Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1296). Services for accessing these data are described at the back of the journal.

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Trichloro[methylenebis(diphenylphosphine oxide-*O*)]antimony(III)

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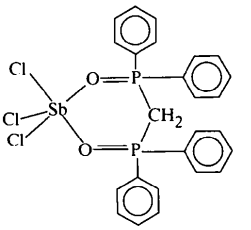
Abstract

The asymmetric unit of the title complex, $[SbCl_3\{[(C_6H_5)_2PO]_2CH_2\}]$, comprises two independent molecules, in both of which the Sb atoms are in flattened square-pyramidal environments, with the basal planes each defined by two O and two Cl atoms, and the apical atom being Cl in each molecule. The geometry can alternatively be described as distorted octahedral as a result of coordination by a sixth (Cl) atom at a longer distance.

Comment

It is known that $SbCl_3$ forms an addition complex with triphenylphosphine oxide, *i.e.* $SbCl_3{\cdot}2(C_6H_5)_3PO$ (Milicev & Hadzi, 1977; Golic & Milicev, 1978). In our study of the reaction of bis(diphenylphosphino)methane with $SbCl_5$ in acetonitrile, an analogous product, (I), was obtained and its structure is reported herein.



(I)

The asymmetric unit consists of two independent molecules which are not related by pseudosymmetry. In both molecules, the Sb atom is coordinated to the methylenebis(diphenylphosphine oxide) ligand through the O atoms. The Sb centres occupy a square-pyramidal geometry, with both oxide O atoms and two Cl atoms (Cl1 and Cl2) occupying the equatorial positions, and the third Cl atom (Cl3) in the axial position. The angles at antimony cluster around 90 or 180°, so that the square pyramid is flattened to an octahedron with one coordination site unoccupied. However, a sixth coordination is observed for both Sb atoms, at longer distances, with Sb1 coordinated to Cl2A^{i} at a distance of 3.6858 (14) Å and Sb2 coordinated to Cl2B^{ii} at a distance of 3.5450 (13) Å [symmetry codes: (i) $2-x$, $-y$, $2-z$; (ii) $2-x$, $1-y$, $1-z$]. These distances are appreciably less than the sum of the relevant van der Waals radii of 4.01 Å. If this bonding is considered to be significant then the coordination around the Sb atoms may alternatively be described as distorted octahedral with the equatorial positions occupied by Cl1, Cl2, O1 and O2 in both molecules; the axial positions around Sb1 are occupied by Cl3 and Cl2A^{i}, and atoms Cl3 and Cl2B^{ii} occupy the axial positions of Sb2. This arrangement results in an edge-sharing octahedral environment involving the Sb1 atoms at (x, y, z) and ($2-x$, $-y$, $2-z$), and the Sb2 atoms at (x, y, z) and ($2-x$, $1-y$, $1-z$).

The Sb—Cl bond lengths range from 2.3881 (9) to 2.5360 (10) Å; the shortest Sb—Cl distance is observed for the non-bridging Cl atom, as was also observed in related structures (Porter & Jacobson, 1970; Yamin *et*

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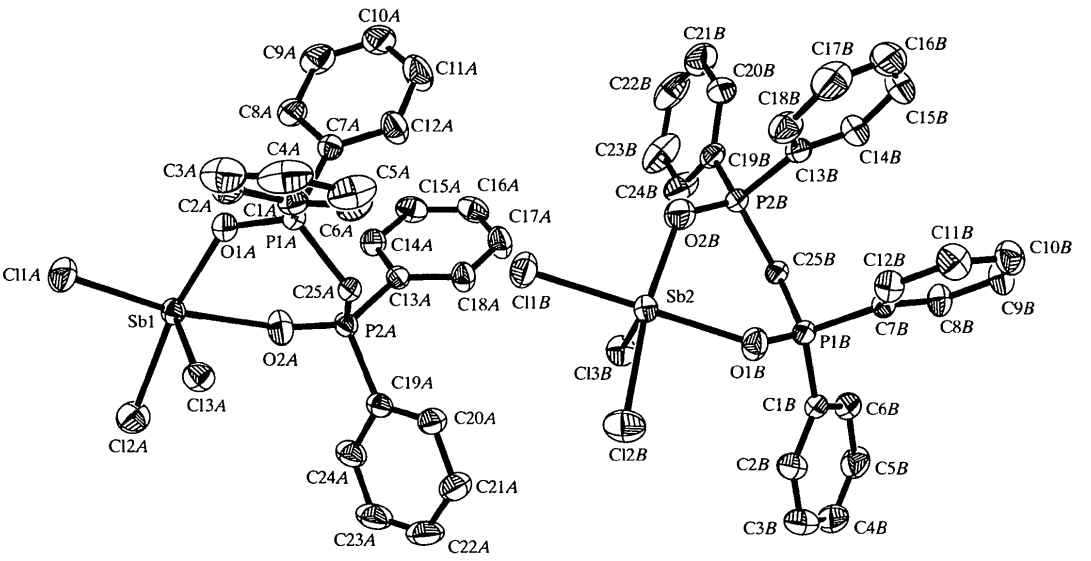

Fig. 1. The structure of title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

al., 1996). The mean P—C distances are comparable with those reported by Carmalt *et al.* (1996). The molecules in the asymmetric unit are linked by C—H···Cl hydrogen bonds involving atoms C25*A* and Cl1*B*, which lead to weak dimer formation; these dimers are linked to those translated along the *c* axis by C25*B*···Cl1*A*(x, y, $z-1$) hydrogen bonds to form infinite chains (Table 2).

Experimental

The title compound was prepared by adding 1,1′-bis(diphenylphosphino)methane to a solution of antimony pentachloride in acetonitrile. The resulting solution was allowed to stand for four days whereupon colourless crystals suitable for X-ray analysis were obtained.

Crystal data

$[SbCl_3(C_{25}H_{22}O_2P_2)]$
M_r = 644.47
Triclinic
$P\bar{1}$
a = 10.6849 (2) Å
b = 15.7703 (4) Å
c = 17.7065 (4) Å
α = 89.543 (1)°
β = 72.857 (1)°
γ = 71.607 (1)°
V = 2693.49 (10) Å^3
Z = 4
D_x = 1.589 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 6883 reflections
θ = 1.36–33.19°
μ = 1.461 mm^{-1}
T = 293 (2) K
Block
0.54 × 0.24 × 0.18 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
10 085 reflections with $I > 2\sigma(I)$
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
T_{min} = 0.568, T_{max} = 0.769
17 547 measured reflections
11 884 independent reflections
R_{int} = 0.014
θ_{max} = 27.5°
$h = -12 \rightarrow 13$
$k = -20 \rightarrow 20$
$l = 0 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.081
S = 1.095
11 884 reflections
595 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0193P)^2 + 3.2576P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.90 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sb1—O1*A*	2.282 (3)	Sb2—O1*B*	2.285 (3)
Sb1—O2*A*	2.368 (3)	Sb2—O2*B*	2.354 (3)
Sb1—Cl1*A*	2.4797 (11)	Sb2—Cl1*B*	2.5360 (10)
Sb1—Cl2*A*	2.5315 (11)	Sb2—Cl2*B*	2.4797 (11)
Sb1—Cl2*A*^{i}	3.6858 (14)	Sb2—Cl2*B*^{ii}	3.5450 (13)
Sb1—Cl3*A*	2.3881 (9)	Sb2—Cl3*B*	2.3996 (9)
P1*A*—O1*A*	1.488 (3)	P1*B*—O1*B*	1.494 (3)
P2*A*—O2*A*	1.476 (3)	P2*B*—O2*B*	1.459 (3)
O1*A*—Sb1—O2*A*	81.45 (10)	O1*B*—Sb2—O2*B*	83.05 (10)
O1*A*—Sb1—Cl3*A*	82.92 (8)	O1*B*—Sb2—Cl3*B*	86.23 (7)
O2*A*—Sb1—Cl3*A*	89.75 (8)	O2*B*—Sb2—Cl3*B*	83.35 (8)
O1*A*—Sb1—Cl1*A*	89.26 (7)	O1*B*—Sb2—Cl2*B*	87.49 (7)
O2*A*—Sb1—Cl1*A*	170.32 (8)	O2*B*—Sb2—Cl2*B*	168.82 (8)
Cl3*A*—Sb1—Cl1*A*	91.78 (4)	Cl3*B*—Sb2—Cl2*B*	90.12 (4)
O1*A*—Sb1—Cl2*A*	168.05 (7)	O1*B*—Sb2—Cl1*B*	176.52 (7)
O2*A*—Sb1—Cl2*A*	93.22 (8)	O2*B*—Sb2—Cl1*B*	95.48 (8)
Cl3*A*—Sb1—Cl2*A*	86.39 (4)	Cl3*B*—Sb2—Cl1*B*	90.47 (4)
Cl1*A*—Sb1—Cl2*A*	96.41 (4)	Cl2*B*—Sb2—Cl1*B*	93.63 (4)

O1A—Sb1—Cl2A^{i}	80.48 (7)	O1B—Sb2—Cl2B^{ii}	82.15 (7)
O2A—Sb1—Cl2A^{i}	73.30 (8)	O2B—Sb2—Cl2B^{ii}	91.34 (8)
Cl3A—Sb1—Cl2A^{i}	157.79 (3)	Cl3B—Sb2—Cl2B^{ii}	167.73 (3)
Cl1A—Sb1—Cl2A^{i}	102.62 (4)	Cl2B—Sb2—Cl2B^{ii}	93.26 (4)
Cl2A—Sb1—Cl2A^{i}	108.34 (3)	Cl1B—Sb2—Cl2B^{ii}	101.07 (3)
P1A—O1A—Sb1	134.07 (16)	P1B—O1B—Sb2	137.54 (16)
P2A—O2A—Sb1	133.82 (17)	P2B—O2B—Sb2	133.86 (18)

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

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C25A—H25B···Cl1B	0.97	2.66	3.608 (3)	163
C25B—H25D···Cl1A^{i}	0.97	2.60	3.559 (3)	168

Symmetry code: (i) $x,y,z-1$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections; it was found to be negligible. Although data were collected to a $2\theta_{max}$ of 66.3°, only reflections having 2θ less than 55° were used for structure solution and refinement. The structure was solved by direct methods and refined by full-matrix least squares. Although all H atoms were located from a difference Fourier map, they were geometrically fixed and allowed to ride on their parent C atoms since free refinement led to unrealistically short C—H distances.

Data collection: *SMART* (Siemens, 1996*b*). Cell refinement: *SAINT* (Siemens, 1996*a*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1263). Services for accessing these data are described at the back of the journal.

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A binuclear cadmium(II) complex: bis[bis(*N*,*N*-diisopropyldithiocarbamato)-cadmium(II)]

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Abstract

The title compound, bis[μ-(*N*,*N*-diisopropyldithiocarbamato)-*S*,*S*′:*S*][bis(*N*,*N*-diisopropyldithiocarbamato-*S*,*S*′)-cadmium(II)], $[Cd_2(C_7H_{14}NS_2)_4]$, a binuclear complex, consists of two bis(*N*,*N*-diisopropyldithiocarbamato)-cadmium complex units bridged by Cd—S bonds. Each Cd^{II} atom has a distorted tetragonal pyramidal environment and the inversion-related complex forms an edge-shared tetragonal pyramidal geometry with a Cd···Cd distance of 3.6049 (6) Å.

Comment

The development of effective antidotes for cadmium intoxication has proven to be a task of considerable difficulty. The reasons for this difficulty are quite numerous. The normal physiological processes by which cadmium is immobilized involve its bonding to an intracellular protein in the liver and the kidney (Cherian & Goyet, 1978). This metal–intracellular protein complex, Cd metallothionein, can destroy the kidney. The general goal of chelate therapy is usually the development of chelating agents which will reduce the body burden of a toxic metal by transforming it into a form in which it can be excreted in the urine. In recent years, it has become apparent that dithiocarbamates can mobilize cadmium

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