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## Structure Reports

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Bis(2-hydroxybenzaldehyde oximato- $\kappa$ O)triphenylantimony(V)

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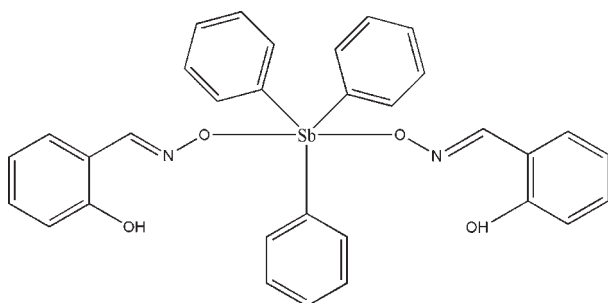
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.031;  $wR$  factor = 0.085; data-to-parameter ratio = 19.6.

The molecule of the title compound,  $[\text{Sb}(\text{C}_6\text{H}_5)_3(\text{C}_7\text{H}_6\text{NO}_2)_2]$ , is located on a twofold axis defined by the metal center and two C atoms of a coordinated phenyl group. The Sb center has a slightly distorted trigonal-bipyramidal geometry, with the axial positions occupied by the O atoms of symmetry-related 2-hydroxybenzaldehyde oximate ligands. An intramolecular  $\text{O}-\text{H}\cdots\text{N}$  interaction is present. The crystal structure is stabilized by  $\text{C}-\text{H}\cdots\text{O}$  interactions.

## Related literature

For related structures, see: Wang *et al.* (2004); Sharutin *et al.* (2004).



## Experimental

## Crystal data

$[\text{Sb}(\text{C}_6\text{H}_5)_3(\text{C}_7\text{H}_6\text{NO}_2)_2]$   
 $M_r = 625.31$   
 Tetragonal,  $I4_1/a$   
 $a = 16.6012$  (15) Å  
 $c = 20.775$  (2) Å  
 $V = 5725.6$  (9) Å<sup>3</sup>

$Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 1.00$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.38 \times 0.30 \times 0.17$  mm

## Data collection

Siemens SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.702$ ,  $T_{\max} = 0.848$

17411 measured reflections  
 3487 independent reflections  
 2410 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$   
 $wR(F^2) = 0.085$   
 $S = 1.09$   
 3487 reflections

178 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 1.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.68$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C7}-\text{H7}\cdots\text{O2}^i$	0.93	2.58	3.294 (5)	134
$\text{O2}-\text{H2}\cdots\text{N1}$	0.82	1.89	2.617 (4)	146

Symmetry code: (i)  $-y + \frac{7}{4}, x + \frac{1}{4}, z + \frac{1}{4}$ .

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: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

We acknowledge the National Natural Science Foundation of China (20771053) and the Natural Science Foundation of Shandong Province (Y2008B48) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2230).

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## supporting information

*Acta Cryst.* (2009). E65, m1438 [https://doi.org/10.1107/S1600536809043542]

**Bis(2-hydroxybenzaldehyde oximato- $\kappa$ O)triphenylantimony(V)****Lei Dong, Handong Yin, Liyuan Wen and Daqi Wang****S1. Comment**

Among numerous multidentate organic ligands, oximes are of particular importance, because of their remarkable structural diversity and biological implications. As a continuation of our interest in this area, we have synthesized the title complex. The molecular structures of the compound is depicted in Fig.1. The antimony atom in the compound is five-coordinate, and its geometries is represented as a distorted trigonal bipyramid. The Sb—O bond distance of 2.078 (2) Å is similar to that reported by Wang (Wang *et al.*, 2004; 2.094 (2)Å). The Sb—C bond distances [2.102 (3) Å; 2.097 (5) Å] fall in the normal range for Sb—C(phenyl) bonds. In the crystal structure, molecules are linked by C—H $\cdots$ O hydrogen bonds.

**S2. Experimental**

Salicylaldoxime (0.4 mmol) was added to a methanol solution of sodium (0.4 mmol) and heated under reflux for 0.5 h. To this solution was added triphenylantimony dichloride (0.2 mmol) in benzene and the mixture was refluxed for 5 h, cooled and filtered. The filtrate was evaporated *in vacuo*. The obtained solid was recrystallized from dichloromethane-petroleum ether. (yield 86%. m.p.431k). Anal. Calcd (%) for C<sub>32</sub>H<sub>27</sub>N<sub>2</sub>O<sub>4</sub>Sb (Mr = 625.33): C, 61.46; H, 4.35; N, 4.48; O,10.23. Found (%): C, 61.47; H, 4.40; N, 4.46; O,10.26.

**S3. Refinement**

The C—H and O—H H atoms were positioned with idealized geometry (O—H = 0.82 Å and C—H = 0.93 Å) and were refined using a riding model approximation with U<sub>iso</sub>(H) = 1.2 U<sub>eq</sub>(C, O).The final difference map showed a high residual peak (1.38 e Å<sup>-3</sup>) at a distance of 0.35 Å from H17.

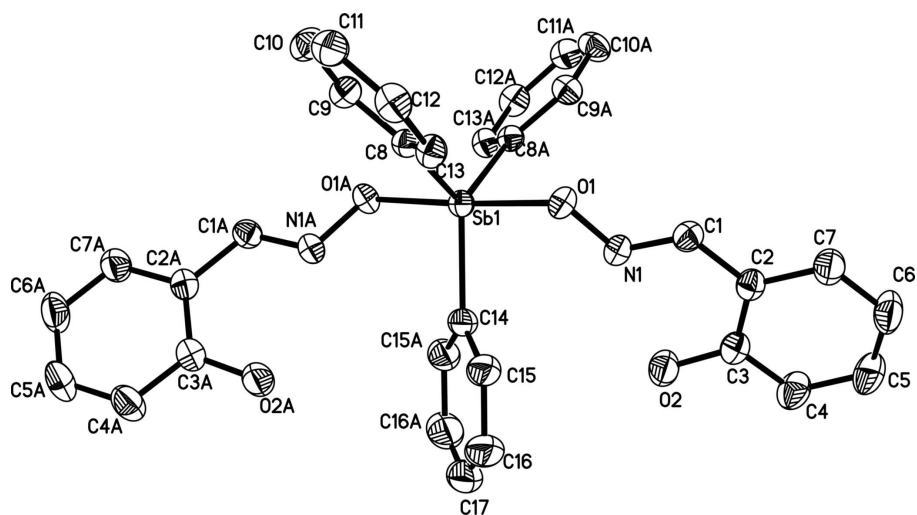


Figure 1

The molecular structure of the compound, showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity. Symmetry code: (A) =  $-x + 1, -y + 3/2, z$ .

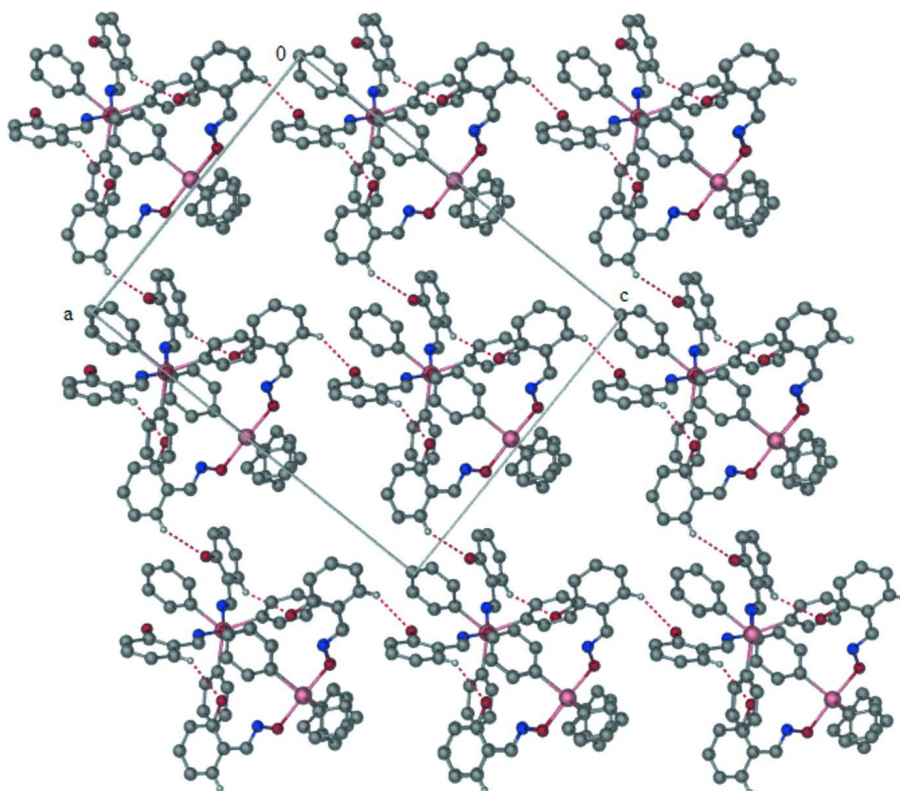


Figure 2

View of the two-dimensional layer structure in the title compound. Intermolecular hydrogen bonds are shown as dashed lines.

Bis(2-hydroxybenzaldehyde oximato- $\kappa$ O)triphenylantimony(V)

Crystal data

[Sb(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>(C<sub>7</sub>H<sub>6</sub>NO<sub>2</sub>)<sub>2</sub>]

$M_r = 625.31$

Tetragonal,  $I4_1/a$

Hall symbol: -I 4ad

$a = 16.6012$  (15) Å

$c = 20.775$  (2) Å

$V = 5725.6$  (9) Å<sup>3</sup>

$Z = 8$

$F(000) = 2528$

$D_x = 1.451$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 5826 reflections

$\theta = 2.5$ – $25.5^\circ$

$\mu = 1.00$  mm<sup>-1</sup>

$T = 298$  K

Block, colourless

$0.38 \times 0.30 \times 0.17$  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)

$T_{\min} = 0.702$ ,  $T_{\max} = 0.848$

17411 measured reflections

3487 independent reflections

2410 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 1.6^\circ$

$h = -21 \rightarrow 21$

$k = -9 \rightarrow 22$

$l = -26 \rightarrow 26$

Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.085$

$S = 1.09$

3487 reflections

178 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.030P)^2 + 9.4858P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.38$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.68$  e Å<sup>-3</sup>

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sb1	0.5000	0.7500	0.977300 (13)	0.03630 (10)
N1	0.65745 (16)	0.80646 (15)	0.93840 (13)	0.0438 (6)
O1	0.62512 (13)	0.74876 (13)	0.97978 (10)	0.0442 (5)
O2	0.66631 (16)	0.89097 (18)	0.83231 (13)	0.0716 (8)
H2	0.6454	0.8611	0.8589	0.107*
C1	0.7300 (2)	0.8254 (2)	0.95055 (16)	0.0474 (8)
H1	0.7551	0.8043	0.9869	0.057*
C2	0.7748 (2)	0.8797 (2)	0.90850 (17)	0.0467 (8)
C3	0.7419 (2)	0.9098 (2)	0.85145 (18)	0.0515 (8)
C4	0.7874 (3)	0.9596 (2)	0.8126 (2)	0.0704 (11)
H4	0.7653	0.9799	0.7747	0.084*
C5	0.8650 (3)	0.9796 (3)	0.8293 (2)	0.0762 (13)
H5	0.8951	1.0130	0.8026	0.091*
C6	0.8981 (3)	0.9506 (3)	0.8848 (2)	0.0778 (13)

H6	0.9506	0.9643	0.8959	0.093*
C7	0.8535 (2)	0.9012 (3)	0.92426 (19)	0.0647 (10)
H7	0.8763	0.8819	0.9621	0.078*
C8	0.50453 (19)	0.64233 (17)	1.03043 (14)	0.0384 (7)
C9	0.4426 (2)	0.62433 (19)	1.07230 (15)	0.0477 (8)
H9	0.3979	0.6579	1.0751	0.057*
C10	0.4475 (3)	0.5556 (2)	1.11027 (17)	0.0573 (9)
H10	0.4062	0.5435	1.1389	0.069*
C11	0.5130 (3)	0.5054 (2)	1.10563 (17)	0.0572 (9)
H11	0.5159	0.4596	1.1313	0.069*
C12	0.5738 (2)	0.5225 (2)	1.06345 (18)	0.0543 (9)
H12	0.6178	0.4880	1.0603	0.065*
C13	0.5701 (2)	0.59106 (19)	1.02534 (16)	0.0458 (8)
H13	0.6114	0.6025	0.9965	0.055*
C14	0.5000	0.7500	0.8764 (2)	0.0523 (12)
C15	0.5554 (3)	0.7042 (2)	0.84211 (19)	0.0655 (11)
H15	0.5931	0.6734	0.8643	0.079*
C16	0.5554 (4)	0.7039 (4)	0.7751 (2)	0.0985 (19)
H16	0.5922	0.6729	0.7522	0.118*
C17	0.5000	0.7500	0.7441 (4)	0.102 (3)
H17	0.5000	0.7500	0.6993	0.123*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sb1	0.03787 (17)	0.03277 (16)	0.03825 (16)	0.00025 (13)	0.000	0.000
N1	0.0412 (15)	0.0414 (14)	0.0487 (15)	-0.0016 (12)	0.0064 (12)	0.0021 (12)
O1	0.0393 (12)	0.0429 (12)	0.0506 (13)	0.0003 (10)	0.0072 (10)	0.0083 (10)
O2	0.0554 (16)	0.093 (2)	0.0665 (17)	-0.0152 (15)	-0.0083 (13)	0.0257 (15)
C1	0.0455 (19)	0.0501 (19)	0.0466 (18)	0.0021 (15)	0.0034 (15)	0.0023 (16)
C2	0.0446 (18)	0.0428 (18)	0.053 (2)	-0.0002 (15)	0.0048 (16)	-0.0019 (16)
C3	0.049 (2)	0.048 (2)	0.057 (2)	-0.0017 (16)	0.0054 (17)	0.0032 (17)
C4	0.067 (3)	0.071 (3)	0.074 (3)	-0.005 (2)	0.005 (2)	0.026 (2)
C5	0.068 (3)	0.068 (3)	0.093 (3)	-0.016 (2)	0.015 (2)	0.022 (2)
C6	0.055 (2)	0.082 (3)	0.096 (3)	-0.023 (2)	0.004 (2)	0.014 (3)
C7	0.054 (2)	0.075 (3)	0.065 (2)	-0.009 (2)	-0.0021 (19)	0.011 (2)
C8	0.0441 (17)	0.0323 (15)	0.0387 (16)	-0.0002 (13)	-0.0010 (13)	-0.0017 (13)
C9	0.052 (2)	0.0418 (18)	0.0489 (19)	0.0026 (15)	0.0063 (16)	-0.0012 (15)
C10	0.074 (3)	0.0462 (19)	0.052 (2)	-0.0065 (18)	0.0121 (18)	0.0057 (16)
C11	0.080 (3)	0.0347 (17)	0.057 (2)	-0.0026 (18)	-0.007 (2)	0.0049 (16)
C12	0.059 (2)	0.0369 (17)	0.067 (2)	0.0070 (16)	-0.0069 (19)	-0.0009 (17)
C13	0.0474 (19)	0.0372 (17)	0.0527 (19)	0.0004 (14)	0.0028 (16)	-0.0015 (15)
C14	0.069 (3)	0.048 (3)	0.041 (2)	-0.015 (2)	0.000	0.000
C15	0.079 (3)	0.064 (2)	0.054 (2)	-0.015 (2)	0.010 (2)	-0.0095 (19)
C16	0.127 (5)	0.105 (4)	0.064 (3)	-0.044 (4)	0.027 (3)	-0.030 (3)
C17	0.141 (8)	0.110 (7)	0.055 (4)	-0.065 (6)	0.000	0.000

*Geometric parameters (Å, °)*

Sb1—O1 <sup>i</sup>	2.078 (2)	C7—H7	0.9300
Sb1—O1	2.078 (2)	C8—C9	1.379 (4)
Sb1—C14	2.097 (5)	C8—C13	1.386 (4)
Sb1—C8 <sup>i</sup>	2.102 (3)	C9—C10	1.390 (5)
Sb1—C8	2.102 (3)	C9—H9	0.9300
N1—C1	1.271 (4)	C10—C11	1.373 (5)
N1—O1	1.394 (3)	C10—H10	0.9300
O2—C3	1.353 (4)	C11—C12	1.367 (5)
O2—H2	0.8200	C11—H11	0.9300
C1—C2	1.459 (5)	C12—C13	1.388 (5)
C1—H1	0.9300	C12—H12	0.9300
C2—C7	1.394 (5)	C13—H13	0.9300
C2—C3	1.397 (5)	C14—C15 <sup>i</sup>	1.389 (5)
C3—C4	1.381 (5)	C14—C15	1.389 (5)
C4—C5	1.375 (6)	C15—C16	1.393 (6)
C4—H4	0.9300	C15—H15	0.9300
C5—C6	1.365 (6)	C16—C17	1.359 (7)
C5—H5	0.9300	C16—H16	0.9300
C6—C7	1.375 (5)	C17—C16 <sup>i</sup>	1.359 (7)
C6—H6	0.9300	C17—H17	0.9300
O1 <sup>i</sup> —Sb1—O1	177.16 (12)	C6—C7—H7	119.4
O1 <sup>i</sup> —Sb1—C14	91.42 (6)	C2—C7—H7	119.4
O1—Sb1—C14	91.42 (6)	C9—C8—C13	120.0 (3)
O1 <sup>i</sup> —Sb1—C8 <sup>i</sup>	86.72 (10)	C9—C8—Sb1	119.3 (2)
O1—Sb1—C8 <sup>i</sup>	91.79 (10)	C13—C8—Sb1	120.7 (2)
C14—Sb1—C8 <sup>i</sup>	121.67 (8)	C8—C9—C10	119.5 (3)
O1 <sup>i</sup> —Sb1—C8	91.79 (10)	C8—C9—H9	120.2
O1—Sb1—C8	86.72 (10)	C10—C9—H9	120.2
C14—Sb1—C8	121.67 (8)	C11—C10—C9	120.3 (3)
C8 <sup>i</sup> —Sb1—C8	116.65 (16)	C11—C10—H10	119.9
C1—N1—O1	114.4 (3)	C9—C10—H10	119.9
N1—O1—Sb1	111.27 (16)	C12—C11—C10	120.3 (3)
C3—O2—H2	109.5	C12—C11—H11	119.9
N1—C1—C2	121.1 (3)	C10—C11—H11	119.9
N1—C1—H1	119.4	C11—C12—C13	120.2 (3)
C2—C1—H1	119.4	C11—C12—H12	119.9
C7—C2—C3	118.3 (3)	C13—C12—H12	119.9
C7—C2—C1	119.7 (3)	C8—C13—C12	119.7 (3)
C3—C2—C1	122.0 (3)	C8—C13—H13	120.2
O2—C3—C4	118.3 (3)	C12—C13—H13	120.2
O2—C3—C2	122.0 (3)	C15 <sup>i</sup> —C14—C15	118.4 (5)
C4—C3—C2	119.8 (4)	C15 <sup>i</sup> —C14—Sb1	120.8 (2)
C5—C4—C3	120.6 (4)	C15—C14—Sb1	120.8 (2)
C5—C4—H4	119.7	C14—C15—C16	121.0 (5)
C3—C4—H4	119.7	C14—C15—H15	119.5

C6—C5—C4	120.3 (4)	C16—C15—H15	119.5
C6—C5—H5	119.8	C17—C16—C15	118.1 (6)
C4—C5—H5	119.8	C17—C16—H16	120.9
C5—C6—C7	119.9 (4)	C15—C16—H16	120.9
C5—C6—H6	120.1	C16 <sup>i</sup> —C17—C16	123.4 (7)
C7—C6—H6	120.1	C16 <sup>i</sup> —C17—H17	118.3
C6—C7—C2	121.1 (4)	C16—C17—H17	118.3
C1—N1—O1—Sb1	160.6 (2)	O1—Sb1—C8—C13	-27.7 (2)
O1 <sup>i</sup> —Sb1—O1—N1	-132.17 (17)	C14—Sb1—C8—C13	61.9 (3)
C14—Sb1—O1—N1	47.83 (17)	C8 <sup>i</sup> —Sb1—C8—C13	-118.1 (3)
C8 <sup>i</sup> —Sb1—O1—N1	-73.93 (19)	C13—C8—C9—C10	1.5 (5)
C8—Sb1—O1—N1	169.48 (19)	Sb1—C8—C9—C10	-176.3 (3)
O1—N1—C1—C2	174.7 (3)	C8—C9—C10—C11	-0.8 (5)
N1—C1—C2—C7	178.1 (3)	C9—C10—C11—C12	-0.3 (6)
N1—C1—C2—C3	-3.6 (5)	C10—C11—C12—C13	0.5 (6)
C7—C2—C3—O2	179.2 (3)	C9—C8—C13—C12	-1.3 (5)
C1—C2—C3—O2	0.8 (5)	Sb1—C8—C13—C12	176.5 (2)
C7—C2—C3—C4	-0.2 (5)	C11—C12—C13—C8	0.2 (5)
C1—C2—C3—C4	-178.6 (3)	O1 <sup>i</sup> —Sb1—C14—C15 <sup>i</sup>	38.99 (19)
O2—C3—C4—C5	-179.0 (4)	O1—Sb1—C14—C15 <sup>i</sup>	-141.01 (19)
C2—C3—C4—C5	0.4 (6)	C8 <sup>i</sup> —Sb1—C14—C15 <sup>i</sup>	-48.0 (2)
C3—C4—C5—C6	-0.3 (7)	C8—Sb1—C14—C15 <sup>i</sup>	132.0 (2)
C4—C5—C6—C7	0.0 (7)	O1 <sup>i</sup> —Sb1—C14—C15	-141.01 (19)
C5—C6—C7—C2	0.2 (7)	O1—Sb1—C14—C15	38.99 (19)
C3—C2—C7—C6	-0.1 (6)	C8 <sup>i</sup> —Sb1—C14—C15	132.0 (2)
C1—C2—C7—C6	178.3 (4)	C8—Sb1—C14—C15	-48.0 (2)
O1 <sup>i</sup> —Sb1—C8—C9	-27.5 (3)	C15 <sup>i</sup> —C14—C15—C16	-0.3 (3)
O1—Sb1—C8—C9	150.1 (3)	Sb1—C14—C15—C16	179.7 (3)
C14—Sb1—C8—C9	-120.2 (2)	C14—C15—C16—C17	0.5 (6)
C8 <sup>i</sup> —Sb1—C8—C9	59.8 (2)	C15—C16—C17—C16 <sup>i</sup>	-0.2 (3)
O1 <sup>i</sup> —Sb1—C8—C13	154.7 (2)		

Symmetry code: (i)  $-x+1, -y+3/2, z$ .

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7 <sup>⋯</sup> O2 <sup>ii</sup>	0.93	2.58	3.294 (5)	134
O2—H2 <sup>⋯</sup> N1	0.82	1.89	2.617 (4)	146

Symmetry code: (ii)  $-y+7/4, x+1/4, z+1/4$ .