (8)	Pb7^{v}—S20—Sb10	93.01 (10)
S12^{vii}—Pb9—S12^{vi}	99.48 (10)	Pb7^{v}—S20—Sb10^{xi}	104.34 (13)
S12^{vii}—Pb9—S14	86.98 (8)	Sb10—S20—Sb10^{xi}	16.27 (4)
Sb3—S9—Pb8^{iv}	97.66 (9)	Sb10^{xi}—S20—Sb10	16.27 (4)
Sb3—S9—Pb8^{v}	97.66 (9)	S16—Hg1—S21^{i}	174.07 (12)
Sb3—S9—Sb8^{iv}	97.66 (9)	S17—Sb7—S19^{iv}	85.25 (10)
Sb3—S9—Sb8^{v}	97.66 (9)	S17—Sb7—S19^{v}	85.25 (10)
Pb8^{iv}—S9—Pb8^{v}	89.03 (11)	S17—Sb7—S23^{iv}	90.20 (10)
Pb8^{iv}—S9—Sb8^{v}	89.03 (11)	S17—Sb7—S23^{v}	90.20 (10)
Pb8^{v}—S9—Pb8^{iv}	89.03 (11)	S19^{iv}—Sb7—S19^{v}	89.11 (7)
Pb8^{v}—S9—Sb8^{iv}	89.03 (11)	S19^{iv}—Sb7—S23^{iv}	85.50 (7)
Sb8^{iv}—S9—Sb8^{v}	89.03 (11)	S19^{iv}—Sb7—S23^{v}	173.21 (8)
Sb8^{v}—S9—Sb8^{iv}	89.03 (11)	S19^{v}—Sb7—S19^{iv}	89.11 (7)
Sb4^{vi}—S10—Sb4^{vii}	102.04 (10)	S19^{v}—Sb7—S23^{iv}	173.21 (8)
Sb4^{vii}—S10—Sb4^{vi}	102.04 (10)	S19^{v}—Sb7—S23^{v}	85.50 (7)
S11—Sb5—S15^{vi}	91.67 (12)	S23^{iv}—Sb7—S23^{v}	99.57 (8)
S11—Sb5—S15^{vii}	91.67 (12)	S23^{v}—Sb7—S23^{iv}	99.57 (8)
S15^{vi}—Sb5—S15^{vii}	101.58 (9)	Hg1^{i}—S21—Sb10^{ix}	103.66 (8)
S15^{vii}—Sb5—S15^{vi}	101.58 (9)	Hg1^{i}—S21—Sb10^{x}	103.66 (8)
Pb2^{vi}—S11—Pb2^{vii}	89.38 (9)	Sb10^{ix}—S21—Sb10^{x}	80.75 (11)
Pb2^{vi}—S11—Sb5	99.90 (7)	Sb10^{x}—S21—Sb10^{ix}	80.75 (11)
Pb2^{vii}—S11—Pb2^{vi}	89.38 (9)	Pb7^{i}—S22—Sb10^{ix}	93.75 (11)
Pb2^{vii}—S11—Sb5	99.90 (7)	Pb7^{i}—S22—Sb10^{x}	93.75 (11)
Sb9^{iv}—S12—Sb9^{v}	99.48 (14)	Sb10^{ix}—S22—Sb10^{x}	85.99 (12)
Sb9^{iv}—S12—Pb9^{v}	99.48 (14)	Sb10^{x}—S22—Sb10^{ix}	85.99 (12)
Sb9^{iv}—S12—Pb5	130.26 (7)	Pb6—S23—Sb7^{vi}	93.92 (8)
Sb9^{v}—S12—Sb9^{iv}	99.48 (14)	Pb6—S23—Sb7^{vii}	93.92 (8)
Sb9^{v}—S12—Pb9^{iv}	99.48 (14)	Sb7^{vi}—S23—Sb7^{vii}	99.57 (11)
Sb9^{v}—S12—Pb5	130.26 (7)	Sb7^{vii}—S23—Sb7^{vi}	99.57 (11)
Pb9^{iv}—S12—Pb9^{v}	99.48 (14)	S18—Hg2—S18^{i}	180
Pb9^{iv}—S12—Pb5	130.26 (7)	S18^{i}—Hg2—S18	180
Pb9^{v}—S12—Pb9^{iv}	99.48 (14)	S20—Sb10—S21^{ix}	89.78 (10)
Pb9^{v}—S12—Pb5	130.26 (7)	S20—Sb10—S22^{ix}	95.80 (10)
S7—Pb8—S9^{vi}	81.18 (8)	S20—Sb10—Sb10^{xi}	81.87 (13)
S7—Pb8—S9^{vii}	81.18 (8)	S21^{ix}—Sb10—S22^{ix}	96.39 (8)
S7—Pb8—S14^{vi}	82.88 (8)	S21^{ix}—Sb10—Sb10^{xi}	130.38 (18)
S7—Pb8—S14^{vii}	82.88 (8)	S22^{ix}—Sb10—Sb10^{xi}	133.00 (17)
S9^{vi}—Pb8—S9^{vii}	89.03 (8)		

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

Fig. 1