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Bis(2-hydroxybenzaldehyde oximato-*κO*)triphenylantimony(V)

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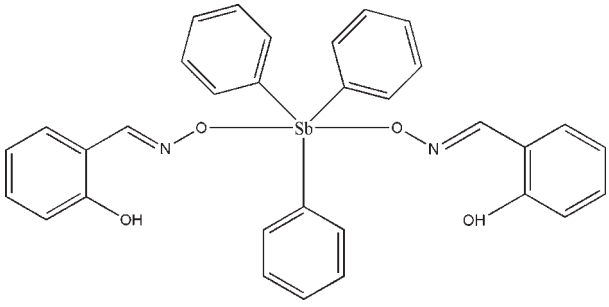
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 19.6.

The molecule of the title compound, $[Sb(C_6H_5)_3(C_7H_6NO_2)_2]$, 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 O—H···N interaction is present. The crystal structure is stabilized by C—H···O interactions.

Related literature

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



Experimental

Crystal data

$[Sb(C_6H_5)_3(C_7H_6NO_2)_2]$
M_r = 625.31
Tetragonal, $I4_1/a$
a = 16.6012 (15) Å
c = 20.775 (2) Å
V = 5725.6 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.00 mm^{-1}
T = 298 K
0.38 × 0.30 × 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_{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
$\Delta\rho_{max}$ = 1.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O2^{i}	0.93	2.58	3.294 (5)	134
O2—H2···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*.

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

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supplementary materials

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Bis(2-hydroxybenzaldehyde oximato-*κO*)triphenylantimony(V)

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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···O hydrogen bonds.

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 $C_{32}H_{27}N_2O_4Sb$ (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.

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_{iso}(H) = 1.2 U_{eq}(C, O).The final difference map showed a high residual peak (1.38 e Å^{-3}) at a distance of 0.35 Å from H17.

Figures

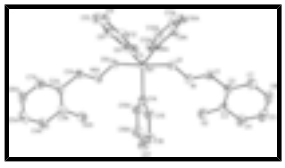


Fig. 1. The molecular structure of the compound, showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity. Symmetry code: (A) = -*x* + 1, -*y* + 3/2, *z*.

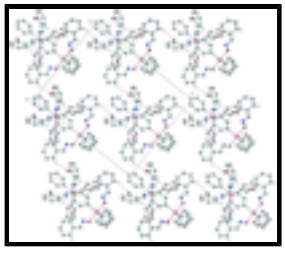


Fig. 2. View of the two-dimensional layer structure in the title compound. Intermolecular hydrogen bonds are shown as dashed lines.

Bis(2-hydroxybenzaldehyde oximato-κ*O*)triphenylantimony(V)

Crystal data

$[Sb(C_6H_5)_3(C_7H_6NO_2)_2]$	$Z = 8$
$M_r = 625.31$	$F_{000} = 2528$
Tetragonal, $I4_1/a$	$D_x = 1.451$ Mg m^{-3}
Hall symbol: -I 4ad	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 16.6012$ (15) Å	Cell parameters from 5826 reflections
$b = 16.6012$ (15) Å	$\theta = 2.5$–25.5º
$c = 20.775$ (2) Å	$\mu = 1.00$ mm^{-1}
$\alpha = 90$º	$T = 298$ K
$\beta = 90$º	Block, colourless
$\gamma = 90$º	0.38 × 0.30 × 0.17 mm
$V = 5725.6$ (9) Å^3	

Data collection

Siemens SMART CCD area-detector diffractometer	3487 independent reflections
Radiation source: fine-focus sealed tube	2410 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
$T = 298$ K	$\theta_{max} = 28.3$º
φ and ω scans	$\theta_{min} = 1.6$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -21 \rightarrow 21$
$T_{min} = 0.702$, $T_{max} = 0.848$	$k = -9 \rightarrow 22$
17411 measured reflections	$l = -26 \rightarrow 26$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.085$	$w = 1/[\sigma^2(F_o^2) + (0.030P)^2 + 9.4858P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.001$
3487 reflections	$\Delta\rho_{max} = 1.38$ e Å^{-3}
178 parameters	$\Delta\rho_{min} = -0.68$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{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^2)

	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^{i}	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^{i}	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^{i}	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^{i}	1.359 (7)
C6—H6	0.9300	C17—H17	0.9300
O1^{i}—Sb1—O1	177.16 (12)	C6—C7—H7	119.4
O1^{i}—Sb1—C14	91.42 (6)	C2—C7—H7	119.4
O1—Sb1—C14	91.42 (6)	C9—C8—C13	120.0 (3)
O1^{i}—Sb1—C8^{i}	86.72 (10)	C9—C8—Sb1	119.3 (2)
O1—Sb1—C8^{i}	91.79 (10)	C13—C8—Sb1	120.7 (2)
C14—Sb1—C8^{i}	121.67 (8)	C8—C9—C10	119.5 (3)
O1^{i}—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^{i}—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^{i}—C14—C15	118.4 (5)
C4—C3—C2	119.8 (4)	C15^{i}—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^{i}—C17—C16	123.4 (7)
C7—C6—H6	120.1	C16^{i}—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^{i}—Sb1—O1—N1	−132.17 (17)	C14—Sb1—C8—C13	61.9 (3)
C14—Sb1—O1—N1	47.83 (17)	C8^{i}—Sb1—C8—C13	−118.1 (3)
C8^{i}—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^{i}—Sb1—C14—C15^{i}	38.99 (19)
O2—C3—C4—C5	−179.0 (4)	O1—Sb1—C14—C15^{i}	−141.01 (19)
C2—C3—C4—C5	0.4 (6)	C8^{i}—Sb1—C14—C15^{i}	−48.0 (2)
C3—C4—C5—C6	−0.3 (7)	C8—Sb1—C14—C15^{i}	132.0 (2)
C4—C5—C6—C7	0.0 (7)	O1^{i}—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^{i}—Sb1—C14—C15	132.0 (2)
C1—C2—C7—C6	178.3 (4)	C8—Sb1—C14—C15	−48.0 (2)
O1^{i}—Sb1—C8—C9	−27.5 (3)	C15^{i}—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^{i}—Sb1—C8—C9	59.8 (2)	C15—C16—C17—C16^{i}	−0.2 (3)
O1^{i}—Sb1—C8—C13	154.7 (2)		

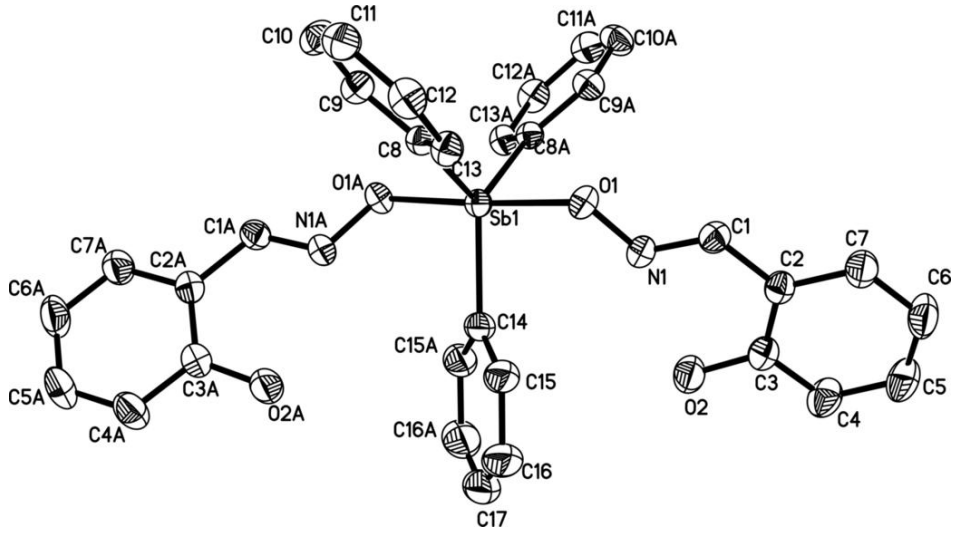
Symmetry codes: (i) −*x*+1, −*y*+3/2, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O2^{ii}	0.93	2.58	3.294 (5)	134
O2—H2···N1	0.82	1.89	2.617 (4)	146

Symmetry codes: (ii) −*y*+7/4, *x*+1/4, *z*+1/4.

Fig. 1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C10
C11A
C10A
C12A
C9
C12
C9A
C13A
C8
C8A
C13
O1A
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C1A
N1A
C1
C7A
C2A
N1
C2
C7
C6A
C6
C14
C15A
C15
C5A
C3A
C3
C5
O2A
O2
C4A
C4
C16A
C16
C17

Fig. 2