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Triphenyl[(2-oxidobenzylideneamino)acetato]-antimony(V) dichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
Disorder in solvent or counterion
R factor = 0.042
wR factor = 0.097
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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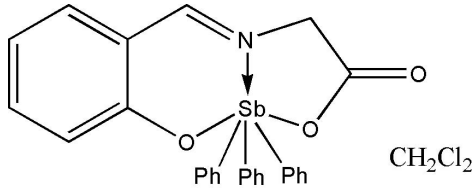
The title compound, $[(OC_6H_4CH{=}NCH_2COO)SbPh_3]\cdot CH_2Cl_2$ or $[Sb(C_6H_5)_3(C_9H_7NO_3)]\cdot CH_2Cl_2$, is a mononuclear antimony(V) complex. The asymmetric unit comprises two independent molecules of the complex and two solvent molecules. In both complex molecules, the Sb atoms are in distorted octahedral environments.

Comment

In recent years, we have found that some triarylantimony carboxylates exhibit high *in vitro* antitumour activity against human tumour cell lines (Li *et al.*, 2001, 2004; Liu *et al.*, 2003; Ma *et al.*, 2001; Yu, Ma, Wang & Li, 2004), often higher than *cis*-platin (Yu, Ma, Liu *et al.*, 2004). As an extension of our work on the structural characterization of the antimony complexes, a heterocyclic mononuclear antimony(V) complex is reported here.



(I)

The asymmetric unit of the title compound, (I), is made up of two crystallographically independent $[(2\text{–}OC_6H_4CH{=}NCH_2COO)SbPh_3]$ molecules (*A* and *B*) and two dichloromethane molecules (Fig. 1). In both complex molecules, the Sb atom is coordinated by the (2-oxidobenzylideneamino)acetate ligand through an O atom from the carboxylate group, a phenoxide O atom and an imino N atom. The Sb centres have a distorted octahedral geometry, with two O atoms (O1/O2 in molecule *A* and O4/O5 in *B*), one N atom (N1 in molecule *A* and N2 in *B*) and one C atom (C16 in molecule *A* and C49 in *B*) occupying the equatorial positions, and two benzene C atoms (C10/C22 in molecule *A* and C37/C43 in *B*) in the axial positions. The three *trans* angles at the Sb^V atom are in the range 159.49 (14)–174.70 (17)° for Sb1 and 159.35 (14)–174.63 (17)° for Sb2. The other angles subtended at the Sb^V atoms are in the range 76.01 (14)–101.55 (16)° for Sb1 and 75.34 (14)–101.26 (16)° for Sb2 (Table 1). Distortions from the ideal geometry may be attributed to the restricted bite angles of the tridentate ligand. None of the five- or six-membered rings formed upon chelation is planar, as seen in the following torsion angles: Sb1—O2—C9—C8 [−4.7 (6)°], Sb1—N1—

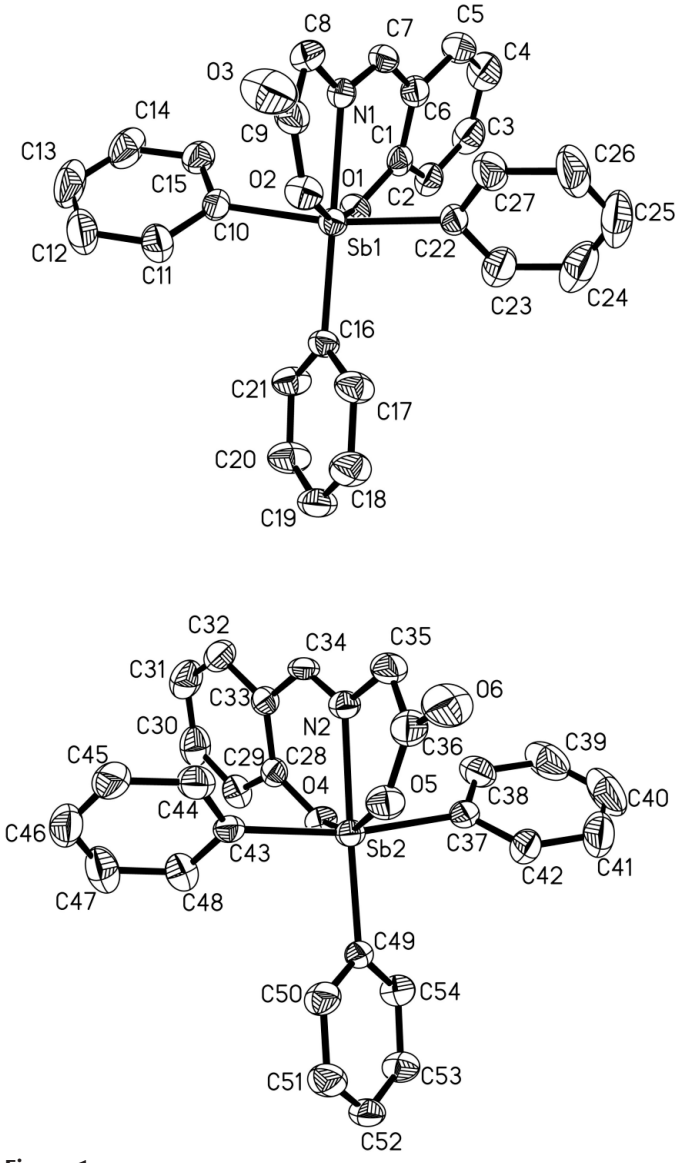


Figure 1
The two complex molecules of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Solvent molecules and H atoms have been omitted for clarity.

C8—C9 [13.8 (5)°], Sb1—O1—C1—C6 [−30.2 (6)°] and Sb1—N1—C7—C6 [15.7 (7)°] for molecule *A*, and Sb2—O5—C36—C35 [14.1 (6)°], Sb2—N2—C35—C36 [−16.3 (5)°], Sb2—O4—C28—C33 [35.4 (6)°] and Sb2—N2—C34—C33 [−8.7 (7)°] for molecule *B*.

Experimental

Potassium (*N*-salicylideneamino)acetate (0.43 g, 2 mmol) in methanol (15 ml) was added dropwise to a solution of triphenylantimony dibromide (0.36 g, 1 mmol) in tetrahydrofuran (15 ml). The reaction mixture was stirred at room temperature for 6 h and then evaporated to dryness *in vacuo*. The resulting solid was recrystallized from dichloromethane–petroleum ether (3:2 *v*:*v*) (yield: 0.75 g, 61%; m.p. 517–519 K). Analysis found: C 54.06, H 4.43, N 2.36%; calculated for $C_{56}H_{48}Cl_4N_2O_6Sb_2$: C 54.67, H 3.93, N 2.28%. ^{1}H NMR: δ 7.89 (*s*, 1H), 6.77–7.59 (*m*, 19H), 4.22 (*s*, 2H).

Crystal data

$[Sb(C_6H_5)_3(C_9H_7NO_3)]$	D_x = 1.518 Mg m^{-3}
M_r = 615.14	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1019 reflections
a = 21.071 (6) Å	θ = 2.8–24.2°
b = 10.944 (3) Å	μ = 1.25 mm^{-1}
c = 25.569 (8) Å	T = 293 (2) K
β = 114.109 (5)°	Block, colourless
V = 5382 (3) Å^3	0.22 × 0.20 × 0.16 mm
Z = 8	

Data collection

Bruker SMART CCD area-detector diffractometer	9460 independent reflections
φ and ω scans	6705 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.041
T_{min} = 0.726, T_{max} = 0.818	θ_{max} = 25.0°
27059 measured reflections	$h = -25 \rightarrow 22$
	$k = -13 \rightarrow 12$
	$l = -23 \rightarrow 30$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 5.6596P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$(\Delta/\sigma)_{max}$ = 0.005
$wR(F^2)$ = 0.097	$\Delta\rho_{max}$ = 0.65 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.53 e Å^{-3}
9460 reflections	
716 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sb1—O1	2.041 (3)	Sb2—O4	2.037 (3)
Sb1—O2	2.083 (3)	Sb2—O5	2.089 (4)
Sb1—C16	2.138 (5)	Sb2—C49	2.139 (5)
Sb1—C22	2.147 (5)	Sb2—C37	2.156 (5)
Sb1—C10	2.151 (5)	Sb2—C43	2.162 (5)
Sb1—N1	2.259 (4)	Sb2—N2	2.249 (4)
O1—Sb1—O2	159.49 (14)	O4—Sb2—O5	159.35 (14)
O1—Sb1—C16	101.55 (16)	O4—Sb2—C49	101.26 (16)
O2—Sb1—C16	98.78 (16)	O5—Sb2—C49	99.29 (17)
O1—Sb1—C22	90.96 (17)	O4—Sb2—C37	87.97 (18)
O2—Sb1—C22	90.19 (18)	O5—Sb2—C37	87.87 (18)
C16—Sb1—C22	94.37 (18)	C49—Sb2—C37	96.07 (18)
O1—Sb1—C10	87.81 (16)	O4—Sb2—C43	89.22 (16)
O2—Sb1—C10	87.73 (16)	O5—Sb2—C43	91.03 (17)
C16—Sb1—C10	94.99 (18)	C49—Sb2—C43	94.93 (17)
C22—Sb1—C10	170.62 (19)	C37—Sb2—C43	168.98 (18)
O1—Sb1—N1	83.71 (14)	O4—Sb2—N2	84.11 (14)
O2—Sb1—N1	76.01 (14)	O5—Sb2—N2	75.34 (14)
C16—Sb1—N1	174.70 (17)	C49—Sb2—N2	174.63 (17)
C22—Sb1—N1	84.75 (16)	C37—Sb2—N2	83.83 (16)
C10—Sb1—N1	85.87 (16)	C43—Sb2—N2	85.28 (16)

Both dichloromethane molecules are found to be disordered: one (C55/Cl1/Cl2) is disordered over two positions and the occupancies of the two disordered positions were refined to 0.557 (7) and 0.443 (7), while the other molecule (C56/Cl3/Cl4) is disordered over three positions and the occupancies of the disordered positions were refined to 0.414 (4), 0.330 (6) and 0.256 (4). The disorder was treated by restraining the C—Cl distance to 1.790 (5) Å and the Cl···Cl distance to 2.82 (1) Å. The displacements of the disordered atoms were approximated to isotropic behaviour. All H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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