

# $(\mu\text{-}2,3\text{-Dibromosuccinato-}\kappa^2\text{O}^1:\text{O}^4)\text{bis-}[\text{methanolato-}\kappa\text{O})\text{triphenylantimony(V)}]$

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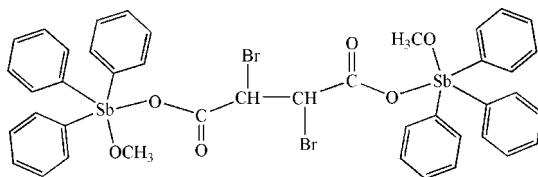
Received 25 April 2011; accepted 28 April 2011

Key indicators: single-crystal X-ray study;  $T = 298\text{ K}$ ; mean  $\sigma(\text{C}-\text{C}) = 0.013\text{ \AA}$ ; disorder in main residue;  $R$  factor = 0.048;  $wR$  factor = 0.131; data-to-parameter ratio = 14.9.

In the title molecule,  $[\text{Sb}_2(\text{C}_6\text{H}_5)_6(\text{C}_4\text{H}_4\text{Br}_2\text{O}_4)(\text{CH}_3\text{O})_2]$ , two  $[\text{Sb}(\text{CH}_3\text{O})\text{Ph}_3]^+$  units are linked by the two carboxylate O atoms of a *meso*-2,3-dibromosuccinate bridging ligand, forming a dinuclear compound. The  $\text{Sb}^{\text{IV}}$  atom is five-coordinated in a slightly distorted trigonal-bipyramid geometry by phenyl C atoms in the equatorial positions and two O atoms in the axial positions.  $\text{C}-\text{H}\cdots\text{O}$  interactions link the molecules into a two-dimensional network parallel to (010). The  $-\text{CH}-$  group of the centrosymmetric 2,3-dibromosuccinate anion is disordered over two sites in a 0.6:0.4 ratio.

## Related literature

For the synthesis and structural characteristics of related organophenylantimony compounds, see: Yin *et al.* (2008).



## Experimental

### Crystal data

$[\text{Sb}_2(\text{C}_6\text{H}_5)_6(\text{C}_4\text{H}_2\text{Br}_2\text{O}_4)(\text{CH}_3\text{O})_2]$

$M_r = 1042.04$

Triclinic,  $P\bar{1}$

$a = 8.707(8)\text{ \AA}$

$b = 9.872(8)\text{ \AA}$

$c = 12.779(10)\text{ \AA}$

$\alpha = 103.74(2)^\circ$

$\beta = 97.93(2)^\circ$

$\gamma = 100.45(2)^\circ$

$V = 1030.3(15)\text{ \AA}^3$

$Z = 1$

Mo  $K\alpha$  radiation

$\mu = 3.29\text{ mm}^{-1}$   
 $T = 298\text{ K}$

$0.22 \times 0.16 \times 0.13\text{ mm}$

### Data collection

Siemens SMART CCD area-detector diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.531$ ,  $T_{\max} = 0.674$

5379 measured reflections  
3582 independent reflections  
2582 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.032$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.131$   
 $S = 1.00$   
3582 reflections  
240 parameters

2 restraints  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 1.10\text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.68\text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

|            |           |            |             |
|------------|-----------|------------|-------------|
| Sb1—O3     | 1.996 (5) | Sb1—C9     | 2.122 (7)   |
| Sb1—C15    | 2.118 (7) | Sb1—O1     | 2.177 (4)   |
| Sb1—C3     | 2.119 (7) |            |             |
| O3—Sb1—C15 | 92.7 (2)  | O3—Sb1—O1  | 176.33 (19) |
| O3—Sb1—C3  | 87.8 (2)  | C15—Sb1—O1 | 90.7 (2)    |
| O3—Sb1—C9  | 93.9 (3)  | C3—Sb1—O1  | 91.2 (2)    |
| C15—Sb1—C9 | 119.3 (3) | C9—Sb1—O1  | 83.2 (2)    |
| C3—Sb1—C9  | 113.2 (3) |            |             |

Table 2

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

| $D-H\cdots A$                     | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|-------|-------------|-------------|---------------|
| C19—H19 $\cdots$ O2 <sup>i</sup>  | 0.93  | 2.50        | 3.388 (11)  | 161           |
| C17—H17 $\cdots$ O3 <sup>ii</sup> | 0.93  | 2.59        | 3.435 (10)  | 152           |

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

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and DIAMOND (Brandenburg, 1998); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2282).

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## supporting information

*Acta Cryst.* (2011). E67, m713 [https://doi.org/10.1107/S1600536811016114]

**( $\mu$ -2,3-Dibromosuccinato- $\kappa^2\text{O}^1:\text{O}^4$ )bis[methanolato- $\kappa\text{O}$ ]triphenylantimony(V)]**

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### S1. Comment

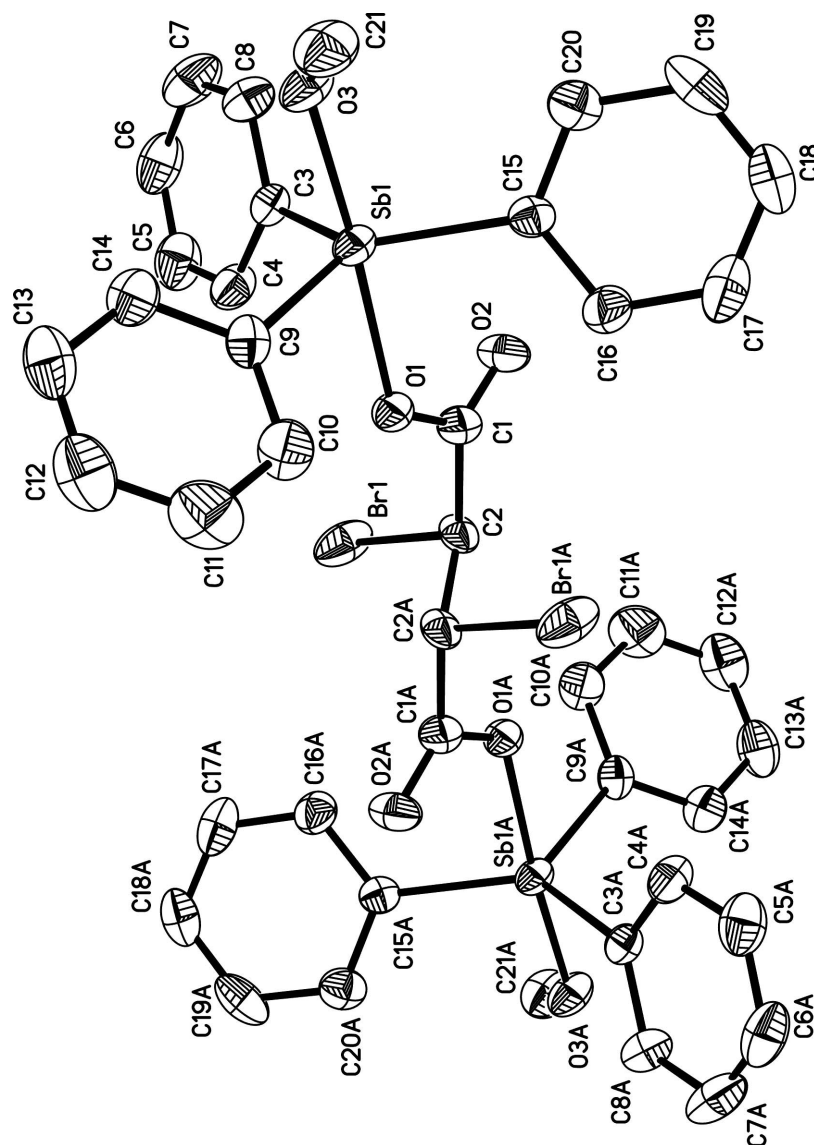
The similar compound of organotin from meso-2,3-dibromosuccinic acid was synthesized by Professor Yin (Yin *et al.*, 2008). The Sb atom is five-coordinated (Figure 1) with an average Sb-C distance of 2.120 Å almost equal to the average Sn-C distance of 2.123 Å and average Sb-O distance of 2.087 Å much shorter than 2.182 Å of Sn-O distance (Yin *et al.*, 2008). The feature in the solid state structure is the 2D network structure infinitely linked by C19-H19...O2 (2.496 Å of H19...O2) and C17-H17...O3 (2.585 Å of H17...O3) interactions (Figure 2), while the organotin give a 3D network triorganotin(IV) polymer (Yin *et al.*, 2008). The title unit is sited in a symmetrical center, and the coordination environment around the antimony atom is described as a slightly distorted trigonal bipyramid with three carbon atoms from the discrete phenyls in equatorial positions and two oxygen atoms occupying the axial positions with O1-Sb1-O2 angle 176.33 (19)°. The SbC<sub>3</sub> unit is planar (the sum of the C-Sb-C angle is 359.9 °) and the O-Sb-C angles are in the range of 83.2 (2)/93.9 (3)°.

### S2. Experimental

2,3-dibromosuccinic acid (0.276 g, 1.00 mmol) was added to the solution of sodium methoxide (0.108 g, 2.00 mmol) in methanol (15 ml), and the mixture was refluxed with stirring for 30 min. Triphenylantimony chloride (0.423 g, 1.00 mmol) in toluene (25 ml) was then added to the mixture, and the reaction was allowed to continue for 8h under refluxing. After cooling down to room temperature, the resulting solution was filtered. The solvent was removed by evaporation under vacuum leaving a colorless solid. The product was recrystallized from dichloromethane/petroleum ether.

### S3. Refinement

All H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å, U<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(C) and C—H = 0.96 Å / 0.98 Å, U<sub>iso</sub>(H) = 1.5U<sub>eq</sub>(C).



**Figure 1**

Molecular structure of compound (1) showing displacement ellipsoids drawn at the 30% probability level.

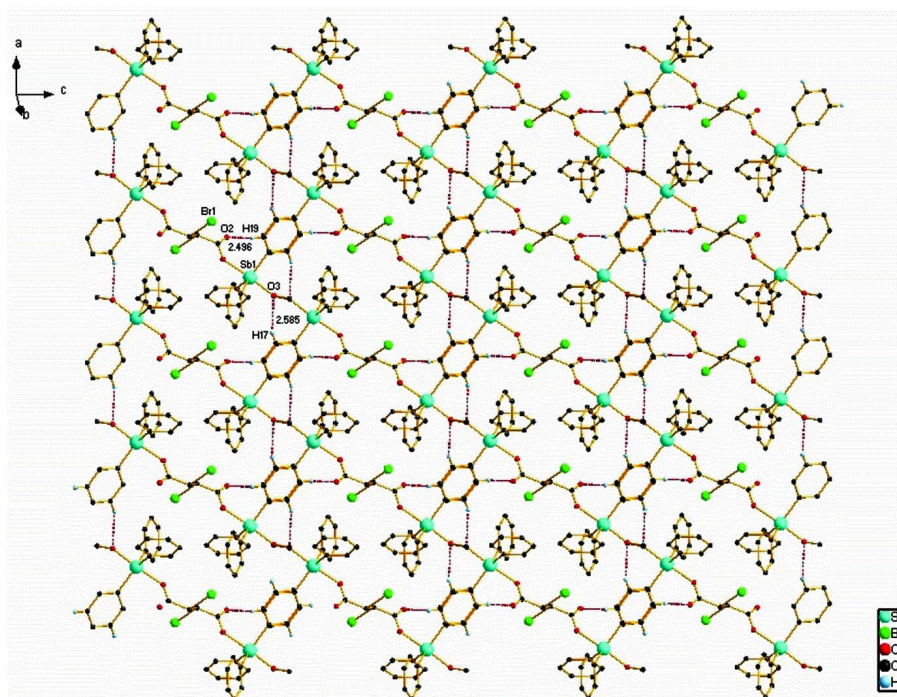


Figure 2

2D network structure infinitely by C19-H19...O2 and C17-H17...O3 interactions [Symmetry code: (i)  $-x, 2-y, 1-z$ ; (ii)  $-1+x, +y, +z$ ].

**( $\mu$ -2,3-Dibromosuccinato- $\kappa^2 O^1:O^4$ )bis[methanolato- $\kappa O$ ]triphenylantimony(V)]**

#### Crystal data

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$c = 12.779(10) \text{ \AA}$

$\alpha = 103.74(2)^\circ$

$\beta = 97.93(2)^\circ$

$\gamma = 100.45(2)^\circ$

$V = 1030.3(15) \text{ \AA}^3$

$Z = 1$

$F(000) = 510$

$D_x = 1.679 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1605 reflections

$\theta = 2.4\text{--}25.2^\circ$

$\mu = 3.29 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, colorless

$0.22 \times 0.16 \times 0.13 \text{ mm}$

#### Data collection

Siemens SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.531$ ,  $T_{\max} = 0.674$

5379 measured reflections

3582 independent reflections

2582 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 1.7^\circ$

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = -8 \rightarrow 15$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.048$  $wR(F^2) = 0.131$  $S = 1.00$ 

3582 reflections

240 parameters

2 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0705P)^2]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 1.10 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.68 \text{ e } \text{\AA}^{-3}$ *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.

**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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|--------------|--------------|-------------|----------------------------------|-----------|
| Sb1 | 0.22561 (5)  | 0.81465 (5)  | 0.69951 (4) | 0.04269 (19)                     |           |
| Br1 | 0.22321 (10) | 1.18121 (10) | 1.08223 (7) | 0.0758 (3)                       |           |
| O1  | 0.1245 (5)   | 0.8897 (5)   | 0.8434 (4)  | 0.0449 (11)                      |           |
| O2  | 0.0650 (6)   | 1.0844 (5)   | 0.8011 (4)  | 0.0593 (13)                      |           |
| O3  | 0.3285 (6)   | 0.7419 (5)   | 0.5737 (4)  | 0.0595 (14)                      |           |
| C1  | 0.0653 (9)   | 1.0029 (9)   | 0.8600 (6)  | 0.0528 (19)                      |           |
| C2  | 0.028 (2)    | 1.0561 (18)  | 0.9733 (9)  | 0.045 (3)                        | 0.60 (2)  |
| H2  | −0.0534      | 1.1125       | 0.9678      | 0.054*                           | 0.60 (2)  |
| C2' | −0.043 (2)   | 1.001 (3)    | 0.9429 (16) | 0.045 (3)                        | 0.40 (2)  |
| H2' | −0.0900      | 1.0854       | 0.9512      | 0.054*                           | 0.40 (2)  |
| C3  | 0.4220 (8)   | 0.9923 (7)   | 0.7547 (6)  | 0.0433 (16)                      |           |
| C4  | 0.4634 (8)   | 1.0663 (8)   | 0.8653 (7)  | 0.059 (2)                        |           |
| H4  | 0.4005       | 1.0413       | 0.9140      | 0.071*                           |           |
| C5  | 0.5985 (10)  | 1.1778 (9)   | 0.9040 (8)  | 0.074 (3)                        |           |
| H5  | 0.6257       | 1.2256       | 0.9783      | 0.089*                           |           |
| C6  | 0.6886 (10)  | 1.2161 (10)  | 0.8353 (9)  | 0.077 (3)                        |           |
| H6  | 0.7770       | 1.2918       | 0.8612      | 0.092*                           |           |
| C7  | 0.6504 (11)  | 1.1430 (11)  | 0.7251 (10) | 0.086 (3)                        |           |
| H7  | 0.7145       | 1.1688       | 0.6774      | 0.103*                           |           |
| C8  | 0.5161 (9)   | 1.0307 (8)   | 0.6851 (7)  | 0.062 (2)                        |           |
| H8  | 0.4911       | 0.9822       | 0.6109      | 0.074*                           |           |
| C9  | 0.2537 (8)   | 0.6454 (7)   | 0.7711 (6)  | 0.0472 (17)                      |           |
| C10 | 0.1370 (10)  | 0.5804 (8)   | 0.8189 (7)  | 0.069 (2)                        |           |
| H10 | 0.0415       | 0.6098       | 0.8200      | 0.083*                           |           |

|      |              |             |            |             |
|------|--------------|-------------|------------|-------------|
| C11  | 0.1654 (14)  | 0.4720 (10) | 0.8645 (9) | 0.096 (3)   |
| H11  | 0.0868       | 0.4269      | 0.8950     | 0.115*      |
| C12  | 0.3079 (14)  | 0.4287 (10) | 0.8658 (9) | 0.086 (3)   |
| H12  | 0.3252       | 0.3558      | 0.8975     | 0.103*      |
| C13  | 0.4230 (12)  | 0.4940 (10) | 0.8202 (8) | 0.079 (3)   |
| H13  | 0.5202       | 0.4673      | 0.8225     | 0.094*      |
| C14  | 0.3953 (9)   | 0.6001 (8)  | 0.7704 (7) | 0.061 (2)   |
| H14  | 0.4721       | 0.6410      | 0.7364     | 0.074*      |
| C15  | 0.0083 (8)   | 0.7988 (7)  | 0.5950 (5) | 0.0427 (16) |
| C16  | −0.1375 (9)  | 0.7702 (8)  | 0.6290 (7) | 0.059 (2)   |
| H16  | −0.1398      | 0.7584      | 0.6989     | 0.071*      |
| C17  | −0.2776 (10) | 0.7594 (10) | 0.5594 (9) | 0.077 (3)   |
| H17  | −0.3734      | 0.7400      | 0.5832     | 0.092*      |
| C18  | −0.2792 (12) | 0.7763 (10) | 0.4578 (9) | 0.076 (3)   |
| H18  | −0.3747      | 0.7696      | 0.4121     | 0.091*      |
| C19  | −0.1357 (13) | 0.8041 (9)  | 0.4223 (7) | 0.079 (3)   |
| H19  | −0.1355      | 0.8148      | 0.3520     | 0.095*      |
| C20  | 0.0100 (10)  | 0.8161 (8)  | 0.4921 (7) | 0.060 (2)   |
| H20  | 0.1057       | 0.8356      | 0.4682     | 0.072*      |
| C21  | 0.2653 (11)  | 0.6019 (9)  | 0.5003 (7) | 0.076 (3)   |
| H21A | 0.1873       | 0.5493      | 0.5306     | 0.114*      |
| H21B | 0.3498       | 0.5524      | 0.4905     | 0.114*      |
| H21C | 0.2167       | 0.6103      | 0.4307     | 0.114*      |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$    | $U^{13}$   | $U^{23}$    |
|-----|------------|------------|------------|-------------|------------|-------------|
| Sb1 | 0.0395 (3) | 0.0443 (3) | 0.0438 (3) | 0.0105 (2)  | 0.0141 (2) | 0.0065 (2)  |
| Br1 | 0.0568 (5) | 0.0780 (6) | 0.0655 (6) | −0.0172 (4) | 0.0232 (4) | −0.0143 (5) |
| O1  | 0.048 (3)  | 0.049 (3)  | 0.043 (3)  | 0.021 (2)   | 0.016 (2)  | 0.013 (2)   |
| O2  | 0.080 (4)  | 0.061 (3)  | 0.048 (3)  | 0.023 (3)   | 0.027 (3)  | 0.022 (3)   |
| O3  | 0.050 (3)  | 0.061 (3)  | 0.062 (3)  | 0.012 (2)   | 0.023 (3)  | −0.002 (3)  |
| C1  | 0.055 (4)  | 0.066 (5)  | 0.044 (4)  | 0.020 (4)   | 0.018 (4)  | 0.017 (4)   |
| C2  | 0.057 (10) | 0.055 (11) | 0.025 (6)  | 0.025 (7)   | 0.006 (7)  | 0.004 (6)   |
| C2' | 0.057 (10) | 0.055 (11) | 0.025 (6)  | 0.025 (7)   | 0.006 (7)  | 0.004 (6)   |
| C3  | 0.038 (4)  | 0.044 (4)  | 0.049 (4)  | 0.012 (3)   | 0.011 (3)  | 0.011 (3)   |
| C4  | 0.044 (4)  | 0.067 (5)  | 0.060 (5)  | 0.008 (4)   | 0.013 (4)  | 0.006 (4)   |
| C5  | 0.057 (5)  | 0.070 (6)  | 0.077 (6)  | 0.007 (4)   | −0.005 (5) | 0.000 (5)   |
| C6  | 0.045 (5)  | 0.061 (6)  | 0.109 (8)  | 0.002 (4)   | 0.004 (5)  | 0.008 (6)   |
| C7  | 0.063 (6)  | 0.082 (7)  | 0.121 (9)  | 0.006 (5)   | 0.046 (6)  | 0.034 (7)   |
| C8  | 0.057 (5)  | 0.060 (5)  | 0.070 (6)  | 0.009 (4)   | 0.027 (4)  | 0.018 (4)   |
| C9  | 0.044 (4)  | 0.038 (4)  | 0.051 (4)  | 0.007 (3)   | −0.001 (4) | 0.004 (3)   |
| C10 | 0.065 (5)  | 0.054 (5)  | 0.091 (7)  | 0.010 (4)   | 0.014 (5)  | 0.030 (5)   |
| C11 | 0.105 (8)  | 0.070 (7)  | 0.108 (9)  | −0.005 (6)  | 0.014 (7)  | 0.038 (6)   |
| C12 | 0.100 (8)  | 0.052 (6)  | 0.100 (8)  | 0.015 (6)   | −0.006 (7) | 0.023 (5)   |
| C13 | 0.082 (6)  | 0.062 (6)  | 0.088 (7)  | 0.028 (5)   | −0.007 (6) | 0.017 (5)   |
| C14 | 0.061 (5)  | 0.052 (5)  | 0.069 (6)  | 0.017 (4)   | 0.013 (4)  | 0.010 (4)   |
| C15 | 0.048 (4)  | 0.038 (4)  | 0.037 (4)  | 0.006 (3)   | 0.010 (3)  | 0.002 (3)   |



|     |           |           |           |           |            |            |
|-----|-----------|-----------|-----------|-----------|------------|------------|
| C16 | 0.054 (5) | 0.071 (5) | 0.051 (5) | 0.019 (4) | 0.015 (4)  | 0.008 (4)  |
| C17 | 0.044 (5) | 0.078 (6) | 0.096 (8) | 0.015 (4) | 0.005 (5)  | 0.001 (6)  |
| C18 | 0.069 (6) | 0.069 (6) | 0.080 (7) | 0.018 (5) | −0.018 (5) | 0.020 (5)  |
| C19 | 0.116 (8) | 0.067 (6) | 0.043 (5) | 0.015 (6) | −0.012 (6) | 0.014 (4)  |
| C20 | 0.063 (5) | 0.054 (5) | 0.057 (5) | 0.000 (4) | 0.010 (4)  | 0.014 (4)  |
| C21 | 0.081 (6) | 0.072 (6) | 0.068 (6) | 0.023 (5) | 0.025 (5)  | −0.003 (5) |

*Geometric parameters (Å, °)*

|                        |             |             |            |
|------------------------|-------------|-------------|------------|
| Sb1—O3                 | 1.996 (5)   | C8—H8       | 0.9300     |
| Sb1—C15                | 2.118 (7)   | C9—C14      | 1.386 (10) |
| Sb1—C3                 | 2.119 (7)   | C9—C10      | 1.391 (10) |
| Sb1—C9                 | 2.122 (7)   | C10—C11     | 1.378 (12) |
| Sb1—O1                 | 2.177 (4)   | C10—H10     | 0.9300     |
| Br1—C2                 | 2.032 (18)  | C11—C12     | 1.383 (14) |
| Br1—C2 <sup>i</sup>    | 2.09 (2)    | C11—H11     | 0.9300     |
| O1—C1                  | 1.299 (8)   | C12—C13     | 1.366 (14) |
| O2—C1                  | 1.225 (8)   | C12—H12     | 0.9300     |
| O3—C21                 | 1.435 (9)   | C13—C14     | 1.387 (11) |
| C1—C2'                 | 1.515 (10)  | C13—H13     | 0.9300     |
| C1—C2                  | 1.521 (9)   | C14—H14     | 0.9300     |
| C2—C2 <sup>i</sup>     | 1.48 (3)    | C15—C20     | 1.367 (10) |
| C2—H2                  | 0.9800      | C15—C16     | 1.401 (10) |
| C2'—C2 <sup>i</sup>    | 1.56 (5)    | C16—C17     | 1.378 (11) |
| C2'—Br1 <sup>i</sup>   | 2.09 (2)    | C16—H16     | 0.9300     |
| C2'—H2'                | 0.9800      | C17—C18     | 1.346 (13) |
| C3—C8                  | 1.362 (10)  | C17—H17     | 0.9300     |
| C3—C4                  | 1.392 (10)  | C18—C19     | 1.390 (13) |
| C4—C5                  | 1.397 (11)  | C18—H18     | 0.9300     |
| C4—H4                  | 0.9300      | C19—C20     | 1.414 (12) |
| C5—C6                  | 1.330 (13)  | C19—H19     | 0.9300     |
| C5—H5                  | 0.9300      | C20—H20     | 0.9300     |
| C6—C7                  | 1.383 (14)  | C21—H21A    | 0.9600     |
| C6—H6                  | 0.9300      | C21—H21B    | 0.9600     |
| C7—C8                  | 1.397 (12)  | C21—H21C    | 0.9600     |
| C7—H7                  | 0.9300      |             |            |
| O3—Sb1—C15             | 92.7 (2)    | C3—C8—C7    | 120.0 (8)  |
| O3—Sb1—C3              | 87.8 (2)    | C3—C8—H8    | 120.0      |
| C15—Sb1—C3             | 127.3 (3)   | C7—C8—H8    | 120.0      |
| O3—Sb1—C9              | 93.9 (3)    | C14—C9—C10  | 119.6 (7)  |
| C15—Sb1—C9             | 119.3 (3)   | C14—C9—Sb1  | 117.8 (6)  |
| C3—Sb1—C9              | 113.2 (3)   | C10—C9—Sb1  | 122.6 (5)  |
| O3—Sb1—O1              | 176.33 (19) | C11—C10—C9  | 118.9 (8)  |
| C15—Sb1—O1             | 90.7 (2)    | C11—C10—H10 | 120.5      |
| C3—Sb1—O1              | 91.2 (2)    | C9—C10—H10  | 120.5      |
| C9—Sb1—O1              | 83.2 (2)    | C10—C11—C12 | 121.6 (10) |
| C2—Br1—C2 <sup>i</sup> | 37.5 (5)    | C10—C11—H11 | 119.2      |

|                                       |            |               |           |
|---------------------------------------|------------|---------------|-----------|
| C1—O1—Sb1                             | 123.3 (4)  | C12—C11—H11   | 119.2     |
| C21—O3—Sb1                            | 120.8 (5)  | C13—C12—C11   | 119.3 (9) |
| O2—C1—O1                              | 125.4 (6)  | C13—C12—H12   | 120.3     |
| O2—C1—C2'                             | 121.6 (11) | C11—C12—H12   | 120.3     |
| O1—C1—C2'                             | 111.1 (11) | C12—C13—C14   | 120.2 (9) |
| O2—C1—C2                              | 116.6 (8)  | C12—C13—H13   | 119.9     |
| O1—C1—C2                              | 116.5 (8)  | C14—C13—H13   | 119.9     |
| C2'—C1—C2                             | 28.0 (7)   | C9—C14—C13    | 120.4 (8) |
| C2 <sup>i</sup> —C2—C1                | 115.5 (14) | C9—C14—H14    | 119.8     |
| C2 <sup>i</sup> —C2—Br1               | 103.3 (13) | C13—C14—H14   | 119.8     |
| C1—C2—Br1                             | 111.7 (10) | C20—C15—C16   | 119.3 (7) |
| C2 <sup>i</sup> —C2—H2                | 108.7      | C20—C15—Sb1   | 119.5 (5) |
| C1—C2—H2                              | 108.7      | C16—C15—Sb1   | 121.2 (5) |
| Br1—C2—H2                             | 108.7      | C17—C16—C15   | 120.3 (8) |
| C1—C2'—C2 <sup>i</sup>                | 113.1 (18) | C17—C16—H16   | 119.9     |
| C1—C2'—Br1 <sup>i</sup>               | 118.2 (14) | C15—C16—H16   | 119.9     |
| C2 <sup>i</sup> —C2'—Br1 <sup>i</sup> | 97.0 (18)  | C18—C17—C16   | 121.7 (9) |
| C1—C2'—H2'                            | 109.3      | C18—C17—H17   | 119.2     |
| C2 <sup>i</sup> —C2'—H2'              | 109.3      | C16—C17—H17   | 119.2     |
| Br1 <sup>i</sup> —C2'—H2'             | 109.3      | C17—C18—C19   | 118.9 (8) |
| C8—C3—C4                              | 118.6 (7)  | C17—C18—H18   | 120.5     |
| C8—C3—Sb1                             | 121.3 (6)  | C19—C18—H18   | 120.5     |
| C4—C3—Sb1                             | 120.0 (5)  | C18—C19—C20   | 120.7 (8) |
| C3—C4—C5                              | 120.6 (8)  | C18—C19—H19   | 119.7     |
| C3—C4—H4                              | 119.7      | C20—C19—H19   | 119.7     |
| C5—C4—H4                              | 119.7      | C15—C20—C19   | 119.2 (8) |
| C6—C5—C4                              | 120.5 (9)  | C15—C20—H20   | 120.4     |
| C6—C5—H5                              | 119.8      | C19—C20—H20   | 120.4     |
| C4—C5—H5                              | 119.8      | O3—C21—H21A   | 109.5     |
| C5—C6—C7                              | 119.9 (9)  | O3—C21—H21B   | 109.5     |
| C5—C6—H6                              | 120.1      | H21A—C21—H21B | 109.5     |
| C7—C6—H6                              | 120.1      | O3—C21—H21C   | 109.5     |
| C6—C7—C8                              | 120.4 (9)  | H21A—C21—H21C | 109.5     |
| C6—C7—H7                              | 119.8      | H21B—C21—H21C | 109.5     |
| C8—C7—H7                              | 119.8      |               |           |

Symmetry code: (i)  $-x, -y+2, -z+2$ .

#### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

| $D-H\cdots A$                      | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C19—H19 $\cdots$ O2 <sup>ii</sup>  | 0.93  | 2.50        | 3.388 (11)  | 161           |
| C17—H17 $\cdots$ O3 <sup>iii</sup> | 0.93  | 2.59        | 3.435 (10)  | 152           |

Symmetry codes: (ii)  $-x, -y+2, -z+1$ ; (iii)  $x-1, y, z$ .