Trimethylantimony(V) diorganomonothiophosphinates: the crystal structure of bis(diphenylmonothiophosphinato)trimethylantimony(V), $Me_3Sb(OSPPh_2)_2$, containing a monodentate monothiophosphinato ligand

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Abstract

The compounds bis(diorganomonothiophosphinato)trimethylantimony(V), $Me_3Sb(OSPR_2)_2$, where R = Me, Et and Ph, have been prepared and characterized by IR, multinuclear (^1H, ^{13}C, ^{31}P) NMR spectroscopy and mass spectrometry. The structure of $Me_3Sb(OSPPh_2)_2$ has been determined by single-crystal X-ray diffraction. The crystal consists of discrete monomers. In the molecule there is a trigonal bipyramidal environment around antimony, with the two monothio ligands bound to the metal only through oxygen atoms (Sb–O(1), 2.107(5) Å; Sb–O(2), 2.114(5) Å. The antimony–sulphur distances, *i.e.* 3.924 and 3.998 Å, are consistent with non-bonding van der Waals contacts (*ca.* 4.0 Å). The average bond distances within the phosphorus ligand are P–O = 1.552, P=S = 1.945 and P–C = 1.808 Å. The SbC₃ unit is planar, and the angle between the axial Sb–O bonds is 177.7(2)°. Coordination of the monothiophosphinato ligand in monodentate mode is unusual; such ligands usually exhibit chelate or bridging coordination through both oxygen and sulphur atoms.

Key words: Antimony; Monothiophosphinate; X-ray diffraction; Crystal structure

1. Introduction

In the context of our interest in the structural chemistry of antimony, we have previously studied both inorganic and diphenylantimony(III) compounds containing diorganodithiophosphorus ligands [1–4]. In addition to providing further evidence of the broad diversity of coordination patterns of the dithiophosphorus

moiety, these studies have shown that antimony can exhibit various coordination modes. Thus in $Sb[S_2P(OR)_2]_3$ (R = Me and ^iPr) [1] and $Sb(S_2PPh_2)_3$ [2], although the dithio ligands are anisobidentate, they give rise to different coordination geometries around the metal atom, namely distorted octahedral in the dithiophosphato derivatives and pentagonal pyramidal in the diphenyldithiophosphinate. Marked variations were also observed for diphenylantimony(III) derivatives, where the dithio ligand can both chelate and bridge, leading to a dimeric structure in $Ph_2SbS_2PPh_2$ [3] or a chain polymeric structure in $Ph_2SbS_2P(O^iPr)_2$ [4]. To investigate any structural changes which might

result from replacement of one sulphur of the phosphorus ligand by oxygen, we have extended our studies to antimony derivatives of monothiophosphinato ligands. In theory, at least, this type of ligand can bond primarily to the metal through the oxygen atom (a) or the sulphur atom (b).

a   b
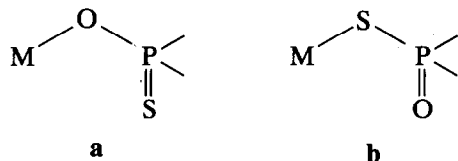
So far, only the former has been confirmed by X-ray diffraction studies.

We previously reported the results of a structural investigation of $Ph_2SbOSPPh_2$, which was found to have a polymeric chain structure, with bridging diphenylmonothiophosphinato groups and pseudotrigonal bipyramidal coordination around antimony, and phenyl groups in equatorial positions [5]. In contrast, Mattes and Rühl [6] reported monomeric distorted octahedral structures for $Sb(OSPR_2)_3$ (R = Cy, Ph). In both of these inorganic and organoantimony(III) monothiophosphinates, the ligands are primarily bonded through oxygen, with secondary antimony-sulphur interactions.

There are very few studies of analogous organoantimony(V) derivatives containing thiophosphorus ligands. In the dithiophosphorus ligand series, Mössbauer data are available for $Me_3Sb(S_2PEt_2)_2$ [7], while in the case of monothiophosphinato ligands an X-ray structure is available for $Me_4SbOSPMe_2$ [8].

We report here the synthesis and structural characterization of bis(diorganomonothiophosphinato)trimethylantimony(V), $Me_3Sb(OSPR_2)_2$ (R = Me, Et, Ph).

2. Results and discussion

The trimethylantimony(V) monothiophosphinates were made by treating trimethylantimony(V) dichloride in benzene with a two molar proportion of the sodium or ammonium salt of the appropriate diorganomonothiophosphinic acid
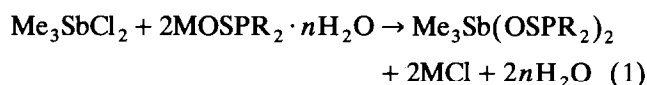
$$Me_3SbCl_2 + 2MOSPR_2 \cdot nH_2O \rightarrow Me_3Sb(OSPR_2)_2$$
$$+ 2MCl + 2nH_2O \quad (1)$$

(R = Me, M = Na, n = 1.5; R = Et, M = Na, n = 3; R = Ph, M = NH$_4$, n = 0).

The new compounds are pale yellow (R = Et) or colourless (R = Me, Ph) crystalline solids, soluble in various organic solvents (e.g. benzene, chloroform), and can be recrystallized without decomposition (Table 1). Although they are reasonably stable in air, if kept for long periods they should be stored under nitrogen.

All attempts to obtain the analogous triphenylantimony(V) derivatives from Ph_3SbCl_2 failed, the metal being reduced to the +3 oxidation state. The stabilization of the +5 oxidation state in the trimethylantimony(V) monothiophosphinates seems to be due to the presence of electron donating methyl groups on the metal.

2.1. IR spectra

Important IR bands for the three new compounds are listed in Table 2; the assignments were made by comparison with those of the starting materials and data reported in the literature for some O- and S-esters of the monothiophosphinic acids.

The compounds showed no strong absorptions in the regions characteristic of either single P–S bonds (550–450 cm^{-1} and 600–550 cm^{-1} for dialkyl- and

TABLE 2. IR spectral data [a]

Compound	ν(P–O)	ν(P=S)	ν(Sb–C)	ρ[(Sb)CH$_3$]
$Me_3Sb(OSPMe_2)_2$	1011vs	588s	[b]	859s
	994vs			
$Me_3Sb(OSPEt_2)_2$	1046s	579m	[b]	854m-s
	1004s			
	997vs			
	977vs			
$Me_3Sb(OSPPh_2)_2$	982vs	647vs	578w	860m-s

[a] In Nujol mulls.
[b] Obscured by the strong P–S stretching band.

TABLE 1. Analytical data and physical properties of $Me_3Sb(OSPR_2)_2$ derivatives

Compound	Formula	%C found	%H found	Yield (%)	m.p. (°C)	Recryst. solvent	Soluble in
$Me_3Sb(OSPMe_2)_2$	$C_7H_{21}O_2P_2S_2Sb$ (FW 384.7)	21.52 (21.84)	5.23 (5.46)	86	148–150	MeCN–C_6H_6	$CHCl_3$
$Me_3Sb(OSPEt_2)_2$	$C_{11}H_{29}O_2P_2S_2Sb$ (FW 440.7)	30.08 (29.95)	6.96 (6.58)	73	83–85	$CHCl_3$–hexane	C_6H_6 $CHCl_3$
$Me_3Sb(OSPPh_2)_2$	$C_{27}H_{29}O_2P_2S_2Sb$ (FW 632.7)	51.29 (51.21)	4.66 (4.58)	87	168–170	MeCN	C_6H_6, $CHCl_3$

diphenylmonothiophosphinic acid derivatives respectively) or double P=O bonds (1250–1150 cm^{-1}), e.g.

$\nu(\text{P-S})$	$\nu(\text{P=O})$	
510 cm^{-1}	1184 cm^{-1}	in Me$_2$P(=O)SMe [9]
520 cm^{-1}	1180 cm^{-1}	in Et$_2$P(=O)S^{n}Pr [10]
565 cm^{-1}	1202 cm^{-1}	in Ph$_2$P(=O)SMe [11]

Therefore, coordination is most likely to occur through the oxygen atom of the ligand; this is a common feature of the organometallic monothiophosphinates investigated thus far by X-ray diffraction, i.e. Ph$_2$-SbOSPPh$_2$ [5], Ph$_2$Sn(OSPPh$_2$)$_2$ [12], Ph$_2$Sn(OSPEt$_2$)$_2$ [13] and Me$_3$SnOSPMe$_2$ [14], as well as the inorganic Main Group metal derivatives, e.g. Sb(OSPCy$_2$)$_3$ and Sb(OSPPh$_2$)$_3$ [6]. It is, however, possible for the sulphur atom doubly bonded to phosphorus to be involved in secondary bonding to the metal atom. This was suggested to be the case in Me$_4$SbO(S)PMe$_2$, which on the basis of Mössbauer data was assigned a dimeric structure with bridging ligands [15]. However, an X-ray study [8] revealed that the monothiophosphinato ligand acts in monodentate mode, bound to the metal through oxygen. (The non-bonding antimony–sulphur distance calculated from the original data is 4.19 Å.) On the other hand, it is difficult to exclude weak antimony–sulphur interactions in the case of Me$_3$Sb(OSPR$_2$)$_2$ derivatives on the basis of IR data only. Moreover, an X-ray diffraction investigation of a dithiocarbamato derivative containing the same organometallic unit, i.e. Me$_3$Sb(S$_2$CNMe$_2$)$_2$ [16], has revealed the ability of the doubly bonded sulphur atom to coordinate to antimony, thus leading to anisobidentate behaviour.

TABLE 3. ^{1}H NMR data ^{a,b}

R Chemical shifts (ppm) and coupling constants (Hz)	Origin
Me 1.8ds (12H), $^2J(^{31}P^1H) = 12.8$	P–CH$_3$
2.17s (9H)	Sb–CH$_3$
Et 1.07dt (12H), $^3J(^{31}PCC^1H) = 19.3$, $^3J(^1HCC^1H) = 7.5$	P–CH$_2$CH$_3$
1.73dq (8H), $^2J(^{31}PC^1H) = 12.2$, $^3J(^1HCC^1H) = 7.5$	P–CH$_2$CH$_3$
2.13s (9H)	Sb–CH$_3$
Ph 2.16s (9H)	Sb–CH$_3$
7.35s,br (12H, meta + para), 7.74dm (8H, ortho),	P–C$_6$H$_5$
$^3J(^{31}PCC^1H) = 6.1$	

^{a} Chemical shifts are reported relative to tetramethylsilane (TMS); abbreviations used: s, singlet; dt, doublet of triplets; dq, doublet of quartets; dm, doublet of multiplets; br, broad.
^{b} In CHCl$_3$.

Each of the three compounds exhibits a medium-strong absorption at approximately 860–850 cm^{-1}, assigned to the C–H rocking vibration of the Me$_3$Sb fragment and, additionally, one weak band at 579 cm^{-1} was observed for R = Ph. This was assigned to the asymmetric antimony–carbon stretching vibration, pointing to a highly symmetrical (D_{3h} point group) planar arrangement of the SbC$_3$ moiety. For the other compounds (R = Me, Et), this band is obscured by the strong phosphorus–sulphur stretching vibrations, since if a D_{3h} symmetry is envisaged in the C$_3$Sb skeleton, one should expect absorptions at ca. 568 and 523 cm^{-1} [17].

2.2. NMR spectra

The ^{1}H, ^{13}C and ^{31}P NMR spectral data for all three compounds are summarized in Tables 3 and 4. The proton and carbon resonance data confirm the identity of the products and provide further evidence for the

TABLE 4. ^{13}C and ^{31}P{^{1}H} NMR data ^{a}

R	^{13}C chemical shifts (ppm) and coupling constants (Hz) ^{b}					$\delta(^{31}P)$ ^{c}
	Sb–CH$_3$	P–CH$_3$	P–CH$_2$CH$_3$	P–CH$_2$CH$_3$	P–C$_6$H$_5$ ^{d}	
Me	16.59s	26.84d ^1J(PC) = 73.3	–	–	–	75.4
Et	16.44s	–	29.3d ^1J(PC) = 71.7	7.16s	–	52.6
Ph	16.44s	–	–	–	C$_i$: 139.1d ^1J(PC) = 107.9 C$_o$: 130.0d ^2J(PCC) = 10.8 C$_m$: 128.2d ^3J(PCCC) = 13.2 C$_p$: 130.8s	75.3

^{a} In CDCl$_3$ solution; abbreviations used: s, singlet; d, doublet.
^{b} Standard CDCl$_3$.
^{c} Standard H$_3$PO$_4$ 85%.
^{d} The assignments are not unambiguous.

TABLE 5. FAB monoisotopic mass spectral data for $Me_3Sb(OSPR_2)_2$ (m/e, % intensity)

Ion	R = Me	R = Et	R = Ph
$Me_3Sb(OSPR_2)_2^{+}$	384 (0.8)(?)	440 (0.5)(?)	–
$Me_2Sb(OSPR_2)_2^{+}$	369 (1)	425 (9)	384 (3)
$Sb(OSPR_2)_2^{+}$	339 (0.3)	395 (4)	–
$Me_2SbO(OSPR_2)^{+}$	276 (6)	304 (70)	400 (30)
$Me_3Sb(OSPR_2)^{+}$	275 (100)	303 (100)	399 (100)
$MeSbO(OSPR_2)^{+}$	261 (5)	289 (13)	385 (12)
$SbO(OSPR_2)^{+}$	246 (1)	274 (8)	370 (3)
$MeSb(OSPR_2)^{+}$	245 (6)	273 (46)	369 (12)
SbO_2^{+} (or SbS^{+})	153 (2)	153 (4)	153 (4)
$SbMe_2^{+}$	151 (2)	151 (15)	151 (5)
Sb^{+}	121 (1)	121 (9)	121 (4)

equivalence of the three methyl groups attached to antimony (single resonances at ca. 2.1 ppm in the ^1H NMR and at 16.4 ppm in the ^{13}C NMR spectra). The signals from the organic groups bound to phosphorus show the expected features, including splitting due to proton–phosphorus coupling, and their equivalence is also demonstrated.

In all cases the $^{31}P\{^1H\}$ NMR spectra show only one resonance, consistent with the equivalence of the two phosphorus atoms in the $Me_3Sb(OSPR_2)_2$ derivatives.

2.3. Mass spectra

The FAB monoisotopic mass spectral data for $Me_3Sb(OSPR_2)_2$ are listed in Table 5, which is self-explanatory, and a tentative fragmentation pattern is given in Fig. 1. The ion of the first fragmentation, i.e. $Me_3Sb(OSPR_2)^{+}$, resulting from cleavage of an antimony–oxygen bond, is always the base peak. In addition, the possibility of phosphorus–oxygen bond cleavage, i.e. the loss of an R_2PS fragment (not shown in Table 5), is supported by the presence of fragments of

m/e 183 (R = Me, 3%; R = Et, 10%; R = Ph, 4%) and 199 (R = Et, 13%), which can be assigned to $Me_2SbO_2^{+}$ (but also to Me_2SbS^{+}) and Me_2SbSO^{+} respectively.

A fragment ion of m/e 332 (42% of the base peak), observed in the spectrum of $Me_3Sb(OSPEt_2)_2$, is tentatively assigned to $Et_2SbO(OSPEt_2)^{+}$, suggesting that a rearrangement of the organic groups from phosphorus to antimony can take place during the fragmentation. A similar process was observed for organotin dithiophosphinates [18].

Other unexpected ions, involving a phosphorus–sulphur bond cleavage, i.e. $MeSbS(OSPR_2)^{+}$ (R = Et, m/e 305, 39%; R = Ph, m/e 401, 11%) and R_2PO (R = Me, m/e 77, 6%; R = Et, m/e 105, 38%; R = Ph, m/e 201, 45%) are also observed. This behaviour might be accounted for either in terms of an anisobidentate coordination of the thiophosphinato ligand, or a rearrangement of the primary bond during the fragmentation process.

2.4. Molecular structure of $Me_3Sb(OSPPh_2)_2$

Important bond distances and angles for bis(diphenylmonothiophosphinato)trimethylantimony(V), $Me_3Sb(OSPPh_2)_2$, are listed in Table 6, and the molecular structure of the compound with the atom numbering scheme is illustrated in Fig. 2.

The crystal consists of discrete monomeric $Me_3Sb(OSPPh_2)_2$ units with both monothiophosphinato ligands bound to the metal atom through oxygen (Sb–O(1), 2.114(5) Å; Sb–O(2), 2.107(5) Å). The geometry at phosphorus is distorted tetrahedral, with angles at phosphorus very close to those observed in the free acid, $Ph_2P(S)OH$ [19]. The distortion is due to the S–P–O angle, which is larger than the usual 109° (average S–P–O angles are 117.5° in the complex and
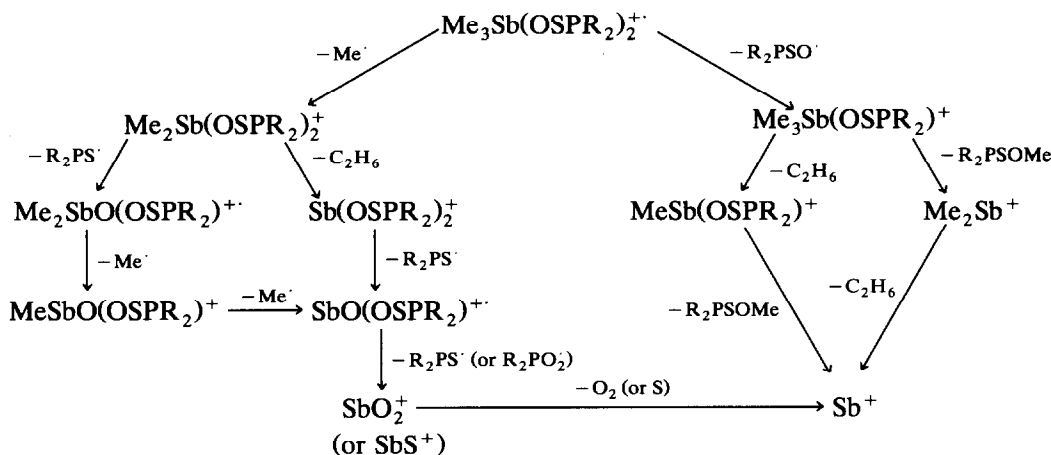
Fig. 1. Possible fragmentation pattern for $Me_3Sb(OSPR_2)_2$.

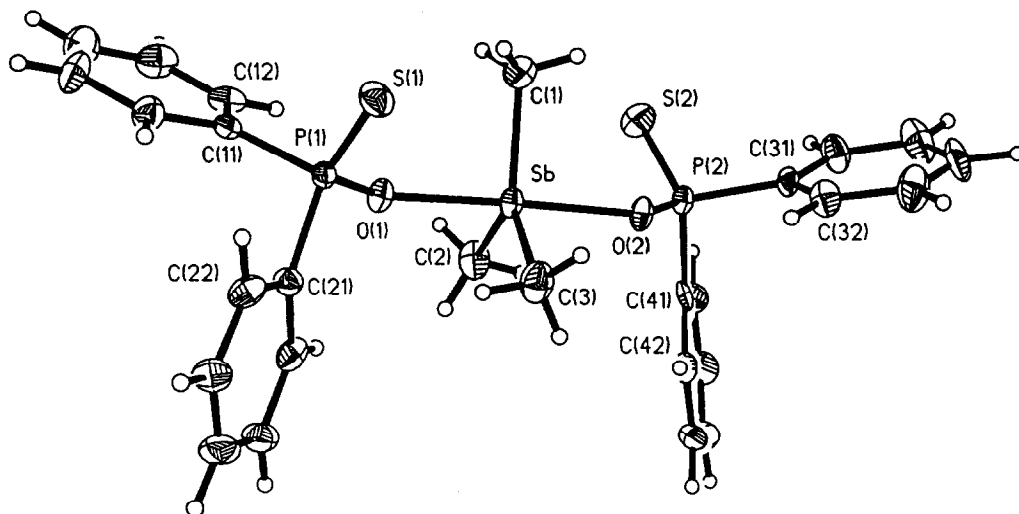Fig. 2. ORTEP drawing of the monomeric structure of $Me_3Sb(OSPPh_2)_2$.

115.4° in the free acid. The average phosphorus–sulphur and phosphorus–oxygen distances are 1.945 and 1.552 Å respectively (*cf.* P=S 1.956 Å, P–O 1.582 Å, in $Ph_2P(S)OH$ [19]), corresponding to typical double P=S and single P–O bonds in the monothiophosphinato unit.

The coordination environment around antimony can be described as a slightly distorted trigonal bipyramid, with equatorial Sb–C bonds and axial oxygen atoms (O(1)–Sb–O(2) angle of 177.7(2)°). The SbC_3 unit is planar (the sum of the C–Sb–C angles is 360°) and the O–Sb–C angles lie in the range 85.7°–93.7°. It will be seen that the bond angles involving sulphur are opened at the expense of those involving the oxygen atoms.

The Sb–O bond lengths, 2.114(5) and 2.107(5) Å, fall in the range observed for antimony(III) complexes containing chelating (in $Sb(OSPPh_2)_3$ [6]) and bridging (in $Ph_2SbOSPPh_2$ [5]) diphenylmonothiophosphinato groups (Table 7). The Sb–O–P angles are larger than in the chelate $Sb(OSPPh_2)_3$ complex and, as a result, the antimony–sulphur distances are very close to the sum of the van der Waals' radii (*ca.* 4.0 Å), indicating that the sulphur atoms are not involved in coordination to the metal. Thus the monothiophosphinato groups act in monodentate mode, which is quite unusual for such ligands. By contrast, in the related dithiocarbamato complex, $Me_3Sb(S_2CNMe_2)_2$, both sulphur atoms of a ligand unit are involved in coordination to antimony (Sb–S, 2.575, 2.614 Å; Sb...S, 3.274 and 3.315 Å as semibonding interactions) [16].

3. Experimental details

3.1. Materials

The starting materials were made by published procedures: Me_3SbCl_2 [20], $NaOSPMe_2 \cdot 1.5H_2O$,

$NaOSPEt_2 \cdot 2H_2O$ [21] and $Ph_2P(S)OH$ [22]. The ammonium salt of diphenylmonothiophosphinic acid was obtained by bubbling dry ammonia gas into the benzene solution of the free acid. IR spectra were recorded on a Perkin–Elmer 983 instrument, as Nujol mulls. NMR spectra were obtained using a Bruker WM250 spectrometer operating at 250 MHz. Mass spectra were obtained using a VG 70E instrument with a FAB attachment. Elemental analyses (carbon and hydrogen) were performed on a Perkin–Elmer 240B analyser.

TABLE 6. Important bond distances (Å) and angles (°) in $Me_3Sb(OSPPh_2)_2$

Bond distance		Bond angle	
Sb–C(1)	2.085(8)	C(1)–Sb–C(2)	119.8(4)
Sb–C(2)	2.095(8)	C(2)–Sb–C(3)	116.3(4)
Sb–C(3)	2.071(9)	C(3)–Sb–C(1)	123.9(3)
Sb–O(1)	2.114(5)	C(1)–Sb–O(1)	90.5(3)
Sb–O(2)	2.107(5)	C(2)–Sb–O(1)	85.7(3)
		C(3)–Sb–O(1)	91.5(3)
P(1)–O(1)	1.545(6)	C(1)–Sb–O(2)	91.8(3)
P(1)–S(1)	1.958(3)	C(2)–Sb–O(2)	93.7(3)
P(1)–C(11)	1.799(7)	C(3)–Sb–O(2)	86.8(3)
P(1)–C(21)	1.808(8)	O(1)–Sb–O(2)	177.7(2)
P(2)–O(2)	1.558(6)	S(1)–P(1)–O(1)	117.6(2)
P(2)–S(2)	1.932(3)	S(1)–P(1)–C(11)	112.1(3)
P(2)–C(31)	1.813(7)	S(1)–P(1)–C(21)	111.3(3)
P(2)–C(41)	1.813(8)	O(1)–P(1)–C(11)	103.7(3)
		O(1)–P(1)–C(21)	106.1(3)
		C(11)–P(1)–C(21)	105.1(3)
		Sb–O(1)–P(1)	134.3(3)
		S(2)–P(2)–O(2)	117.4(2)
		S(2)–P(2)–C(31)	112.4(3)
		S(2)–P(2)–C(41)	112.2(3)
		O(2)–P(2)–C(31)	105.0(3)
		O(2)–P(2)–C(41)	105.7(3)
		C(31)–P(2)–C(41)	102.9(3)
		Sb–O(2)–P(2)	130.4(3)

TABLE 7. Comparison of relevant bond lengths (Å) and angles (°) in $Me_3Sb(OSPPh_2)_2$ and related compounds

Compound	P=S	P–O	S=P–O angle	Sb–O	Sb···S	X–O–P angle	Reference
$Ph_2P(S)OH$	1.956	1.582	115.4	–	–	122	17
$Ph_2SbOSPPh_2$	2.010	1.504	116.5	2.299	2.753 [a]	138.7	5
$Sb(OSPPh_2)_3$	1.953	1.563	111.3	2.063	3.223	117.2	6
	1.973	1.556	115.1	2.037	3.001	124.2	
	1.956	1.568	111.4	2.061	3.164	113.6	
$Me_3Sb(OSPPh_2)_2$	1.958	1.545	117.6	2.114	3.998	134.3	This
	1.932	1.558	117.4	2.107	3.924	130.4	work

[a] Distance to a symmetry-related antimony atom.

3.2. Preparation of bis(dimethylmonothiophosphinato)trimethylantimony(V)

A mixture of trimethylantimony(V) dichloride (0.54 g, 2.5 mmol) and $NaOSPMe_2 \cdot 1.5H_2O$ (0.8 g, 5 mmol) in 50 ml of anhydrous benzene was stirred under reflux for 2 h. Initially, the mixture became clear, and then a small amount of a white crystalline precipitate appeared. The solution was filtered hot, and allowed to cool to room temperature. The product separated as white crystals. Yield, 0.83 g (86%); m.p. 148–150°C. The compound can be recrystallized from benzene or a benzene–acetonitrile mixture.

3.3. Preparation of bis(diethylmonothiophosphinato)trimethylantimony(V)

A mixture of 0.59 g (2.5 mmol) of Me_3SbCl_2 and 1.07 g (5 mmol) of $NaOSPEt_2 \cdot 3H_2O$ in 25 ml of anhydrous benzene was stirred under reflux for 2 h, and then the NaCl formed was filtered off. The clear filtrate was evaporated in vacuum and the yellowish crystals obtained were recrystallized from chloroform–hexane. Yield, 0.81 g (73%); m.p. 83–85°C.

3.4. Preparation of bis(diphenylmonothiophosphinato)trimethylantimony(V)

A mixture of 0.48 g (2 mmol) of Me_3SbCl_2 and 1.0 g (4 mmol) of NH_4OSPPh_2 in 25 ml of anhydrous benzene was stirred at room temperature for 4 h. The ammonium chloride formed was filtered off and the clear filtrate was evaporated in vacuum. The resulting white solid was recrystallized from acetonitrile. Yield, 1.1 g (87%); m.p. 168–170°C.

3.5. Crystal structure determination

3.5.1. Crystal data

$C_{27}H_{29}O_2P_2S_2Sb$, M 636.5, orthorhombic, $a =$ 11.885(2), $b = 8.657(2)$, $c = 27.493(5)$ Å, $V = 2828.6(9)$ Å^3, $Z = 4$, $D_c = 1.495$ g cm^{-3}, $F(000) = 1286$, space group $Pna2_1$, Mo Kα radiation, $\lambda = 0.71073$ Å, μ(Mo Kα) 1.259 mm^{-1}, crystal size 0.5 × 0.5 × 0.2 mm^3.

3.5.2. Structure determination

Data were collected on a Siemens P4 four-circle diffractometer for 7097 reflections in the 2Θ–ω mode, of which 3706 were independent reflections ($R_{int} =$ 1.86%) and 3056 ($F > 4.0\sigma(F)$) were used in the full

TABLE 8. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients (10^{-1} pm^2)

	x	y	z	U_{eq} [a]
Sb	5206.0(3)	4395.7(5)	7500	18.9(1)
C(1)	5694 (7)	2089 (10)	7446 (4)	31 (2)
C(2)	3545 (6)	5025 (12)	7341 (3)	32 (3)
C(3)	6291 (7)	6179 (12)	7679 (3)	33 (3)
S(1)	7971 (2)	3938 (3)	6684.6(9)	29.2(6)
P(1)	6587 (2)	4877 (2)	6436.7(7)	16.6(5)
O(1)	5513 (5)	4717 (7)	6749 (2)	23 (2)
C(11)	6180 (6)	4087 (9)	5858 (3)	18 (2)
C(12)	5227 (8)	3209 (10)	5795 (4)	26 (3)
C(13)	4978 (7)	2548 (12)	5354 (3)	35 (3)
C(14)	5697 (9)	2774 (13)	4963 (4)	42 (4)
C(15)	6645 (9)	3662 (12)	5009 (3)	39 (3)
C(16)	6870 (7)	4320 (11)	5452 (3)	30 (3)
C(21)	6768 (6)	6922 (9)	6330 (3)	20 (2)
C(22)	7799 (7)	7500 (11)	6147 (3)	25 (3)
C(23)	7926 (7)	9007 (10)	6036 (3)	28 (3)
C(24)	7036 (9)	10041 (12)	6101 (3)	34 (3)
C(25)	6019 (9)	9514 (11)	6276 (3)	30 (3)
C(26)	5891 (6)	7940 (10)	6384 (3)	25 (2)
S(2)	3188 (2)	1643 (3)	8224.8(9)	34.7(7)
P(2)	3894 (2)	3411 (3)	8526.2(7)	19.2(5)
O(2)	4896 (5)	4173 (7)	8252 (2)	23 (2)
C(31)	4435 (7)	2977 (10)	9127 (2)	22 (2)
C(32)	5423 (8)	3595 (12)	9294 (3)	28 (3)
C(33)	5781 (8)	3326 (14)	9769 (3)	40 (3)
C(34)	5145 (8)	2391 (15)	10066 (3)	41 (4)
C(35)	4161 (9)	1706 (14)	9899 (3)	47 (4)
C(36)	3814 (7)	2029 (11)	9433 (3)	32 (3)
C(41)	2901 (6)	4957 (10)	8643 (2)	19 (2)
C(42)	3259 (7)	6477 (10)	8676 (3)	23 (2)
C(43)	2524 (8)	7625 (11)	8801 (4)	28 (3)
C(44)	1421 (7)	7292 (11)	8911 (3)	33 (3)
C(45)	1039 (9)	5798 (12)	8874 (4)	36 (3)
C(46)	1781 (7)	4612 (11)	8743 (3)	29 (3)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

matrix least-squares refinement with the SHELXTL PLUS (VMS) [23] program system. The structure was solved by direct methods for antimony and the difference Fourier synthesis revealed the positions for the other non-hydrogen atoms. All non-hydrogen atoms were refined anisotropically and hydrogen atoms were calculated as a riding model with isotropic U thermal parameters.

The final R values are $R = 4.00\%$ and $wR = 4.34\%$ ($R = 5.40\%$ and $wR = 4.70\%$ for all data) with weights $w^{-1} = \sigma^2(F) + 0.0003F^2$; goodness-of-fit 1.13. The residual electron density from a final difference Fourier synthesis was in the range of -0.63 to 0.89 s Å^{-3} (near antimony). Refined values for the atomic coordinates are given in Table 8.

Further details of the crystal structure investigation can be obtained from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen, Germany, on quoting the deposit number CSD-57317, the names of the authors and the journal citation.

Acknowledgments

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