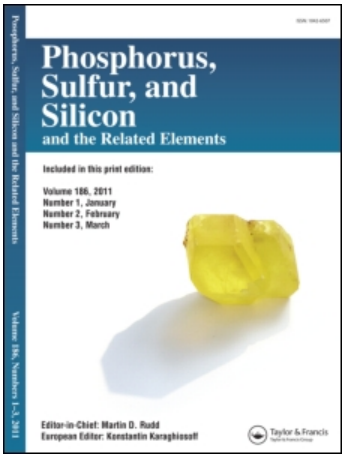

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

Mixed Chloro Bis[Alkylenedithiophosphato] Antimony(III) and Their Heterobinuclear Derivatives with Boron Tetraisopropoxide: Synthesis and Characterization

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Online publication date: 24 February 2010

To cite this Article Agrawal, Reena , Sharma, Jyoti , Singh, Yashpal , Nandani, Durgesh and Batra, Amla(2010) 'Mixed Chloro Bis[Alkylenedithiophosphato] Antimony(III) and Their Heterobinuclear Derivatives with Boron Tetraisopropoxide: Synthesis and Characterization', Phosphorus, Sulfur, and Silicon and the Related Elements, 185: 3, 516 — 525
To link to this Article: DOI: 10.1080/10426500902839848
URL: http://dx.doi.org/10.1080/10426500902839848

Phosphorus, Sulfur, and Silicon, 185:516–525, 2010

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500902839848

MIXED CHLORO BIS[ALKYLENEDITHIOPHOSPHATO] ANTIMONY(III) AND THEIR HETEROBINUCLEAR DERIVATIVES WITH BORON TETRAISOPROPOXIDE: SYNTHESIS AND CHARACTERIZATION

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Chloro bis(alkylenedithiophosphato)antimony(III) complexes of the type [$\overline{OGOP}$(S)S]$_2$SbCl (1–5) [where G = $-C(Me)_2-CH_2-CH(Me)-${1} $-C(Me)_2-C(Me)_2-${2}, $-CH_2-C(Me)_2-CH_2-${3}, $-CH(Me)-CH(Me)-$ {4} and $-CH(Me)-CH_2-CH_2-${5}] have been synthesized by the reaction of $SbCl_3$ with sodium salts of alkylenedithiophosphoric acids in a 1:2 molar ratio in refluxing benzene. Reactions of chloro bis(alkylenedithiophosphato) antimony(III) compounds, [$\overline{OGOP}$(S)S]$_2$SbCl with sodium tetraisopropoxoborate, NaB(OPr^i)$_4$, in a 1:1 molar ratio in refluxing benzene yielded some new heterobinuclear derivatives of antimony(III) and boron(III) of the type[$\overline{OGOP}$(S)S]$_2$Sb(μ-OPr^i)$_2$B(OPr^i)$_2$. These newly synthesized complexes have been characterized by elemental analysis and molecular weight measurement, and their plausible structures have been proposed on the basis of IR, NMR (^{1}H, ^{13}C, ^{31}P, ^{11}B), and FAB-mass spectral studies. On the basis of the spectroscopic evidence, a pseudo octahedral geometry around antimony and tetrahedral geometry around boron atom has been proposed. Cyclic O,O′-alkylenedithiophosphate ligands and their corresponding chloro bis(alkylenedithiophosphato)antimony(III) compounds have been screened for microbial activities. These compounds showed significant antifungal activity against* **Fusarium** *and* **Trichoderma** *and antibacterial activity against* **E. Coli** *and* **Pseudomonas.

Supplemental materials are available for this article. Go to the publisher's online edition of* Phosphorus, Sulfur, and Silicon and the Related Elements *to view the free supplemental file.

Keywords Cyclic O,O′-alkylenedithiophosphoric acid; NMR and FAB-mass spectral data; pseudo-octahedral geometry of antimony; tetracoordinated boron

INTRODUCTION

Organocompounds of antimony find extensive applications in chemotherapy,[1–3] as these compounds exhibit significant antimicrobial,[1] antiparasitic,[2] and antitumor[3] activities. The biological toxicity of these derivatives is much less than those of Pt and Pd anticancer

Received 31 August 2008; accepted 22 February 2009.
We thank CDRI Lucknow (SAIF Centre) for recording the FAB-mass of one of the compounds.
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substances.[4] A large number of antimony compounds have also been tested as bactericides,[5] fungicides,[6] and antifertility agents.[7]

Cyclic O,O′-alkylenedithiophosphates of transition metals have been extensively studied,[8–10] and the studies have also been extended to the derivatives of nontransition metals, including organoantimony(III) moieties.[11–17] Metal derivatives of antimony(III) containing alkylenedithiophosphates also exhibit significant antimicrobial activities.[16,17] Although chloro bis(dialkyldithiophosphates) antimony(III) $[(RO)_2P(S)S]_2SbCl$[18,19] [where R = Et, Pr^i, Pr^n and Bu^n] and only one compound of chloro bis(alkylenedithiophosphates) antimony(III) $[\overline{OGOP}(S)S]_2SbCl$[12] [where G = $-CH(CH_3)-CH(CH_3)-$] have been reported, heterobinuclear compounds of antimony containing alkylenedithiophosphates have not been reported to date.

In this article, we report the synthesis and characterization of some chloro bis(alkylenedithiophosphates) and heterobinuclear derivatives of antimony(III) and boron(III) containing alkylenedithiophosphate ligands. A comparative study of the antimicrobial activity of the ligands and corresponding chloro bis(alkylenedithiophosphato) antimony(III) derivatives is also reported.

RESULTS AND DISCUSSION

Chloro bis(alkylenedithiophosphato)antimony(III) derivatives $[\overline{OGOP}(S)S]_2SbCl$ (**1–5**) have been synthesized by the reaction of $SbCl_3$ with sodium salts of alkylenedithiophosphoric acids in 1:2 molar ratio in refluxing benzene.

$$SbCl_3 + \overline{OGOP}(S)SNa \xrightarrow[\text{stirring}]{C_6H_6} [\overline{OGOP}(S)]_2SbCl + Nacl \downarrow$$

[Where G = $-C(Me)_2 - CH_2 - CH(Me) - \{1\}$, $- C(Me)_2 - C(Me)_2 - \{2\}$, $-CH_2 - C(Me)_2 - CH_2 - \{3\}$, $- CH(Me) - CH(Me) - \{4\}$ and $- CH(Me) -CH_2 - CH_2 - \{5\}$]

All these derivatives are found to be yellowish colored solids or viscous liquids, soluble in common organic solvents and monomeric in freezing benzene solution.

Heterobinuclear derivatives of antimony (III) $[\overline{OGOP}(S)S]_2Sb(\mu\text{-}OPr^i)_2B(OPr^i)_2$ (**1a–5a**) were prepared by the reaction of sodium tetraisopropoxoborate, $NaB(OPr^i)_4$, with chloro bis (alkylenedithiophosphato)antimony(III) compounds $[\overline{OGOP}(S)S]_2SbCl$ (**1–5**) in 1:1 molar ratio in refluxing benzene under anhydrous conditions.

$$[\overline{OGOP}(S)S]_2SbCl + NaB(OPr^i)_4 \xrightarrow[\text{stirring}]{C_6H_6} [\overline{OGOP}(S)S]_2Sb(\mu - OPr^i)_2B(OPr^i)_2 + NaCl \downarrow$$

All these derivatives (**1a–5a**) are found to be yellowish colored solids or viscous liquids, moisture-sensitive soluble in common organic solvents and monomeric in freezing benzene solution.

Infrared Spectra

The IR spectra of these compounds have been recorded in the range 4000–400 cm^{-1}. The bands in the regions 950–1050 cm^{-1} and 800–890 cm^{-1} may be assigned to ν(P)O—C and νP—O(C) stretching vibrations,[20,21] respectively, and other strong bands in the region 950–965 cm^{-1} may be assigned to dioxaphospholane and dioxaphosphorinane ring vibrations.[22,23] A strong band in the region 620–680 cm^{-1} may be assigned to νP=S stretching vibrations,[24] and the bands of medium intensity present in the region 510–625 cm^{-1} may be due to asymmetric and symmetric νP—S stretching vibrations.[21] Some additional bands have also been observed in the region 1010–1050 cm^{-1} and 1250–1460 cm^{-1} due to ν(Sb)O—C[25] and νB—O[26] stretching vibrations, respectively. The νSb—O(C)[27] stretching vibrations appeared in the region 540–575 cm.$^{-1}$ νSb—S and νSb—Cl could not be observed, as the spectra have been recorded in the range 4000–400 cm^{-1}.

^{1}H NMR Spectra

The ^{1}H NMR spectral data of the these complexes are summarized in Table I. The proton resonances due to all alkylene protons of the (OGO) group attached to P atom have been observed at their expected positions without any appreciable shift in their position as compared to their position in the spectra of the corresponding ligand. In addition to these signals, the spectra of heterobinuclear compounds also exhibit two sets of signals for terminal and bridging isopropoxy groups in the regions δ 1.13–1.39, δ 1.26–1.41 ppm (doublets) due to methyl proton [CH(C**H**$_3$)$_2$] and δ 3.68–4.79, δ 4.09–4.98 ppm (multiplets) [C**H**(CH$_3$)$_2$] due to methine proton, respectively.

^{13}C NMR Spectra

^{13}C NMR chemical shifts of these compounds have been summarized in Table I. ^{13}C NMR spectra of heterobinuclear derivatives (**1a–5a**) and chloro bis(alkylenedithiophosphato)antimony(III) compounds (**1–5**) provide useful information about the mode of bonding in these compounds.

The carbon signals due to all alkylene carbons of the glycol moieties have been observed at their expected position (Table I) without any appreciable shift in their position as compared to their position in the spectra of the corresponding ligands. The spectra of heterobinuclear derivatives exhibit an upfield shift $\sim \delta$ 2–8 ppm in the position of the signals of different alkylene group carbons of alkylenedithiophosphate moieties as compared to their positions in corresponding chloro bis(alkylenedithiophosphato)antimony(III) (**1–5**) compounds. This upfield shift may be due to the fact that the coordination number of the central antimony atom has increased in binuclear compounds.

The signals due to the methyl carbon atoms of the terminal and bridging isopropoxy group appeared at δ 20.67–23.64 and δ 21.52–31.59 ppm, respectively, while the methine (>CH—) carbons of the terminal and the bridging isopropoxy groups appeared in the range δ 73.17–76.92 and δ 75.61–77.43 ppm, respectively, indicating the nonequivalent nature of these groups.

Table I ^{1}H, ^{13}C, ^{31}P, and ^{11}B NMR spectral data (δ ppm) of new chloro bis(alkylenedithiophosphato) antimony(III) [$\overline{OGOP}(S)S]_2SbCl$ (**1–5**) and heterodinuclear derivatives [$\overline{OGOP}(S)S]_2Sb(\mu\text{-}OPr^i)_2B(OPr^i)_2$ (**1a–5a**)

Compound	^{1}H NMR chemical shift (δ ppm) (J in Hz)	^{13}C NMR chemical shift (δ ppm)	^{31}P NMR chemical shift (δ ppm)	^{11}B NMR chemical shift (δ ppm)
1	1.48–1.75, m, 22H (CH_3, CH_2) 4.84–4.91, m, 2H (OC**H**)	25.31 (**C**H_3) 29.53 (**C**H_2) 64.39 (O**C**H) 128.82 (O**C**)	78.93	—
1a	1.19, d, 12H $\{CH(CH_3)_2\}_t$; (6.9) 1.34, d, 12H $\{CH(CH_3)_2\}_b$; (6.6) 1.37–1.96, m, 22H (CH_3, CH_2)dtp 3.68–4.79, m, 2H $\{CH(CH_3)\}_t$ 4.81–4.98, merged, 4H $\{CH(CH_3)_2\}_b$, (OC**H**)dtp	22.22 (**C**H_3)dtp 23.64 $\{CH(CH_3)_2\}_t$ 27.35$\{CH(CH_3)_2\}_b$ 38.79 (**C**H_2)dtp 73.17 $\{CH(CH_3)$t 76.62 $\{CH(CH_3)_2\}_b$ 86.86 $(OCH)_{dtp}$ 128.32 $(OC)_{dtp}$	82.14	–17.03
2	1.42, s, 24H (CH_3)	23.66 (**C**H_3) 128.28 (O**C**)	102.35	—
2a	1.14, d, 12H $\{CH(CH_3)_2\}_t$; (6.2) 1.27, d, 12H $\{CH(CH_3)_2\}_b$; (6.2) 1.39, s, 24H $(CH_3)_{dtp}$ 4.66–4.72, merged,4H $\{CH(CH_3)_2\}_t$, $\{CH(CH_3)_2\}_b$	22.96 $(CH_3)_{dtp}$ 23.16 $\{CH(CH_3)_2\}_t$ 25.48 $\{CH(CH_3)_2\}_b$ 76.92 $\{CH(CH_3)_2\}_t$ 77.36 $\{CH(CH_3)_2\}_b$ 89.46 $(OC)_{dtp}$	104.72	–16.35
3	1.06, s, 12H (CH_3) 3.97, d, 8H (CH_2); (9.7)	19.31 (**C**H_3) 30.24 $\{C(CH_3)_2\}$ 126.33 (O**C**H_2)	90.17	—
3a	1.03, s, 12H $(CH_3)_{dtp}$ 1.17, d, 12H $\{CH(CH_3)_2\}_t$;(5.7) 1.37, d, 12H $\{CH(CH_3)_2\}_b$; (6.4) 2.55, d, 8H $[OCH_2]_{dtp}$; (21.0) 3.92–4.01, m, 2H $\{CH(CH_3)_2\}_t$ 4.09–4.14, m, 2H $\{CH(CH_3)_2\}_b$	20.69 $(CH_3)_{dtp}$ 21.06 $\{CH(CH_3)_2\}$t 21.52 $\{CH(CH_3)_2\}_b$ 32.02 $\{C(CH_3)_2\}_{dtp}$ 74.05 $\{CH(CH_3)_2\}_t$ 75.61 $(CH(CH_3)_2\}_b$ 128.36 $(OCH_2)_{dtp}$	91.45	–19.54
4	1.45, d, 12H (CH_3); (5.8) 4.23–4.68, m, 4H (OC**H**)	15.26 (**C**H_3) 78.40 (O**C**H)	108.05	—
4a	1.35, d, 12H $\{CH(CH_3)_2\}_t$; (6.4) 1.39,d, 12H $\{CH(CH_3)_2\}_b$; (6.0) 1.47, d, 12H $(CH_3)_{dtp}$; (6.4) 4.72–4.76, m, 2H $\{CH(CH_3)_2\}_t$ 4.76–4.81, merged, 8H $\{CH(CH_3)_2\}_b$, $[OCH]_{dtp}$	15.41 $(CH_3)_{dtp}$ 22.61 $\{CH(CH_3)_2\}_t$ 23.88 $\{CH(CH_3)_2\}_b$ 78.12 $(OCH)_{dtp}$ 76.14 $\{CH(CH_3)_2\}_t$ 77.09 $\{CH(CH_3)_2\}_b$	108.93	–20.41
5	1.40, d, 6H (CH_3); (6.0) 1.79–1.84, m, 4H (CH_2) 2.05–2.18, dt, 4H (OCH_2) 4.57–4.66, m, 2H(OC**H**)	22.27(**C**H_3) 37.42(**C**H_2) 67.63 (O**C**H_2) 128.32(O**C**H)	89.26	—
5a	1.37, d, 12H $\{CH(CH_3)_2\}_t$; (6.7) 1.40, d, 12H $\{CH(CH_3)_2\}_b$; (6.7) 1.45, d, 6H $(CH_3)_{dtp}$; (10.7) 1.64–1.77, m, 4H $(CH_2)_{dtp}$ 2.03–2.12, dt, 4H $(OCH_2)_{dtp}$ 4.28–4.42, m, 2H $\{CH(CH_3)_2\}_t$ 4.65–4.86, m, 2H$\{CH(CH_3)_2\}_b$ 4.57–4.64, m, 2H$(OCH)_{dtp}$	20.54 $(CH_3)_{dtp}$ 20.67 $\{CH(CH_3)_2\}_t$ 31.59 $\{CH(CH_3)_2\}_b$ 37.42 $(CH_2)_{dtp}$ 65.29 $(OCH_2)_{dtp}$ 76.56 $\{CH(CH_3)_2\}_t$ 77.43 $\{CH(CH_3)_2\}_b$ 126.63 $(OCH)_{dtp}$	90.27	–21.35

t = terminal, b = bridging, dtp = alkylenedithiophosphate, d = doublet, m = multiplet, dt = doublet of triplet.

^{31}P NMR Spectra

The ^{31}P NMR spectra exhibit a sharp singlet for the chloro bis(alkylenedithio phosphato)antimony(III) (**1–5**) and heterobinuclear derivatives (**1a–5a**) in the range δ 78.93–108.93 ppm (Table I). A comparative study of ^{31}P NMR spectra of chloro bis(alkylenedithiophosphato)antimony(III) (**1–5**) and bimetallic derivatives of antimony (III) (**1a–5a**) with their corresponding ligands show remarkable downfield shift $\sim \delta$ 5–10 ppm of >P(S)S phosphorous atom, indicating the bidentate nature of the ligands (Table II).[28,29]

^{11}B NMR Spectra

^{11}B NMR spectra also exhibit only one signal in the range δ –16.35 to –21.35 ppm (Table I) for **1a–5a** derivatives. There is a shift of 0.37–4.63 ppm as compared to parent precursor $Na[B(OPr^i)_4]$, which exhibits a signal at –16.72 ppm. Appearance of this signal in this range indicates the presence of a tetracoordinated boron atom in these compounds.[30,31]

FAB-Mass Spectra

The FAB-mass spectrum of one of the heterobinuclear compounds of O,O′-alkylenedithiophosphate derivatives of antimony (III) (**3a**) has been recorded, which shows the monomeric nature of this compound. The mass spectral fragmentation pattern of compound **3a** is summarized in Table S2 (Available online in the supplementary materials.

Structure Elucidation

In view of the above spectral data and the presence of two bidentate alkylenedithiophosphate ligand moieties and one chloro group attached to the antimony atom, the structure shown in Figure 1 may be proposed for these new chloro bis(alkylenedithiophosphato) antimony(III) derivatives (**1–5**) in which the geometry of the central antimony atom is pseudo-octahedral.

In view of the presence of two bidentate alkylenedithiophosphate ligand moieties around the central antimony atom and the presence of two types (terminal and bridging) of isopropoxy groups, the hexacoordination around the central antimony atom and tetracoordination around boron atom in the structure shown in Figure 2 may be tentatively proposed for these new heterobinuclear alkoxide derivatives (**1a–5a**).

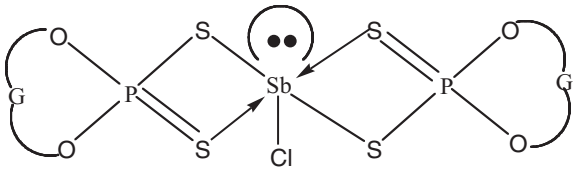

Figure 1 Proposed structure of compounds (**1–5**).

Table II Synthetic, physical, and analytical data of new chloro bis(alkylenedithiophosphato)antimony(III) derivatives $[\overline{OGOP}(S)S]_2SbCl$ (**1–5**)

S. No.	Reactants g (mmol)		NaCl (g) (mmol)	Color/ Physical state (mp) °C	Empirical formula and yield (%)	Mol. Wt. Found (Calc.)	Analysis (%)		
	$[\overline{OGOP}(S)S]_2Na$	$SbCl_3$					Sb Found (Calc.)	S Found (Calc.)	Cl Found (Calc.)
1.	1.62	0.78	0.40	Yellow/sticky solid	$C_{12}H_{24}P_2S_4O_4SbCl$	542.78	20.90	22.01	5.77
	(3.46)	(3.42)	(3.42)	70	93	(579.75)	(21.00)	(22.07)	(6.11)
2.	1.56	0.76	0.39	Yellow/solid	$C_{12}H_{24}P_2S_4O_4SbCl$	541.79	20.93	22.07	5.82
	(3.33)	(3.33)	(3.34)	**	90	(579.75)	(21.00)	(22.07)	(6.11)
3.	1.59	0.82	0.42	Pale yellow/ viscous	$C_{10}H_{20}P_2S_4O_4SbCl$	523.66	22.03	23.19	6.38
	(3.61)	(3.59)	(3.59)	liq.	92	(551.7)	(22.07)	(23.20)	(6.43)
4*.	1.57	0.87	0.45	Yellow/sticky solid	$C_8H_{16}P_2S_4O_4SbCl$	516	23.12	24.45	6.41
	(3.80)	(3.81)	(3.85)	65	94	(523.65)	(23.25)	(24.44)	(6.77)
5.	1.97	1.09	0.56	White/solid	$C_8H_{16}P_2S_4O_4SbCl$	498.62	23.10	24.41	6.41
	(4.78)	(4.78)	(4.79)	90	92	(523.65)	(23.25)	(24.44)	(6.77)

*Reported earlier.[12]

**Decomposed upon heating at 120°C.

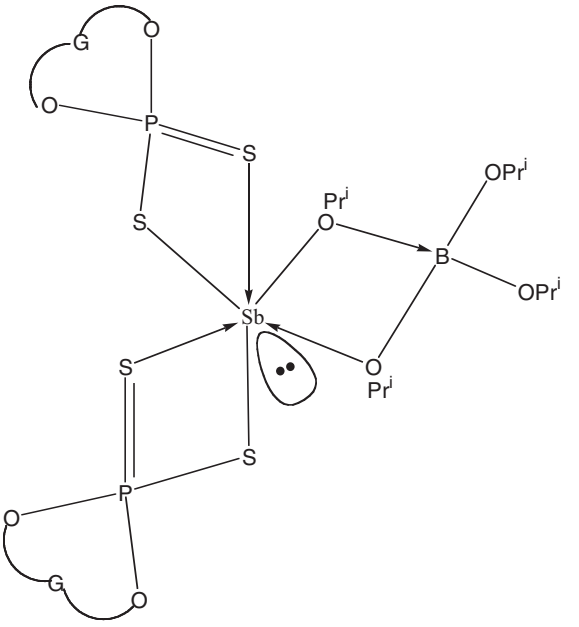

Figure 2 Proposed structure of compounds (**1a–5a**).

BIOLOGICAL ACTIVITY

A summary of the biological activities is given in the Supplemental Materials (available online).

EXPERIMENTAL

All the chemicals used were of reagent grade. All reactions were carried out under anhydrous conditions. Solvents (E. Merck) were carefully dried by standard methods[32] before use. Chloro bis(alkylenedithiophosphato) antimony (III), $[\overline{OGOP}(S)S]_2SbCl$,[12] and sodium tetraisopropoxoborate, $NaB(OPr^i)_4$[33] were prepared according to the methods in the literature. Sulfur was estimated by Messenger's method.[34] Boron and antimony were determined as methyl borate and by iodometric methods, respectively.[35] Isopropoxide was determined by the chromate oxidometric method.[36]

Molecular weights were determined cryoscopically in freezing benzene solution using a Beckmann's thermometer. The IR spectra were recorded on an 8400S Shimadzu FT-IR spectrophotometer as liquid films on a KBr cell in the range 4000–400 cm^{-1}. ^1H, ^{13}C, ^{31}P, and ^{11}B NMR spectra were recorded in $CDCl_3$ solution on a JEOL-FT AL 300 MHz spectrometer. TMS was used as an internal reference for ^1H and ^{13}C NMR spectra, whereas H_3PO_4 and $B(OCH_3)_3$ were used as external standards for ^{31}P and ^{11}B NMR spectra, respectively.

Since a similar method has been used for the synthesis of all compounds (**1a–5a**), the synthetic detail of only one compound has been discussed in detail. Synthetic and analytical data of other compounds have been summarized in Table III.

Table III Synthetic, physical, and analytical data of new heterodinuclear derivatives $[\overline{OGOP}(S)S]_2Sb(\mu\text{-}OPr^i)_2B(OPr^i)_2$ (**1a–5a**)

S.No.	Reactants g (m mol)		NaCl (g) (mmol)	Color/ Physical state (mp) °C	Empirical formula and yield (%)	Mol. Wt. Found (Calc.)	Analysis (%)			
	$[\overline{OGOP}(S)S]_2SbCl$	$NaB(OPr^i)_4$					Sb Found (Calc.)	S Found (Calc.)	B Found (Calc.)	OPr^i Found (Calc.)
1a	1.46	0.68	0.15	Pale yellow/ Viscous liq.	$C_{24}H_{52}P_2S_4O_8SbB$	748	15.33	16.16	1.29	29.83
	(2.52)	(2.52)	(2.57)		92.5	(791.36)	(15.38)	(16.20)	(1.36)	(29.86)
2a	1.50	0.70	0.15	Pale yellow/ Viscous liq.	$C_{24}H_{52}P_2S_4O_8SbB$	740	15.32	16.16	1.28	29.43
	(2.59)	(2.59)	(2.57)		90.9	(791.36)	(15.38)	(16.20)	(1.36)	(29.86)
3a	1.80	0.88	0.19	Pale yellow/ solid	$C_{22}H_{48}P_2S_4O_8SbB$	747	15.84	16.76	1.32	30.50
	(3.26)	(3.26)	(3.25)	95	90	(763.31)	(15.95)	(16.80)	(1.41)	(30.95)
4a	1.76	0.91	0.20	Creamish / solid	$C_{20}H_{44}P_2S_4O_8SbB$	735	16.41	17.40	1.35	31.92
	(3.36)	(3.37)	(3.42)	80	89.2	(735.26)	(16.56)	(17.44)	(1.47)	(32.14)
5a	1.78	0.92	0.20	Creamish / solid	$C_{20}H_{44}P_2S_4O_8SbB$	735	16.41	17.40	1.41	32.10
	(3.40)	(3.40)	(3.42)	85	92	(735.26)	(16.56)	(17.44)	(1.47)	(32.14)

Synthesis of Heterobinuclear Derivatives 3a

Freshly prepared sodium tetraisopropoxoborate, $NaB(OPr^i)_4$ (0.88 g, 3.25 mmol), was added to a benzene solution (~40 mL) of $[\overline{OGOP}(S)S]_2SbCl$ (**3**) [Where G = $-CH_2-C(Me)_2-CH_2-$(3)] (1.80 g, 3.26 mmol), and the reaction mixture was refluxed with stirring for ~5 h. The precipitated NaCl (0.19 g, 3.25 mmol) was removed by filtration, and the solvent was removed under reduced pressure. A pale yellow solid compound was obtained in quantitative yield (92%), derivative **3a**, which was purified by recrystallization from a 1:2 mixture of benzene and n-hexane. The pure compound was obtained in quantitative yield (90%).

REFERENCES

1. C. Silvestru and I. Haiduc, *Main Group Elements and Their Compounds* (Narosa, New Delhi, 1996).
2. P. D. Marden, *Rev. Soc. Pras. Med. Trop.*, **18,** 187 (1985).
3. C. Silvestru, M. Curtui, I. Haiduc, M. J. Begley, and D. B. Sowerby, *J. Organomet. Chem.*, **426,** 49 (1992).
4. C. Silvestru, C. Socaciu, A. Bara, and I. Haiduc, *Anticancer Res*., **10,** 803 (1990).
5. N.V. Sawant and S.S. Garje, *Main Group Met. Chem..*, **28,** 213 (2005).
6. J. D. Curry, U.S. Pat. 3,383,565 (1974); *Chem. Abstr.*, **82,** 88069 (1974).
7. P. K. Sharma, H. Rehwani, A. K. Rai, R. S. Gupta, and Y. P. Singh, *Bioinorg. Chem. Appl.*, Article No. 16895, 1 (2006).
8. C. P. Bhasin, G. Srivastava, and R. C. Mehrotra, *Inorg. Chim. Acta*, **77,** L131 (1983).
9. R. Ratnani, G. Srivastava, and R. C. Mehrotra, *Trans. Metal Chem.*, **16,** 204 (1991).
10. A. Kumar and S. K. Pandey, *J. Chem.*, **79,** 951 (2005).
11. P. K. Sharma, A. K. Rai, and Y. P. Singh, *Main Group Met. Chem.*, **29,** 311 (2006).
12. H. P. S. Chauhan, G. Srivastava, and R. C. Mehrotra, *Polyhedron*, **3,** 1337 (1984).
13. R. K. Gupta, A. K. Rai, R. C. Mehrotra, and V. K. Jain, *Inorg. Chim. Acta*, **88,** 201 (1984).
14. A. K. Jain and R. Bohra, *Appl. Organometal. Chem.*, **20,** 411 (2006).
15. H. P. S. Chauhan, B. Porwal, and R. K. Singh, *Phosphorous, Sulfur, and Silicon*, **160,** 93 (2000).
16. H. P. S. Chauhan, U. P. Singh, N. M. Shaik, S. Mathur, and V. Huch, *Polyhedron*, **25,** 2841 (2006).
17. H. P. S. Chauhan and U. P. Singh, *Appl. Organomet. Chem.*, **21,** 880 (2007).
18. H. P. S. Chauhan, G. Srivastava, and R. C. Mehrotra, *Polyhedron*, **2,** 359 (1983).
19. H. P. S. Chauhan and S. Lunkad, *Main Group Met. Chem.*, **17,** 313 (1994).
20. D. E. C. Corbridge, *Topics in Phosphorous Chem.*, **6,** 235 (1969).
21. D. B. Sowerby, I. Haiduc, A. B. Rusu, and M. Salajan, *Inorg. Chim. Acta*, **68,** 87 (1983).
22. J. Cason, W. N. Baxter, and W. Deacetis, *J. Org. Chem.*, **24,** 247 (1959).
23. R. A. Y. Jones and A. R. Katritzky, *J. Chem. Soc.*, 4376 (1960).
24. B. Walther, *Z. Anorg. Allg. Chem.*, **395,** 211 (1972).
25. M. L. Hitchman and K. F. Jensen, *Chemical Vapour Deposition: Principles and Applications* (Academic Press, London, 1993).
26. W. J. Lehmann, H. G. Weiss, and I. Shapiro, *J. Chem. Phys.*, **30,** 1226 (1959).
27. T. Athar, R. Bohra, and R. C. Mehrotra, *Indian J. Chem.*, **28A,** 302 (1989).
28. C. Gilidewell, *Inorg. Chim. Acta*, **25,** 159 (1977).
29. P. K. Sharma and Y. P. Singh, *J. Chem. Res.*, 727 (2006).
30. M. P. Hughes and B. D. Smith, *J. Org. Chem.*, **62,** 4492 (1997).
31. P. K. Sharma and Y. P. Singh, *Phosphorous, Sulfur, and Silicon*, **182,** 1183 (2007).

32. D. D. Parrin, W. L. F. Armarago, and D. R. Parrin, *Purification of Laboratory Chemicals*, 2nd Ed. (Pergamon Press, New York, 1980).
33. D. C. Bradley, R. C. Mehrotrta, I. P. Rothwell, and A. Singh, In *Alkoxo and Aryloxo Derivatives of Metals* (Academic Press, London, 2001), p. 183.
34. A. I. Vogel, *A Textbook of Quantitative Inorganic Analysis* (Longmans, London, 1989).
35. D. C. Bradley, F. M. Abdel-Halim, and W. Wardlaw, *J. Chem. Soc.*, 3450 (1950).