The channel structure of trithallium pentaantimonate(V), \( Tl_3Sb_5O_{14} \)

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Single crystals of \( Tl_3Sb_5O_{14} \) were grown by solid-state reaction in a corundum crucible under air (1273 K, 12 h). The structure was determined by single-crystal X-ray diffraction. It is isotypic to the \( K_3Sb_5O_{14} \), \( Rb_3Sb_5O_{14} \) and \( Cs_3Sb_5O_{14} \) analogues with orthorhombic \( Pbam \) symmetry and cell parameters \( a = 24.2899 (9) \) Å, \( b = 7.1931 (3) \) Å, \( c = 7.4182 (3) \) Å. The Sb atoms form irregular \([SbO_6]\) octahedra, which are linked via edges and corners into a triperiodic network. The \( Tl^+ \) ions are located in distinct channels of the network extending along [010] and [001].

1. Chemical context

During an extensive study of \( M[SbF_6] \) compounds (\( M = Li, Na, Tl \)), precursors in the form of \( MSbO_3 \) were synthesized. Whereas the chosen conditions (1273 K, 12 h) yielded the expected product for \( LiSbO_3 \) and \( NaSbO_3 \), the \( Tl \)-poor title compound \( Tl_3Sb_5O_{14} \) was inadvertently obtained in the case of \( Tl \). \( TlSbO_3 \) was later successfully synthesized at 1073 K. In fact, prior syntheses of \( TlSbO_3 \) were performed at even lower temperatures (Bouchama & Tournoux, 1975).

The analogues \( K_3Sb_5O_{14} \) (Hong, 1974), \( Rb_3Sb_5O_{14} \) and \( Cs_3Sb_5O_{14} \) (Hirschle et al., 2001) have been synthesized at 1373 K using more involved routes. The first structural characterization of \( K_3Sb_5O_{14} \) was published by Aurivillius (1966). However, the author gives an incorrect Schoenbeck space-group symmetry of type \( Pba2_1 \), which was later corrected to \( Pbam \) by (Hong, 1974).

Hong (1974) noted unusual enlargement of the atomic displacement parameters (ADP) of \( K \) in \( K_3Sb_5O_{14} \), which are located in distinct channels, suggesting ion conductivity. In fact, the author could partially substitute \( K \) for \( Rb, Ag \) and \( Tl \) in the respective nitrate salt melts. Accordingly, it is expected that the hitherto structurally uncharacterized \( Ag_3Sb_5O_{14} \) likewise exists. In contrast, substitution with the smaller Na’ ion in an \( NaNO_3 \) melt led to a collapse of the structure and formation of the \( Na \)-poor \( Na_2Sb_4O_{11} \). The instability of \( M_3Sb_5O_{14} \) with small ions might explain the successful syntheses of \( MSbO_3 \) (\( M = Li, Na \)) at 1273 K.

2. Structural commentary

\( Tl_3Sb_5O_{14} \) crystallizes in the space group \( Pbam \) and is isotypic to \( M_3Sb_5O_{14} \) (\( M = K, Rb, Cs \)). Two different settings of the \( Pbam \) space group were used to describe the structures: \( a > b \) by Hong (1974) and \( a < b \) by Hirschle et al. (2001). These are equivalent descriptions, because the \((a', b', c') = (b, -a, c)\) operation is an element of the affine normalizer of the \( Pbam \)
space group. Herein we use the original setting and atom labeling of Hong (1974).

In structures of the $M_3Sb_5O_{14}$ type, the monovalent metal atoms $M$ are located in channels of a triperiodic network formed by $[SbO_6]$ octahedra. There are two distinct channels parallel to $[010]$, both with $\overline{p}2_1b2m$ symmetry (Fig. 1). In the second channel, the $M_2$ atoms are likewise arranged in the form of zigzag lines (Fig. 2). All of the $M$ atoms are located on or very close to the reflection plane of the channels. Additionally, channels with a smaller diameter extend in the $[001]$ direction (Fig. 3). For $K_3Sb_5O_{14}$, Hong (1974) reports excessive enlargement of the ADPs of the $K_1$ and $K_2$ atoms in the $[010]$ and $[001]$ directions of the channels, with the ‘thermal motions’ in these directions being ‘eight times bigger’ than in the $[100]$ direction. The $Tl_1$ and $Tl_2$ atoms in the title compound show a much milder enlargement of the ADPs. The ratio of the mean-square displacement of the longest and shortest principal axes of the ADP tensor is 3.2 for $Tl_1$ and 2.9 for $Tl_2$. Note that the value for $Tl_2$ is not directly comparable, since it was refined as disordered about the reflection plane. However, even when placing the atom on the reflection plane, the ratio increases to only 3.2. From these values, it appears that $Tl_3Sb_5O_{14}$ is not a prime candidate for ion conductivity, at least at the measurement temperature of 100 K. For $Rb_3Sb_5O_{14}$ and $Cs_3Sb_5O_{14}$, similarly mild enlargement of the ADPs has been reported (Hirschle et al., 2001). In contrast to the $Tl_3Sb_5O_{14}$ title compound, these were derived from data collected at room temperature.

All $Sb$ atoms are coordinated by six $O$ atoms forming highly irregular $[SbO_6]$ octahedra (Table 1) with $O$—$Sb$—$O$ cis angles ranging from 73.37 (17) to 103.83 (13)° and trans angles up to 150.66 (16)°. As noted by Hirschle et al. (2001), the framework can be described as being composed of four distinct parts: two infinite octahedra chains and two edge-connected pairs of octahedra. In general, these elements are connected via corners but there is an additional connection between a pair and a chain via an edge.

A quantitative comparison of $Tl_3Sb_5O_{14}$ and the alkali-metal analogues $M_3Sb_5O_{14}$ ($M = K$, $Rb$, $Cs$) was performed using the COMPSTRU (de la Flor et al., 2016) module of the Bilbao Crystallographic Server (Aroyo et al., 2006). The $Tl_2$ atom was moved onto the reflection plane to make the sets of Wyckoff positions compatible. The degree of lattice distortion with respect to the $Tl$ compound is $S = 0.0042$ ($M = K$), $S = 0.0048$ ($M = Rb$) and $S = 0.0262$ ($M = Cs$). This shows that the $K$, $Rb$ and $Tl$ compounds feature very similar cell parameters, with the volume increasing slightly according to $K > Rb > Tl$ (Table 2). In contrast, the lattice of $Cs_3Sb_5O_{14}$ features a pronounced distortion with a ca 11% larger unit-cell volume. The enlargement affects foremost the $a$ and $b$ lattice parameters, whereas $c$ is smaller than for the $Tl$ compound. We
| Table 1 | Selected geometric parameters (Å, °). |
|---------|-------------------------------------|
| TII—TII | 3.3972 (4) Sb2—O10 1.919 (3)        |
| TII—TII' | 3.4507 (7) Sb2—O2v 1.983 (4)        |
| TII—TII'' | 3.6130 (4) Sb2—O4v 2.140 (4)        |
| TII—TII" | 3.3972 (4) Sb2—O4u 2.215 (4)        |
| TII—TII' | 3.6129 (4) Sb3—O5vii 1.952 (4)      |
| TII—O3 | 2.565 (4) Sb3—O5 1.979 (4)          |
| TII—O6 | 2.775 (4) Sb3—O9v 1.998 (3)         |
| TII—O5 | 2.495 (4) Sb3—O9 1.998 (3)          |
| Sh1—O6 | 1.925 (3) Sb3—O7vii 2.002 (3)       |
| Sh1—O8 | 1.925 (3) Sb3—O7 2.002 (3)          |
| Sh1—O6'vii | 1.971 (4) Sb4—O3 1.9233 (15)       |
| Sh1—O1'vii | 1.996 (2) Sb4—O7vii 1.936 (3)      |
| Sh1—O1 | 1.996 (2) Sb4—O9 1.954 (3)          |
| Sh1—O2 | 2.081 (4) Sb4—O8 1.975 (3)          |
| Sh1—O2' | 1.911 (4) Sb4—O4 2.0284 (11)       |
| Sh2—O10vii | 1.919 (3) Sb4—O10vii 2.041 (3)     |

| Table 2 | Comparison of unit-cell parameters (Å, Å³) of the M3Sb5O14 structures. |
|---------|-----------------------------------------------------------------------|
| Compound | K₃Sb₅O₁₄ | Rb₃Sb₅O₁₄ | Cs₃Sb₅O₁₄ | Tl₃Sb₅O₁₄ |
| a       | 24.247 (4) | 24.478 (2) | 26.251 (5) | 24.2899 (9) |
| b       | 7.157 (2)  | 7.1881 (9) | 7.4337 (13) | 7.1931 (3)  |
| c       | 7.334 (2)  | 7.331 (2)  | 7.396 (3)  | 7.4182 (3)  |
| V       | 1272.7 (3) | 1289.8 (4) | 1443.3 (7) | 1296.11 (9) |

3. Synthesis and crystallization

A mixture of 0.682 g TiNO₃ and 0.373 g Sb₂O₃ (which makes for an approximate molar ratio of 1:1 for Ti:Sb) was heated in a corundum crucible at 1273 K for 12 h in air. From the reaction, a dark-orange powder was obtained. The single crystals formed as rectangular-prismatic plates. Crystals were isolated under a polarizing microscope and cut to an appropriate size for single crystal diffraction of a highly absorbing crystal.

4. Refinement

Crystal data, collection and structure refinement are summarized in Table 3. A starting model was generated using the coordinates of K₃Sb₅O₁₄ (Hong, 1974). Owing to distinct peaks in the difference-Fourier map, the Ti2 atom was removed from the reflection plane and refined as disordered. Even though the refined distance of the atom from the reflection plane is minute, the residuals improved significantly

| Table 3 | Experimental details. |
|---------|-----------------------|
| Crystal data | Tl₃Sb₅O₁₄ |
| Chemical formula | Ti₃Sb₅O₁₄ |
| M_r | 1445.86 |
| Crystal system, space group | Orthorhombic, Pham |
| Temperature (K) | 293 (274, 296, 300, 308, 320) |
| V (Å³) | 1272.7 (3) |
| Z | 4 |
| Radiation type | Mo Kα |
| µ (mm⁻¹) | 47.48 |
| Crystal size (mm) | 0.11 × 0.06 × 0.02 |

Data collection

Diffractometer | Bruker Kappa APEX2 CCD |
Absorption correction | Multi-scan (SADABS; Bruker, 2007) |
T_max, T_min | 0.7100, 0.058 |
No. of reflections | 27499, 3084, 2850 |
R(int) | 0.023, 0.055, 1.07 |
S | 2086.6 |
No. of parameters | 121 |
\( \Delta \rho_{\text{max}}, \Delta \rho_{\text{min}} \) (e Å⁻³) | 2.55, -1.52 |

Computer programs: APEX2 and SAINT-Plus (Bruker, 2021), SHELXL2014/7 (Sheldrick, 2015), DIAMOND (Putz & Brandenburg, 2021) and pubCIF (Westrip, 2010).
[\( R[I > 2\sigma(I)] \) from 0.028 to 0.023], which might be in part due to the increased number of anisotropic displacement parameters.

Funding information

The authors acknowledge TU Wien Bibliothek for financial support through its Open Access Funding Programme.

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The channel structure of trithallium pentaantimonate(V), $\text{Tl}_3\text{Sb}_5\text{O}_{14}$

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Computing details

Data collection: *APEX3* (Bruker, 2021); cell refinement: *APEX3* (Bruker, 2021); data reduction: *SAINT-Plus* (Bruker, 2021); program(s) used to solve structure: undef; program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Putz & Brandenburg, 2021); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Trithallium pentaantimonate(V)

Crystal data

$\text{Tl}_3\text{Sb}_5\text{O}_{14}$  
$M_r = 1445.86$  
Orthorhombic, $Pbam$  
$a = 24.2899$ (9) Å  
b = 7.1931 (3) Å  
c = 7.4182 (3) Å  
$V = 1296.11$ (9) Å$^3$  
$Z = 4$  
$F(000) = 2440$  

$D_x = 7.410$ Mg m$^{-3}$  
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å  
Cell parameters from 9928 reflections  
$\theta = 2.8–35.3^\circ$  
$\mu = 47.48$ mm$^{-1}$  
$T = 250$ K  
Plate, colourless  
$0.11 \times 0.06 \times 0.02$ mm

Data collection

Bruker Kappa APEXII CCD  
diffractometer  
Graphite monochromator  
$\omega$- and $\varphi$-scans  
Absorption correction: multi-scan  
(SADABS; Bruker, 2021)  
$T_{\text{min}} = 0.010$, $T_{\text{max}} = 0.058$  
27499 measured reflections  

3084 independent reflections  
2850 reflections with $I > 2\sigma(I)$  
$R_{\text{int}} = 0.051$  
$\theta_{\text{max}} = 35.3^\circ$, $\theta_{\text{min}} = 3.0^\circ$  
h = $-39\rightarrow39$  
k = $-11\rightarrow11$  
l = $-12\rightarrow12$

Refinement

Refinement on $F^2$  
Least-squares matrix: full  
$R[F^2 > 2\sigma(F^2)] = 0.023$  
$wR(F^2) = 0.055$  
$S = 1.07$  
3084 reflections  
121 parameters  
0 restraints

Primary atom site location: isomorphous  
structure methods

$w = 1/[\sigma^2(F_c^2) + (0.0199P)^2 + 6.584P]$  
where $P = (F_c^2 + 2F_e^2)/3$  
$(\Delta/\sigma)_{\text{max}} = 0.001$  
$\Delta\rho_{\text{max}} = 2.55$ e Å$^{-3}$  
$\Delta\rho_{\text{min}} = -1.52$ e Å$^{-3}$  
Extinction correction: *SHELXL-2014/7* (Sheldrick 2015),  
$Fc^c = kFc[1+0.001xFc^2]/\sin(2\theta)]^{1/4}$  
Extinction coefficient: 0.00075 (4)
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)

| Atom | x     | y     | z     | Uiso*/Ueq | Occ. (<1) |
|------|-------|-------|-------|-----------|-----------|
| Tl1  | 0.01569 (2) | 0.23393 (5) | 0.5000 | 0.03732 (8) | 0.5       |
| Tl2  | 0.29264 (2) | 0.12150 (6) | −0.0170 (5) | 0.0353 (4) | 0.5       |
| Tl3  | 0.38418 (2) | 0.10536 (4) | 0.5000 | 0.03177 (7) | 0.5       |
| Sb1  | 0.05715 (2) | 0.41738 (4) | 0.0000 | 0.00993 (6) | 0.5       |
| Sb2  | 0.43805 (2) | 0.40456 (4) | 0.0000 | 0.01042 (6) | 0.5       |
| Sb3  | 0.25558 (2) | 0.32863 (4) | 0.5000 | 0.00998 (6) | 0.5       |
| Sb4  | 0.14535 (2) | 0.11009 (3) | 0.26233 (3) | 0.01011 (5) | 0.5       |
| O1   | 0.0000 | 0.5000 | 0.1759 (5) | 0.0131 (6) | 0.5       |
| O2   | 0.01735 (15) | 0.1611 (5) | 0.0000 | 0.0130 (6) | 0.5       |
| O3   | 0.11974 (15) | 0.1728 (6) | 0.5000 | 0.0139 (6) | 0.5       |
| O4   | 0.14514 (15) | 0.0305 (5) | 0.0000 | 0.0124 (6) | 0.5       |
| O5   | 0.28203 (16) | 0.0685 (5) | 0.5000 | 0.0146 (6) | 0.5       |
| O6   | 0.40613 (16) | 0.1618 (5) | 0.0000 | 0.0146 (6) | 0.5       |
| O7   | 0.21049 (11) | 0.2637 (4) | 0.2830 (4) | 0.0144 (5) | 0.5       |
| O8   | 0.10390 (11) | 0.3355 (4) | 0.1939 (4) | 0.0138 (4) | 0.5       |
| O9   | 0.31369 (11) | 0.3832 (4) | 0.3169 (4) | 0.0136 (4) | 0.5       |
| O10  | 0.42520 (10) | 0.4563 (4) | 0.2502 (3) | 0.0132 (4) | 0.5       |

Atomic displacement parameters (Å²)

|       | U11   | U22   | U33   | U12   | U13   | U23   |
|-------|-------|-------|-------|-------|-------|-------|
| Tl1   | 0.01779 (11) | 0.04162 (17) | 0.05255 (18) | 0.00612 (10) | 0.0000 | 0.0000 |
| Tl2   | 0.01922 (12) | 0.04252 (19) | 0.0442 (12) | −0.00204 (11) | −0.0027 (2) | 0.0106 (5) |
| Tl3   | 0.01232 (10) | 0.02797 (13) | 0.05504 (18) | 0.00031 (8) | 0.0000 | 0.0000 |
| Sb1   | 0.00754 (12) | 0.01037 (13) | 0.01187 (12) | 0.00045 (9) | 0.0000 | 0.0000 |
| Sb2   | 0.00826 (12) | 0.01106 (13) | 0.01193 (12) | 0.00000 (9) | 0.0000 | 0.0000 |
| Sb3   | 0.00756 (11) | 0.01032 (13) | 0.01205 (12) | −0.00028 (9) | 0.0000 | 0.0000 |
| Sb4   | 0.00803 (9) | 0.01136 (10) | 0.01094 (9) | 0.00039 (6) | −0.00072 (6) | −0.00010 (7) |
| O1    | 0.0106 (14) | 0.0163 (16) | 0.0123 (13) | 0.0028 (12) | 0.0000 | 0.0000 |
| O2    | 0.0080 (14) | 0.0098 (15) | 0.0214 (16) | −0.0005 (11) | 0.0000 | 0.0000 |
| O3    | 0.0107 (15) | 0.0223 (18) | 0.0086 (13) | 0.0045 (13) | 0.0000 | 0.0000 |
| O4    | 0.0119 (14) | 0.0150 (16) | 0.0101 (13) | −0.0016 (12) | 0.0000 | 0.0000 |
| O5    | 0.0114 (15) | 0.0106 (15) | 0.0217 (16) | −0.0008 (12) | 0.0000 | 0.0000 |
| O6    | 0.0113 (15) | 0.0087 (15) | 0.0238 (17) | −0.0011 (12) | 0.0000 | 0.0000 |
| O7    | 0.0123 (11) | 0.0159 (12) | 0.0150 (10) | −0.0035 (9) | −0.0031 (9) | 0.0011 (9) |
| O8    | 0.0142 (11) | 0.0128 (11) | 0.0144 (10) | 0.0034 (9) | −0.0038 (9) | −0.0013 (9) |
| O9    | 0.0119 (10) | 0.0125 (11) | 0.0165 (10) | −0.0029 (8) | 0.0023 (9) | −0.0003 (9) |
| O10   | 0.0093 (10) | 0.0161 (11) | 0.0141 (10) | 0.0003 (8) | 0.0005 (8) | 0.0001 (9) |
**Geometric parameters (Å, °)**

| Bond                  | Distance (Å) | Bond                  | Distance (Å) |
|-----------------------|--------------|-----------------------|--------------|
| Tl1—Tl3i              | 3.3972 (4)   | Sb2—Sb2x              | 3.079 (6)    |
| Tl1—Tl1ii             | 3.4507 (7)   | Sb3—O5iii             | 1.952 (4)    |
| Tl1—Tl3iii            | 3.6130 (4)   | Sb3—O5                | 1.979 (4)    |
| Tl2—Tl2iv             | 0.252 (7)    | Sb3—O9ii              | 1.998 (3)    |
| Tl3—Tl1v              | 3.3972 (4)   | Sb3—O9                | 1.998 (3)    |
| Tl3—Tl2vi             | 3.6129 (4)   | Sb3—O7vii             | 2.002 (3)    |
| Tl1—O3                | 2.565 (4)    | Sb3—O7                | 2.002 (3)    |
| Tl2—O6                | 2.775 (4)    | Sb4—O3                | 1.9233 (15)  |
| Tl3—O5                | 2.495 (4)    | Sb4—O7                | 1.936 (3)    |
| Tl3—Sb3               | 3.5123 (4)   | Sb4—O9vii             | 1.954 (3)    |
| Sb1—O8iv              | 1.925 (3)    | Sb4—O8                | 1.975 (3)    |
| Sb1—O8                | 1.925 (3)    | Sb4—O4                | 2.0284 (11)  |
| Sb1—O6vii             | 1.971 (4)    | Sb4—O10vii            | 2.041 (3)    |
| Sb1—O1viii            | 1.996 (2)    | Sb4—Sb2xiii           | 3.1743 (3)   |
| Sb1—O1                | 1.996 (2)    | O1—Sb1viii            | 1.996 (2)    |
| Sb1—O2                | 2.081 (4)    | O2—Sb2                | 1.983 (4)    |
| Sb1—Sb1viii           | 3.0199 (6)   | O2—Sb2vii             | 2.140 (4)    |
| Sb2—O6                | 1.911 (4)    | O3—Sb4v               | 1.9233 (15)  |
| Sb2—O10v              | 1.919 (3)    | O4—Sb4v               | 2.0283 (11)  |
| Sb2—O10               | 1.919 (3)    | O4—Sb2vii             | 2.215 (4)    |
| Sb2—O2v               | 1.983 (4)    | O5—Sb3v               | 1.952 (4)    |
| Sb2—O2vii             | 2.140 (4)    | O6—Sb1viii            | 1.971 (4)    |
| Sb2—O4vii             | 2.215 (4)    | O6—Tl2v               | 2.775 (4)    |
| Sb2—Sb4viii           | 3.1742 (3)   | O9—Sb4v               | 1.954 (3)    |
| Sb2—Sb4vii            | 3.1742 (3)   | O10—Sb4v              | 2.042 (3)    |
| O3—Tl1—Tl3i           | 169.98 (10)  | Sb4vii—Sb2—Sb2x      | 112.786 (12) |
| O3—Tl1—Tl1ii          | 92.89 (10)   | Sb4vii—Sb2—Sb2x      | 112.786 (12) |
| Tl3—Tl1—Tl1ii         | 97.130 (13)  | O5vii—Sb3—O5         | 171.04 (9)   |
| O3—Tl1—Tl1—Tl3iii     | 57.56 (10)   | O5vii—Sb3—O9vii      | 99.04 (11)   |
| Tl3—Tl1—Tl3iii        | 112.419 (11) | O5—Sb3—O9vii         | 87.50 (11)   |
| Tl1v—Tl1—Tl3iii       | 150.451 (14) | O5viii—Sb3—O9        | 99.03 (11)   |
| Tl2—O6—Tl2—O6         | 87.40 (7)    | O5—Sb3—O9            | 87.50 (11)   |
| O5—Tl3—Tl1v           | 166.21 (9)   | O9v—Sb3—O9           | 85.66 (16)   |
| O5—Tl3—Sb3            | 33.32 (9)    | O5vii—Sb3—O7vii      | 87.14 (11)   |
| Tl1v—Tl3—Sb3          | 132.96 (12)  | O5—Sb3—O7vii         | 87.54 (11)   |
| O5—Tl3—Tl1vi          | 126.21 (9)   | O9v—Sb3—O7vii        | 83.44 (11)   |
| Tl1v—Tl3—Tl1vi        | 67.581 (11)  | O9v—Sb3—O7vii        | 168.21 (11)  |
| Sb3—Tl3—Tl1v          | 159.523 (11) | O5vii—Sb3—O7         | 87.14 (11)   |
| O8v—Sb1—O8            | 96.70 (16)   | O5—Sb3—O7            | 87.54 (11)   |
| O8v—Sb1—O6vii         | 90.34 (11)   | O9vii—Sb3—O7         | 168.21 (11)  |
| O8—Sb1—O6vii          | 90.34 (11)   | O9—Sb3—O7            | 83.44 (11)   |
| O8v—Sb1—O1viii        | 90.74 (11)   | O7vii—Sb3—O7         | 107.02 (16)  |
| O8—Sb1—O1viii         | 171.91 (11)  | O5viii—Sb3—Tl3       | 145.11 (12)  |
| O6vii—Sb1—O1viii      | 92.82 (8)    | O5—Sb3—Tl3           | 43.84 (11)   |
| O8v—Sb1—O1            | 171.91 (11)  | O9vii—Sb3—Tl3        | 57.42 (8)    |
|Bond| Value 1| Value 2| Value 3|
|---|---|---|---|
|O8—Sb1—O1| 90.74 (11)| O9—Sb3—Tl3| 57.42 (8)|
|O6vii—Sb1—O1| 92.82 (8)| O7vi—Sb3—Tl3| 112.32 (8)|
|O1viii—Sb1—O1| 81.67 (15)| O3—Sb4—O7| 93.31 (15)|
|O8vii—Sb1—O2| 90.17 (11)| O3—Sb4—O9vi| 99.82 (14)|
|O6—Sb1—O2| 90.17 (11)| O7—Sb4—O9vi| 92.53 (12)|
|O1viii—Sb1—O2| 179.23 (15)| O3—Sb4—O8| 83.01 (13)|
|O1—Sb1—O2| 86.60 (8)| O7—Sb4—O8| 88.19 (12)|
|O8vii—Sb1—Sb1viii| 131.51 (8)| O9vi—Sb4—O8| 177.03 (11)|
|O8—Sb1—Sb1viii| 131.51 (8)| O3—Sb4—O4| 160.89 (15)|
|O6—Sb1—Sb1viii| 93.72 (11)| O7—Sb4—O4| 103.83 (13)|
|O1viii—Sb1—Sb1viii| 40.84 (8)| O9vi—Sb4—O4| 87.96 (13)|
|O1—Sb1—Sb1viii| 40.84 (8)| O8—Sb4—O4| 89.07 (13)|
|O2—Sb1—Sb1viii| 85.51 (10)| O3—Sb4—O10vii| 84.03 (14)|
|O6—Sb2—O10v| 96.40 (9)| O7—Sb4—O10vii| 177.09 (11)|
|O6—Sb2—O10| 96.40 (9)| O9vi—Sb4—O10vii| 89.09 (11)|
|O10v—Sb2—O10| 150.66 (16)| O8—Sb4—O10vii| 90.31 (11)|
|O6—Sb2—O2v| 100.16 (16)| O4—Sb4—O10vii| 78.63 (13)|
|O10v—Sb2—O2v| 101.78 (8)| O3—Sb4—Sb2viii| 117.70 (12)|
|O10—Sb2—O2v| 101.78 (8)| O7—Sb4—Sb2viii| 146.72 (8)|
|O6—Sb2—O2vii| 173.53 (15)| O9v—Sb4—Sb2viii| 93.61 (8)|
|O10v—Sb2—O2vii| 85.12 (9)| O8—Sb4—Sb2viii| 93.52 (8)|
|O10—Sb2—O2vii| 85.12 (9)| O4—Sb4—Sb2viii| 43.86 (10)|
|O2v—Sb2—O2vii| 73.37 (17)| O10v—Sb4—Sb2viii| 35.42 (7)|
|O6—Sb2—O4vii| 90.22 (16)| Sb1v—O1—Sb1| 98.33 (15)|
|O10v—Sb2—O4vii| 76.83 (8)| Sb2i—O2—Sb1| 131.46 (19)|
|O10—Sb2—O4vii| 76.83 (8)| Sb2i—O2—Sb2viii| 106.63 (17)|
|O2v—Sb2—O4vii| 169.63 (15)| Sb1—O2—Sb2viii| 121.92 (17)|
|O2vii—Sb2—O4vii| 96.25 (14)| Sb4—O3—Sb4v| 132.9 (2)|
|O6—Sb2—Sb4vii| 99.60 (9)| Sb4—O3—Tl1| 111.03 (11)|
|O10v—Sb2—Sb4vii| 38.07 (8)| Sb4v—O3—Tl1| 111.03 (11)|
|O10—Sb2—Sb4vii| 113.51 (8)| Sb4v—O4—Sb4| 147.2 (2)|
|O2v—Sb2—Sb4vii| 136.95 (5)| Sb4v—O4—Sb2viii| 96.76 (11)|
|O2vii—Sb2—Sb4vii| 85.49 (8)| Sb4—O4—Sb2viii| 96.76 (11)|
|O4viii—Sb2—Sb4vii| 39.39 (3)| Sb3v—O5—Sb3| 133.2 (2)|
|O6—Sb2—Sb4v| 99.60 (9)| Sb3v—O5—Tl3| 124.01 (18)|
|O10v—Sb2—Sb4v| 113.51 (8)| Sb3—O5—Tl3| 102.84 (16)|
|O10—Sb2—Sb4v| 38.07 (8)| Sb2—O6—Sb1v| 129.2 (2)|
|O2v—Sb2—Sb4v| 136.95 (5)| Sb2—O6—Tl2| 119.90 (17)|
|O2v—Sb2—Sb4v| 85.49 (8)| Sb1v—O6—Tl2| 110.88 (16)|
|O4viii—Sb2—Sb4v| 39.39 (3)| Sb2—O6—Tl2v| 119.90 (17)|
|Sb4v—Sb2—Sb4v| 75.620 (11)| Sb1—O6—Tl2v| 110.88 (16)|
|O6—Sb2—Sb2v| 138.46 (12)| Tl2—O6—Tl2v| 5.21 (15)|
|O10v—Sb2—Sb2v| 93.86 (8)| Sb4—O7—Sb3| 130.09 (14)|
|O10—Sb2—Sb2v| 93.86 (8)| Sb1—O8—Sb4| 138.08 (15)|
|O2v—Sb2—Sb2v| 38.31 (11)| Sb4iv—O9—Sb3| 131.67 (14)|
O2vii—Sb2—Sb2x 35.07 (10)  
O4vii—Sb2—Sb2x 131.32 (10)  
Sb2—O10—Sb4x 106.51 (12)  

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