DIPHENYLPHOSPHINATES OF TRIBUTYLANTIMONY(V)

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Abstract
Reactions of $Bu^x_3SbBr_2$ with AgO_2PPh_2 in 1:2 stoichiometry afford $Bu^x_3Sb(O_2PPh_2)_2$ (**1**) [x = *n* (**1a**); *i* (**1b**)]. The reaction in 1:1 stoichiometry results in an equilibrating mixture of $Bu^x_3Sb(Br)_N(O_2PPh_2)_{2-N}$ (**2**) (N = 1, 2). The latter also forms immediately when (**1**) is treated with $Bu^x_3SbBr_2$. Treatment of $Bu^x_3Sb(OPr^i)_2$ with Ph_2PO_2H (1:1 ratio) followed by hydrolysis affords colourless crystals of $Bu^x_3Sb(OH)(O_2PPh_2)$ (**3**) [x = *n* (**3a**); *i* (**3b**)]. All these complexes have been characterized by elemental analysis, IR and NMR (^1H, ^{13}C, ^{31}P) spectroscopies, and in the case of **3b** by single crystal X-ray diffraction studies. The structure of **3b** features trigonal bipyramidal Sb with the axial positions occupied by an hydroxide-O atom and a phosphinate-O derived from a monodentate ligand. Zigzag chains of molecules, mediated by intermolecular -Sb-OH...O=P- interactions, are found in the crystal lattice.

Introduction
The chemistry of classical and organometallic main group metal complexes with oxy- and thio-phosphorous acids has attracted considerable attention during the last 15 years or so [1]. These complexes show diverse structural possibilities ranging from discrete monomeric structures to supramolecular assemblies. In this context, structural patterns of antimony complexes with phosphorous acids have also been reviewed recently by Silvestru and Haiduc [2]. This survey showed that often, phosphinic acid complexes are associated aggregates in the solid state. In pursuance of our recent work [3,4] and those of others [5] on such molecules, we report an investigation of some chemistry of tri-*n*- and *i*-butylantimony(V) complexes with diphenyl-phosphinic acid.

Results and Discussion
Treatment of $Bu^x_3SbBr_2$ with two equivalents of AgO_2PPh_2 in benzene solution readily afforded $Bu^x_3Sb(O_2PPh_2)_2$ (**1**) [x = *n* (**1a**); *i* (**1b**)]. When the reaction was carried out in 1:1 stoichiometry with the view to isolate $Bu^x_3Sb(Br)(O_2PPh_2)$ (**2**) [x = *n* (**2a**); *i* (**2b**)], an equilibrium between (**2**), (**1**) and $Bu^x_3SbBr_2$ (eq.1) was established instead:

$$Bu^x_3SbBr_2 + AgO_2PPh_2 \rightarrow Bu^x_3Sb(Br)(O_2PPh_2) \rightleftharpoons (\mathbf{1}) + Bu^x_3SbBr_2 \qquad \text{...(eq.1)}$$
$$\text{-AgBr}$$

Similar equilibria were established when $Bu^x_3SbBr_2$ were treated with Ph_2PO_2H in the presence of triethylamine. Treatment of a $CDCl_3$ solution of **1** with an equimolar solution of $Bu^x_3SbBr_2$ immediately established an equilibrium containing **2**, **1** and $Bu^x_3SbBr_2$ for each of x = *n* and *i*. The relative concentration of all three species (*ca* 3:2:2, respectively) did not change on keeping the solution either at room temperature for one week or refluxing the solution for one hour, as revealed by NMR spectroscopy. However, in the presence of excess of $Bu^x_3SbBr_2$ (4 equivalents or more) only **2** and $Bu^x_3SbBr_2$ existed in solution. Reaction of $Bu^x_3Sb(OPr^i)_2$ with Ph_2PO_2H in benzene solution followed by hydrolysis yields the hydroxo complexes $Bu^x_3Sb(OH)(O_2PPh_2)$ (**3**) [x = *n* (**3a**); *i* (**3b**)].

All of the complexes reported are colourless crystalline solids that are soluble in common organic solvents. The IR spectra of each of **1** - **3** exhibited a band in the region 429 ± 2 cm^{-1}, absent in the spectra of Ph_2PO_2H and $Bu^x_3SbBr_2$, which has been attributed to νSb-O [6]. $Bu^x_3SbBr_2$ and **2** showed absorptions due to νSb-Br at 270 ± 2 cm^{-1} [5]. The νSb-C could not be assigned unambiguously due to the presence of overlapping phenyl group absorptions. The spectra of **3a** and **3b** displayed a broad band at 3325 and 3175 cm^{-1}, respectively, which can be assigned to νO-H absorptions. ^1H NMR spectra exhibited the expected peak multiplicities and integrations. The butyl-H resonances for **1** are shielded relative to the corresponding signals for **2** and **3**. The $^{13}C\{^1H\}$ NMR spectra for **1** showed a single set of resonances for Bu^x_3Sb carbons and doublets for the diphenylphosphinate group due to $^nJ(^{31}P\text{-}^{13}C)$ couplings. The $^{31}P\{^1H\}$ spectra exhibited a single resonance and appeared at lower frequencies

when compared to that of uncoordinated diphenylphosphinic acid. The following trend of ^{31}P NMR signal deshielding is evident:

$$Ph_2PO_2H > Bu^x_3Sb(Br)(O_2PPh_2) \approx Bu^x_3Sb(O_2PPh_2)_2 > Bu^x_3Sb(OH)(O_2PPh_2)]$$

Weakly diffracting crystals were obtained for **3b** and one of these was subjected to a crystallographic study. The analysis is not optimal owing, in part, to disorder in the positions of the atoms, in particular for the light atom positions. However, the structure has been determined unambiguously. There are two independent molecules comprising the crystallographic asymmetric unit, labeled molecule *a* and *b*, that are similar to each other. The molecular structure for molecule *a* is illustrated in Fig. 1 and selected geometric parameters are collected in Table 1.

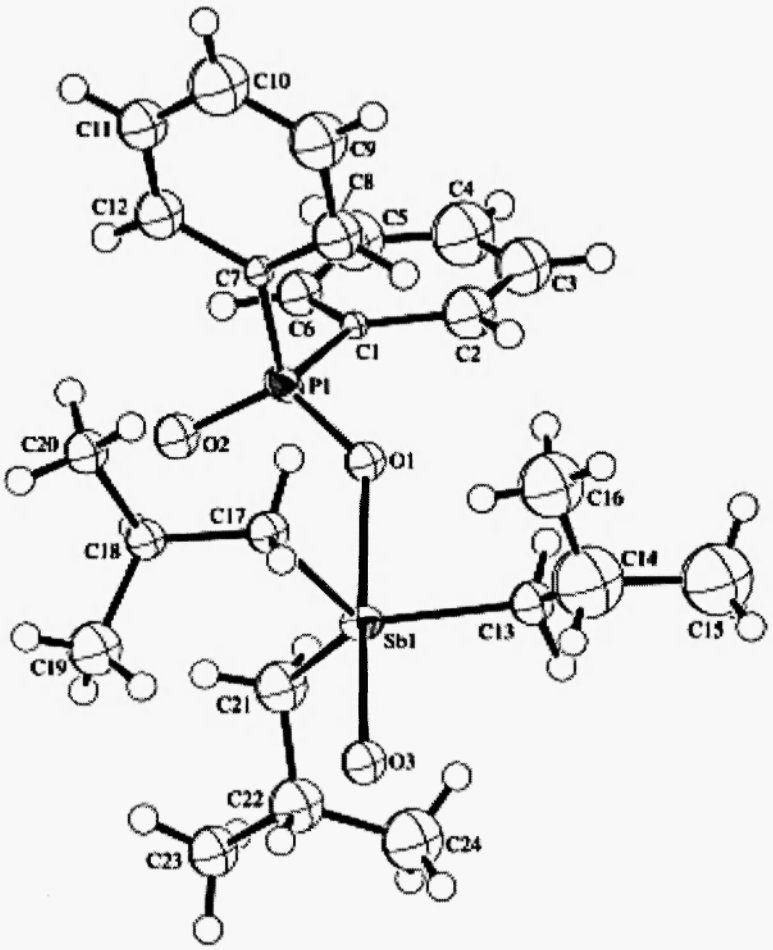

Fig. 1. Molecular structure and crystallographic numbering scheme for one of the independent molecules in $[Bu^t_3Sb(OH)(O_2PPh_2)]$ (**3b**)

In the following description parameters for the second molecule are given in parentheses; see Figs 1 and 2 for numbering schemes. The Sb atom in each molecule exists in a distorted trigonal bipyramidal geometry with the three organic substituents defining the equatorial plane. The axial positions are occupied by the hydroxide group and an O atom derived from a diphenylphosphinate ligand. The Sb(1) atom lies 0.233(2) Å (0.104(2) Å) out of the C_3 plane in the direction of the hydroxide group. The O-Sb-O axial angle is 178.6(6)° (176.4(6)°). The Sb-O-P distances are significantly longer than the Sb-OH distances, as expected. The diphenylphosphinate ligand coordinates in the monodentate mode as the secondary Sb-O2 distance of 3.94(1) Å (Sb-O5 is 3.91(1) Å) is longer than the sum of the van der Waals radii of 3.78 Å for Sb and O [7]. This feature of the structure may be

explained in terms of the participation of the O2 and O5 atoms in intermolecular associations to symmetry related hydroxide groups (see below).
The major differences in the Sb atom coordination geometries between the two independent molecules relate to the disposition of the *i*-butyl groups about the Sb atom as manifested in some of the O-Sb-C angles, which differ by up to 6°. In terms of the *i*-butyl groups themselves, there are some minor differences in the relative orientations of the methyl groups (the high thermal motion notwithstanding) with the major difference being associated with the relative orientations of the methyl groups in the C13- and C45- *i*-butyl groups. The crystal lattice of **3b** is stabilized by a series of intermolecular interactions leading to the formation of hydrogen-bonded aggregates.

Table 1. Selected geometric parameters (Å, °) for $[Bu^i_3Sb(OH)(O_2PPh_2)]$ (**3b**)

Sb-O1	2.27(2)	Sb2-O4	2.28(1)
Sb1-O3	1.97(2)	Sb2-O6	2.02(1)
Sb1-C13	2.11(3)	Sb2-C37	2.12(2)
Sb1-C17	2.12(2)	Sb2-C41	2.16(3)
Sb1-C21	2.11(3)	Sb2-C45	2.07(3)
P1-O1	1.54(1)	P2-O4	1.51(2)
P1-O2	1.52(2)	P2-O5	1.49(2)
O1-Sb1-O3	178.2(6)	O4-Sb2-O6	176.4(6)
O1-Sb1-C13	81.6(7)	O4-Sb2-C37	87.5(6)
O1-Sb1-C17	82.9(8)	O4-Sb2-C41	87.9(9)
O1-Sb1-C21	86.7(9)	O4-Sb2-C45	86.2(8)
O3-Sb1-C13	96.6(8)	O6-Sb2-C37	89.9(7)
O3-Sb1-C17	98.4(9)	O6-Sb2-C41	95.5(9)
O3-Sb1-C21	93.9(9)	O6-Sb2-C45	93.0(9)
C13-Sb1-C17	121.0(9)	C37-Sb2-C41	118(1)
C13-Sb1-C21	117(1)	C37-Sb2-C45	122(1)
C17-Sb1-C21	118(1)	C41-Sb2-C45	119(1)
Sb1-O1-P1	138.1(9)	Sb2-O4-P2	135.4(9)

The O2 atom is 2.66(2) Å from a symmetry related hydroxide-O3 (OH atoms were not located in the X-ray study; symmetry operation: 1-*x*, -0.5+*y*, 1.5-*z*). For the second independent molecule O5...O6 is 2.71(2) Å (symmetry operation: -*x*, -0.5+*y*, 1.5-*z*). These distances are indicative of significant hydrogen bonding interactions. This mode of association leads to the formation zigzag chains, mediated by -Sb-OH...O=P- hydrogen bonds, extending along the *b*-direction and as illustrated in Fig. 2.

Chains are comprised of linked molecules of one or the other of the independent molecules comprising the asymmetric unit. Links connecting the chains appear to be afforded by C-H...π interactions between centrosymmetrically and as well as between translationally related chains. Thus, for centrosymmetrically-related chains, the C48-H73...ring centroid of C1-C6 distance is 2.70 Å with an angle at H of 165° and the C48-H74...ring centroid of C7-C13 is 2.91 Å with an angle of 159° (symmetry operation –*x*, -*y*, 1-*z*). Between translationally-related chains (1+*x*, *y*, *z*), the C24-H36...ring centroid of C25-C30 distance is 2.88 Å with an angle at H of 168°.
The structure reported here bears a close resemblance to that of the trimethylantimony(V) analogue that has been reported recently [5].

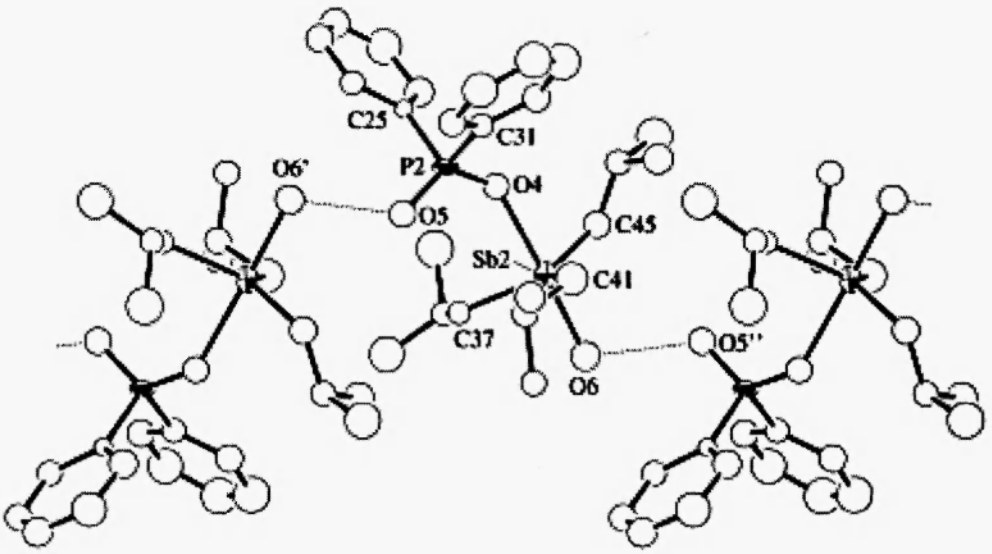

Fig. 2. Zigzag chains, mediated by -Sb-OH...O=P- hydrogen bonds, as found for the second independent molecule in $[Bu^t_3Sb(OH)(O_2PPh_2)]$ (**3b**). Symmetry operations: single prime *-x*, -0.5+*y*, 1.5-*z* and double prime -*x*, 0.5+*y*, 1.5-*z*

Experimental

Bu_3Sb and Bu^t_3Sb were prepared by the reaction of $SbCl_3$ with Bu^xMgBr in diethylether [7]. The dibromides, $Bu^x_3SbBr_2$, were obtained by the reaction of the tributylstibines in benzene with bromine in CCl_4. $Bu^n_3SbBr_2$, pale yellow oil (88% yield) Br% Found. 35.4; Calcd. 35.3. ^{1}H NMR in $CDCl_3$: 0.97 (t, 7.3 Hz, $SbCH_2CH_2CH_2$*Me*); 1.45 (sextet, 7.3Hz, $SbCH_2CH_2$*CH*$_2$-); 1.97 (quintet, 7.3 Hz, $SbCH_2$*CH*$_2$-); 2.87 (t, 8.1 Hz, Sb*CH*$_2$-); $Bu^t_3SbBr_2$, white crystalline solid (m.p. 90° C, lit 95 °C [8], 26% yield) Br% Found 35.1, Calcd. 35.3. ^{1}H NMR in $CDCl_3$: 1.14 (d, 6.6 Hz, $SbCH_2CH$*Me*$_2$), 2.57 (sextet, 6.6 Hz, $SbCH_2$*CH*<), 2.91 (d, 6.6 Hz Sb*CH*$_2$-). Diphenylphosphinic acid was prepared by the literature method ($^{31}P\{^1H\}$ in $CDCl_3$ δ: 33.2 ppm) [9]. The NMR spectra (^{1}H, $^{13}C\{^1H\}$, $^{31}P\{^1H\}$) were recorded on a Bruker DPX-300 NMR spectrometer in 5 mm tubes in $CDCl_3$ solution. The chemical shifts were referenced to the internal chloroform peak, δ 7.26 and 77.0, for ^{1}H and $^{13}C\{^1H\}$, respectively and external 85% H_3PO_4 for $^{31}P\{^1H\}$.

Preparations

$[Bu^t_3Sb(O_2PPh_2)_2]$ (**1a**)

To a benzene solution (20 ml) of $Bu^n_3SbBr_2$ (490 mg, 1.08 mmol), solid AgO_2PPh_2 (707 mg, 2.17 mmol) was added with stirring which was continued for 10 h at room temperature. Precipitated AgBr was filtered off and the filtrate was concentrated *in vacuo* to give a white solid in nearly quantitative yield (97%). This was recrystallized from a toluene-hexane mixture as a colourless crystalline solid, m.p. 78-80 °C. Analysis Found C, 59.6; H, 6.8; Calcd for $C_{36}H_{47}O_4P_2Sb$; C, 59.4; H, 6.5. ^{1}H NMR in $CDCl_3$ δ: 0.74 (t, 7.0 Hz, $SbCH_2CH_2CH_2$*Me*); 1.21 (sextet, 7.0 Hz, $SbCH_2CH_2$*CH*$_2$-); 1.80 (quintet, 7.0 Hz, $SbCH_2$*CH*$_2$-); 2.48 (t, 7.0 Hz, Sb*CH*$_2$-); 7.40 (m, 3,4,5-H, C_6H_5); 7.75 (m, 2,6-H, C_6H_5). ^{13}C NMR in $CDCl_3$ δ: 13.0 (s, $SbCH_2CH_2CH_2$*Me*); 25.0 (s, $SbCH_2CH_2$*CH*$_2$-); 26.1 (s, $SbCH_2$*CH*$_2$-); 33.6 (s, Sb*CH*$_2$); 128.0 (d, 12.7 Hz, C-3,5, C_6H_5); 130.6 (d, 1.9 Hz, C-4, C_6H_5); 131.0 (d, 9.6 Hz, C-2,6, C_6H_5); 137.4 (d, 135.0 Hz, C-1, C_6H_5); $^{31}P\{^1H\}$ in $CDCl_3$ δ: 21.5 ppm.

Similarly, $[Bu^t_3Sb(O_2PPh_2)_2]$ (**1b**) was prepared and recrystallized from a toluene-hexane solution in 66% yield as a colourless crystalline solid, m.p. 124-126 °C. Analysis Found C, 59.4; H, 6.2; Calcd for $C_{36}H_{47}O_4P_2Sb$; C, 59.4; H, 6.5. ^{1}H NMR in $CDCl_3$ δ: 0.92 (d, 6.6 Hz, $SbCH_2CH$*Me*$_2$); 2.36 (m, $SbCH_2$*CH*<); 2.54 (d, 7.1 Hz, Sb*CH*$_2$-); 7.32-7.42 (m, 3,4,5-H, C_6H_5); 7.69-7.76 (m, 2,6-H, C_6H_5); $^{13}C\{^1H\}$ NMR in $CDCl_3$ δ: 24.8 (s, $SbCH_2CH$*Me*$_2$); 25.2 (s, $SbCH_2$*CH*<); 44.8 (s, Sb*CH*$_2$-); 128.1 (d, 12.6 Hz, C-3,5, C_6H_5); 130.7 (d, 2.0 Hz, C-4, C_6H_5); 131.2 (d, 9.6 Hz, C-2,6, C_6H_5); 137.5 (d, 135.2 Hz, C-1, C_6H_5). $^{31}P\{^1H\}$ in $CDCl_3$ δ: 20.9 ppm.

$[Bu^t_3Sb(Br)(O_2PPh_2)]$ (**2b**)

(i) To a benzene solution of AgO_2PPh_2 (384 mg, 1.18 mmol) a solution of $Bu^t_3SbBr_2$ (536 mg, 1.18 mmol) was added with stirring which was continued at room temperature for 10 h. Precipitated AgBr was filtered off through a fine silica filter. The filtrate was concentrated *in vacuo* to give a white solid (684 mg, 98%) which was recrystallized from a benzene-hexane mixture, m.p. 81 °C. Analysis Found C, 48.9; H, 6.1; Calcd for $C_{24}H_{37}BrO_2PSb$; C, 48.8; H, 6.3. NMR spectra (^{1}H and ^{31}P) showed the existence of **1b**, **2b** and $Bu^t_3SbBr_2$ in an *ca* ratio of 2:3:2. ^{1}H NMR in $CDCl_3$ for **2b** δ: 1.02 (d, 6.6 Hz, $SbCH_2CH$*Me*$_2$); 2.44 (m, $SbCH_2$*CH*<); 2.72 (d, 7.0 Hz,Sb*CH*$_2$-); $^{31}P\{^1H\}$ NMR in $CDCl_3$ for **2b** δ: 21.0 ppm.

Similarly, $[Bu^n_3Sb(Br)(O_2PPh_2)]$ (**2a**) was prepared as a colourless oil. Analysis Found C, 48.0; H, 6.5. Calcd for $C_{24}H_{37}BrO_2PSb$; C, 48.8; H, 6.3. NMR spectra showed resonances for **1a**, **2a** and $Bu^n_3SbBr_2$ in 2:3:2 ratio. ^1H NMR in $CDCl_3$ for **2a** δ: 0.86 (t, 7.3 Hz, $SbCH_2CH_2CH_2\mathit{Me}$); 1.33 (sextet, 7.3 Hz, $SbCH_2CH_2\mathit{CH_2}$-); 1.87 (pentet, $SbCH_2\mathit{CH_2}$-); 2.66 (t, 7.3 Hz, $Sb\mathit{CH_2}$-). $^{31}P\{^1H\}$ in $CDCl_3$ for **2a** δ: 21.9 ppm.

(ii) To a $CDCl_3$ solution of **1b** (13 mg, 0.018 mmol), $Bu^t_3SbBr_2$ (8.1 mg, 0.018 mmol) was added in an NMR tube. ^1H and ^{31}P NMR spectra were recorded immediately after mixing which were identical with the spectra for the sample prepared in (i). The spectra did not change either on keeping the solution for one week at room temperature or refluxing it for one hour. However, on increasing the concentration of $Bu^t_3SbBr_2$ (4 mole and above) only **2b** and $Bu^t_3SbBr_2$ existed in solution.

Similarly, reaction between **1a** and $Bu^n_3SbBr_2$ investigated by ^1H and ^{31}P NMR. These showed spectra comparable to the product prepared in (i).

$[Bu^n_3Sb(OH)(O_2PPh_2)]$ (**3a**)

To **a** $NaOPr^i$ solution [prepared from 835 mg (36.3 mmol) Na in 25 ml Pr^iOH], a benzene solution (20 ml) of $Bu^n_3SbBr_2$ (8.23 g, 18.2 mmol) was added and stirred at room temperature for 4 h. Precipitated NaBr was filtered off and the filtrate was concentrated *in vacuo* and the residue was distilled under vacuum (125-130 °C/5 mm) to give $[Bu^n_3Sb(OPr^i)_2]$ as a colourless liquid (3.57g, 48%).

To a benzene solution of $[Bu^n_3Sb(OPr^i)_2]$ (1.05 g, 2.55 mmol), solid Ph_2PO_2H (492 mg, 2.25 mmol) was added and stirred at room temperature for 30 min and then solvent was evaporated *in vacuo* to give a paste which on crystallization from moist acetone-hexane solution gave colourless crystals of $[Bu^n_3Sb(OH)(O_2PPh_2)]$ (740 mg, 87%), m.p. 90 °C. Analysis Found C, 54.0; H, 7.2; Calcd for $C_{24}H_{38}O_3PSb$; C, 54.6; H, 7.2. ^1H NMR in $CDCl_3$ δ: 0.87 (t, 7.3 Hz, $SbCH_2CH_2CH_2\mathit{Me}$); 1.35 (sextet, 7.3 Hz, $SbCH_2CH_2\mathit{CH_2}$-); 1.75 (pentet, 7.3 Hz, $SbCH_2\mathit{CH_2}$-); 2.32 (t, 8.0 Hz, $Sb\mathit{CH_2}$-); 7.35 (br, 3,4,5-H, C_6H_5); 7.70-7.76 (m, 2,6-H, C_6H_5). $^{31}P\{^1H\}$ in $CDCl_3$ δ: 18.2 ppm.

Similarly, $[Bu^t_3Sb(OH)(O_2PPh_2)]$ (**3b**) was prepared and recrystallised from toluene, m.p. 126 °C. Analysis Found C, 53.9; H, 7.2; Calcd for $C_{24}H_{38}O_3PSb$; C, 54.6; H, 7.2. ^1H in $CDCl_3$ δ: 1.02 (d, 6.6 Hz, $SbCH_2CH\mathit{Me_2}$); 2.28-2.33 (overlapping signals due to $Sb\mathit{CH_2}$- and $SbCH_2\mathit{CH}<$); 7.32-7.36 (m, 3,4,5-H, C_6H_5); 7.70-7.77 (m, 2,6-H, C_6H_5). $^{31}P\{^1H\}$ in $CDCl_3$ δ: 17.6 ppm.

Crystallography

Crystallographic data are summarized in Table 2. A correction was applied to the data to allow for significant decomposition (*ca* 11 %) of the crystal during the course of the data collection. Despite the data collection having been measured at 173 K, significant thermal motion was found in each of the two molecules comprising the crystallographic asymmetric unit. As a consequence only the Sb and P atoms were refined with anisotropic displacement parameters. H atoms were included in their calculated positions; the O-H atoms were not located.

Table 2. Crystal data for $[Bu^t_3Sb(OH)(O_2PPh_2)]$ (**3b**)

Formula	$C_{24}H_{38}O_3PSb$	Formula weight	527.3
Crystal system	monoclinic	Crystal size, mm	0.08 x 0.16 x 0.21
Space group	$P2_1/c$	*a*, Å	22.043(2)
b, Å	13.2276(7)	*c*, Å	18.600(2)
β, °	110.75(1)	*V*, Å^3	5071.3(2)
Z	8	Diffractometer	Rigaku AFC7R
Temperature, K	173	μ(Mo-Kα), cm^{-1}	11.70
Trans. factors	0.561 – 1	D_{calcd}, g cm^{-3}	1.381
F(000)	2176	θ_{max}, °	27.5
Reflns meas.	12517	Reflns unique	12156
Reflns with $I > 3.0\sigma(I)$	3136	Weighting scheme	$1/[\sigma^2(F_o) + 0.00002F_o^2]$
$R(F)$, $R_w(F)$	0.064, 0.075	ρ, e A^{-3}	1.02
Programs used	DIFABS [11], teXsan [12], DIRDIF [13], ORTEP [14], PLATON [15]		
Deposition number	CCDC 145932		

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