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Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:

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To cite this Article Singh, Yashpal(2007) 'Studies on Homo- and Hetero-Dinuclear Derivatives of Antimony(III) Containing Schiff Base Ligands', *Phosphorus, Sulfur, and Silicon and the Related Elements*, 182: 8, 1805 – 1817

To link to this Article: DOI: 10.1080/10426500701323358

URL: <http://dx.doi.org/10.1080/10426500701323358>

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Studies on Homo- and Hetero-Dinuclear Derivatives of Antimony(III) Containing Schiff Base Ligands

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Some new heterodinuclear antimony(III) derivatives of Schiff base having the composition, $K(\mu\text{-OPr}^i)_2\text{Sb}[\text{SC}_6\text{H}_4\text{NC(R)CHC(O)COOCH}_3]$ (1a–1e) have been synthesized by the reactions of $\text{KSb(OPr}^i)_4$ with benzothiazolines, $\text{HN}(\text{C}_6\text{H}_4\text{SC(R)CHC(O)COOCH}_3$ in 1:1 molar ratio. Homodinuclear derivatives of antimony(III), $\text{Ph}_2\text{Sb}(\mu\text{-OPr}^i)_2\text{Sb}[\text{SC}_6\text{H}_4\text{NC(R)CHC(O)COOCH}_3]$ (2a–2e) have been synthesized by equimolar reaction of $\text{Ph}_2\text{Sb}(\mu\text{-OPr}^i)_2\text{Sb(OPr}^i)_2$ (Prepared by the reaction of $\text{KSb(OPr}^i)_4$ with Ph_2SbCl in 1:1 molar ratio) with benzothiazoline ligands in benzene. The reactions proceed with rearrangement of benzothiazoline ring. All the derivatives have been characterized by elemental analysis, molecular weight determinations, as well as conductance measurements and their plausible structure have been proposed on the basis of IR and NMR (^1H and ^{13}C) spectral studies.

Keywords Antimony(III); benzothiazolines; heterodinuclear; homodinuclear; Schiff base

INTRODUCTION

Currently much attention is being paid to the preparation of metal oxide materials which are used as microelectronics,^{1,2} ceramics,³ coating,⁴ and catalytic materials.^{5,6} Antimony oxides have potential applications in the field of electronic ceramics and heterogenous catalysis,^{7,8} etc. Recently Horley et al.⁹ and Haycock et al.¹⁰ have prepared homoleptic antimony(III) derivatives of carbamates, which have been used as single source precursor for preparation of antimony oxide by MOCVD technique. A survey of literature reveals that a significant number of homo- and hetero-metallic alkoxide derivatives of antimony(III)^{11–13} have been reported, however, homo- and hetero-nuclear antimony(III) derivatives with chelating ligands have received little attention.

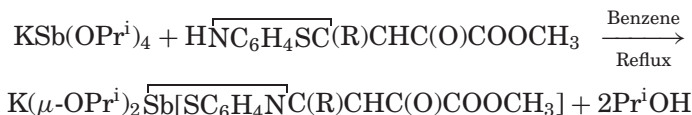
Received January 7, 2007; accepted February 19, 2007.

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In the present article, we report the synthesis and characterization of homo- and hetero-nuclear antimony(III) alkoxo derivatives of benzothiazoline ligands.

RESULTS AND DISCUSSION

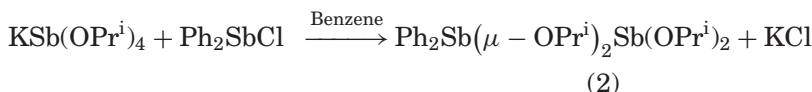
The equimolar reaction of $\text{KSb}(\text{OPr}^i)_4$ with ligands, $\text{H}\overline{\text{NC}_6\text{H}_4\text{SC}}(\text{R})\text{CHC}(\text{O})\text{COOCH}_3$ [where $\text{R} = \text{C}_6\text{H}_5(\text{L}^1\text{H}_2)$, $4\text{-BrC}_6\text{H}_4(\text{L}^2\text{H}_2)$, $4\text{-ClC}_6\text{H}_4(\text{L}^3\text{H}_2)$, $4\text{-CH}_3\text{OC}_6\text{H}_4(\text{L}^4\text{H}_2)$, $4\text{-CH}_3\text{C}_6\text{H}_4(\text{L}^5\text{H}_2)$] have been carried out in refluxing benzene. The reactions proceed with the opening of benzothiazoline ring and yield heterodinuclear derivatives of antimony(III), $\text{K}(\mu\text{-OPr}^i)_2\text{Sb}[\overline{\text{SC}_6\text{H}_4\text{NC}}(\text{R})\text{CHC}(\text{O})\text{COOCH}_3]$.



[where $\text{R} = \text{C}_6\text{H}_5(\mathbf{1a})$, $4\text{-BrC}_6\text{H}_4(\mathbf{1b})$, $4\text{-ClC}_6\text{H}_4(\mathbf{1c})$, $4\text{-CH}_3\text{OC}_6\text{H}_4(\mathbf{1d})$, $4\text{-CH}_3\text{C}_6\text{H}_4(\mathbf{1e})$]

Progress, as well as completion of reaction, was checked by estimating the isopropanol in the azeotrope, which was fractionated off azeotropically with benzene during the course of reaction.

The reaction of $\text{KSb}(\text{OPr}^i)_4$ with Ph_2SbCl have been carried out in benzene. KCl was filtered off and the removal of excess solvents under vacuum yield yellow brown liquid compound, $\text{Ph}_2\text{Sb}(\mu\text{-OPr}^i)_2\text{Sb}(\text{OPr}^i)_2$, (**2**).



The reactions of compound (**2**) with ligands have been carried out in 1:1 molar ratio yield homodinuclear Schiff base derivatives of antimony(III) (**2a-2e**).



[where $\text{R} = \text{C}_6\text{H}_5(\mathbf{2a})$, $4\text{-BrC}_6\text{H}_4(\mathbf{2b})$, $4\text{-ClC}_6\text{H}_4(\mathbf{2c})$, $4\text{-CH}_3\text{OC}_6\text{H}_4(\mathbf{2d})$, $4\text{-CH}_3\text{C}_6\text{H}_4(\mathbf{2e})$].

Isopropanol liberated during the reaction was fractionated off azeotropically with benzene, which was estimated to check the progress, as well as completion of reaction.

All the complexes are soluble in common organic solvents. Conductance measurements of the compounds (1a–1e) in nitrobenzene show their nonelectrolytic nature. Molecular weight measurements of these complexes reveal their monomeric nature.

SPECTROSCOPIC STUDIES

Infra-Red Spectra

The bands for –OH and –NH groups observed at $3638\text{--}3417\text{ cm}^{-1}$ and $3325\text{--}3225\text{ cm}^{-1}$ in the spectra of free ligands are found to be absent in the spectra of the corresponding complexes (1a–1e and 2a–2e). The new bands observed at $1625\text{--}1590\text{ cm}^{-1}$, $425\text{--}410\text{ cm}^{-1}$, $408\text{--}400\text{ cm}^{-1}$, and $590\text{--}580\text{ cm}^{-1}$ have been assigned to $\nu\text{C}=\text{N}$, $\nu\text{Sb}\leftarrow\text{N}$,¹⁴ $\nu\text{Sb}\text{--S}$,^{15,16} and $\nu\text{Sb}\text{--O}$ ¹⁷ modes, respectively. Disappearance of NH band and appearance of C=N, Sb \leftarrow N, and Sb-S bands indicate the rearrangement of benzothiazoline ring during complexation. Moreover, the deprotonation of –OH group and formation of Sb–O bond, show the involvement of > C–O group in bonding.

The absorption band observed for isopropoxy groups at 1065 cm^{-1} , 970 cm^{-1} , and 600 cm^{-1} in the spectrum of $\text{KSb}(\text{OPr}^i)_4$ have been assigned to $\nu\text{C}\text{--O}$, **I** (Symmetric stretching, terminal- OPr^i)¹⁸, $\nu\text{C}\text{--O}$, **II** (Symmetric stretching, bridging- OPr^i)¹⁸ and $\nu\text{Sb}\text{--O}$, **III** (Symmetric stretching, bridging - OPr^i)¹⁸ mode of vibrations, respectively.

The spectrum of compound (**2**) shows the bands at 1120 cm^{-1} , 950 cm^{-1} , 610 cm^{-1} and 735 cm^{-1} , which have been assigned to $\nu\text{C}\text{--O}$ (**a**) (Symmetric stretching, terminal- OPr^i)¹⁹, $\nu\text{C}\text{--O}$ (**b**) (Symmetric stretching, bridging- OPr^i)¹⁹, $\nu\text{Sb}\text{--O}$ (**c**) (Symmetric stretching, bridging- OPr^i)¹⁹ and $\nu\text{Sb}\text{--O}\text{--Sb}$ ²⁰ modes, respectively.

In the spectra of the complexes (1a–1e and 2a–2e), absence of the band for νCO [**I** and (**a**)] indicates that the terminal isopropoxy groups of $\text{KSb}(\text{OPr}^i)_4$ and compound (**2**) have been substituted by ligand moiety during complex formation. It is further supported by the deprotonation of –OH and –NH groups of ligands and shift in the positions of the band for $\nu\text{C}\text{--O}$ [**II** ($1010\text{--}980\text{ cm}^{-1}$) and (**b**) ($1000\text{--}990\text{ cm}^{-1}$)], and $\nu\text{Sb}\text{--O}$ [**III** ($625\text{--}615\text{ cm}^{-1}$) and (**c**) ($695\text{--}690\text{ cm}^{-1}$)] of the bridging isopropoxy groups as compared to their position in $\text{KSb}(\text{OPr}^i)_4$ and compound (**2**), respectively.

¹H NMR Spectra

In the ¹H NMR spectra of the complexes [1a–1e and 2a–2e] the signals observed at $\delta\ 15.1\text{--}15.35\text{ ppm}$ and $\delta\ 3.90\text{--}4.40\text{ ppm}$ due to –OH and –NH

groups in the spectra of free ligands, are found to be absent, indicating the deprotonation of these groups during complex formation.

The signals which were observed at δ 4.01 ppm and δ 1.21 ppm due to $-\text{OCH}$ and $-\text{CH}_3$ groups of terminal isopropoxy groups of $\text{KSb}(\text{OPr}^i)_4$, disappeared from the spectra of the complexes (1a–1e). In addition to these, the signal for $-\text{OCH}$ and $-\text{CH}_3$ protons of bridging isopropoxy groups show upfield shift in their position as compared to their position in the spectra of $\text{KSb}(\text{OPr}^i)_4$, and they have been observed at δ 4.51 ppm and δ 1.27 ppm, respectively [Table I].

The signals appearing at δ 4.05 ppm and δ 1.07 ppm and assigned to $-\text{OCH}$ and $-\text{CH}_3$ protons of terminal isopropoxy groups of compound (2) are found to be absent in the spectra of the complexes (2a–2e). The upfield shifts were observed in the position of the signals for $-\text{OCH}$ and $-\text{CH}_3$ protons of bridging isopropoxy groups in the spectra of the complexes as compared to their position in compound (2). These signals are being observed at δ 4.40 ppm and δ 1.17 ppm, respectively.

The signals for $=\text{OCH}$, $\text{OCH}_3(\text{ester})$ and phenyl group protons appeared in the spectra of these complexes have been summarized in Tables I and II.

¹³C NMR Spectra

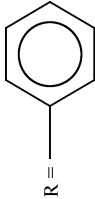
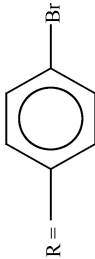
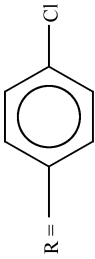
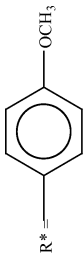
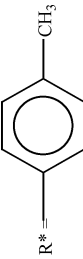
In the ¹³C NMR spectra of the complexes (1a–1e and 2a–2e), The signals appearing at δ 158.68–160.07 ppm and δ 161.88–163.1 ppm, due to CN and C–O groups in the spectra of free ligands, show downfield shift on complexation, which is further supported by a downfield shift observed in the position of signal for $=\text{CH}$ group as compared to its position in the spectra of free ligands.

The terminal isopropoxy groups are found to be absent in the spectra of the complexes (1a–1e), which were observed at δ 61.61 ppm ($-\text{OCH}$) and δ 25.87 ppm ($-\text{CH}_3$) in the spectra of $\text{KSb}(\text{OPr}^i)_4$. Moreover, the signal for $-\text{OCH}$ (δ 79.17 ppm) and $-\text{CH}_3$ (δ 28.15 ppm) group carbons of bridging $-\text{OPr}^i$ groups of $\text{KSb}(\text{OPr}^i)_4$ show upfield shift on complexation (Table III).

In the spectra of compound (2) the signals observed at δ 61.98 ppm and δ 25.43 ppm have been assigned to $-\text{OCH}$ and $-\text{CH}_3$ group carbons of terminal $-\text{OPr}^i$ groups, which are found to be absent in the spectra of the complexes (2a–2e). The bridging isopropoxy groups of compound (2) appear at δ 79.16 ppm ($-\text{OCH}$) and δ 30.50 ppm ($-\text{CH}_3$). These signals show an upfield shift in the spectra of complexes (2a–2e) (Table IV).

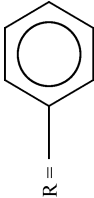
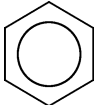
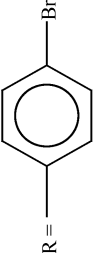
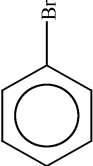
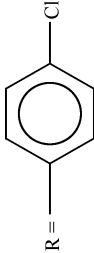
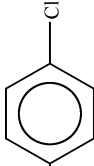
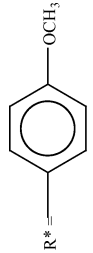
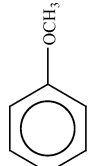
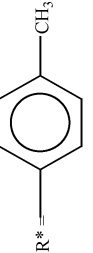
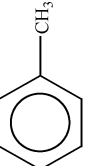
The C=O, OCH_3 (ester) and aromatic group carbons are also observed in the spectra of the complexes 1a–1e and 2a–2e, have been summarized in Tables III and IV. In addition to the signals observed in the spectra of 1a–1e some new signals have been observed in the

TABLE I ¹H NMR Spectral Data (δ ppm) of the Complexes, K(μ-OPrⁱ)₂Sb[SC₆H₄NC(R)CHC(O)COOCH₃](1a-1e)

S. no.	Complexes	R	-NC ₆ H ₄ S-	=CH	OCH ₃ (etser)	CH(OPr ⁱ)	CH ₃ (OPr ⁱ)
1		7.11-7.32 (m, 5H)	7.47-8.18 (m, 4H)	9.02 (s, 1H)	2.63 (s, 3H)	4.04 (m, 2H)	1.22 (d, 12H)
2	(1a) 	6.93-7.48 (m, 4H)	7.50-8.17 (m, 4H)	9.01 (s, 1H)	2.59 (s, 3H)	4.03 (m, 2H)	1.21 (d, 12H)
3	(1b) 	7.07-7.49 (m, 4H)	7.51-8.16 (m, 4H)	9.01 (s, 1H)	2.58 (s, 3H)	4.02 (m, 2H)	1.21 (d, 12H)
4	(1c) 	6.94-7.90 (m, 4H)	7.50-8.15 (m, 4H)	9.02 (s, 1H)	2.56 (s, 3H)	4.04 (m, 2H)	1.23 (d, 12H)
5	(1d) 	7.24-7.48 (m, 4H)	7.51-8.17 (m, 4H)	9.01 (s, 3H)	2.57 (s, 3H)	4.05 (m, 2H)	1.23 (d, 12H)
	(1e)						

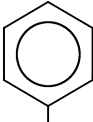
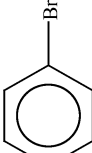
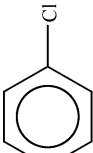
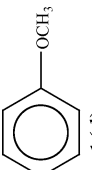
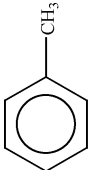
* = -OCH₃ and -CH₃ group protons are observed as singlet at δ 3.88 and 2.40, respectively. s = singlet; d = doublet; m = multiplet.

TABLE II ¹H NMR Spectral Data (δ ppm) of the Complexes, Ph₂Sb(μ-OPrⁱ)₂Sb[SC₆H₄NC(R)CHC(O)COOCH₃] (2a-2e)

S. no.	Complexes	R	-NC ₆ H ₄ S-	=CH	OCH ₃ (etser)	CH(OPr ⁱ)	CH ₃ (OPr ⁱ)	Ph-Sb
1	 R = 	7.15-7.29 (m, 5H)	7.50-8.07 (m, 4H)	9.00 (s, 1H)	2.51 (s, 3H)	4.02 (m, 2H)	1.13 (d, 12H)	7.32-7.46 (m, 5H)
2	(2a)  R = 	7.16-7.28 (m, 4H)	7.51-8.07 (m, 4H)	8.90 (s, 1H)	2.50 (s, 3H)	3.94 (m, 2H)	1.11 (d, 12H)	7.31-7.48 (m, 5H)
3	(2b)  R = 	7.17-7.29 (m, 4H)	7.48-8.09 (m, 4H)	8.94 (s, 1H)	2.53 (s, 3H)	3.95 (m, 2H)	1.14 (d, 12H)	7.33-7.46 (m, 5H)
4	(2c)  R* = 	6.01-7.28 (m, 4H)	7.52-8.06 (m, 4H)	8.87 (s, 1H)	2.44 (s, 3H)	4.04 (m, 2H)	1.17 (d, 12H)	7.30-7.49 (m, 5H)
5	(2d)  R* = 	7.20-7.39 (m, 4H)	7.58-8.15 (m, 4H)	8.94 (s, 1H)	2.54 (s, 3H)	4.15 (m, 2H)	1.18 (d, 12H)	7.40-7.49 (m, 5H)
	(2e)							

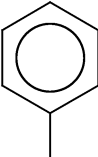
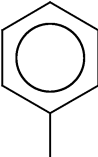
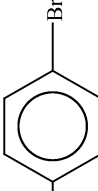
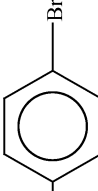
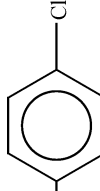
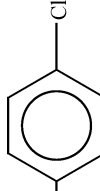
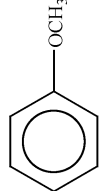
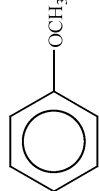
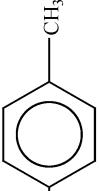
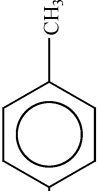
* = -OCH₃ and -CH₃ group protons are observed as singlet at δ 3.14 and 2.37, respectively. s = singlet; d = doublet; m = multiplet.

TABLE III ¹³C NMR Spectral Data (δ ppm) of the Complexes, K(μ-OPrⁱ)₂Sb[SC₆H₄NC(R)CHC(O)COOCH₃] (1a-1e)

S. no.	Complexes	R	-NC ₆ H ₄ S-	C-O	C-N	=CH	CH(OPr ⁱ)	CH ₃ (OPr ⁱ)	CH ₃ (ester)	C=O
1	 R = (1a)	136.78 128.64 127.36 126.32	156.01 139.34 128.89 127.87 126.32 122.98	166.90	163.95	97.25	61.97	25.45	26.67	196.50
2	 R = (1b)	133.78 129.89 127.37 126.98	156.86 138.36 130.95 128.34 126.64 122.42	167.31	163.07	97.52	61.92	25.41	26.47	196.24
3	 R = (1c)	133.50 130.05 127.86 126.78	156.00 138.07 130.72 128.75 127.15 122.98	167.00	164.50	97.20	61.97	25.44	26.69	196.90
4	 R* = 1(d)	130.43 129.86 126.37 125.42	155.98 133.50 130.22 128.00 126.11 122.45	168.01	163.06	96.90	61.91	25.44	26.34	196.23
5	 R* = (1e)	133.00 128.20 126.09 122.96	15.95 134.10 129.13 127.34 125.40 122.42	167.15	163.20	97.55	61.96	25.41	26.50	197.50

* = -OCH₃ and -CH₃ group carbon appeared at δ 55.48 ppm and 27.70 ppm, respectively.

TABLE IV ¹³C NMR Spectral Data (δ ppm) of the Complexes, Ph₂Sb(μ-OPrⁱ)₂Sb[SC₆H₄NC(R)CHC(O)COOCH₃] (2a-2e)

S. no.	Complexes	R	-NC ₆ H ₄ S-	C-O	C=N	=CH	CH(OPr ⁱ)	CH ₃ (OPr ⁱ)	CH ₃ (ester)	C=O	Ph-Sb
1	 R =  (2a)	138.20 130.26 128.92 126.13	155.97 135.83 129.23 128.64 127.42 122.32	166.84	163.10	97.80	62.5	25.45	26.66	197.89	138.72 130.83 128.11 125.43
2	 R =  (2b)	140.00 130.70 129.23 126.17	156.02 136.50 129.46 128.15 125.20 123.20	167.15	162.20	98.01	62.03	25.51	26.75	196.96	135.20 129.85 126.34 125.49
3	 R =  (2c)	140.10 131.46 129.15 125.64	155.92 136.26 128.46 127.83 125.42 123.32	16.39	162.13	97.92	62.19	25.64	26.70	196.70	135.46 128.10 126.41 125.32
4	 R* =  2(d)	135.81 131.39 128.92 126.13	155.97 138.19 130.43 128.65 125.43 122.45	167.50	162.90	97.50	63.10	25.44	26.34	196.50	141.00 134.29 127.50 122.98
5	 R* =  (2e)	135.81 130.65 128.43 126.23	156.19 139.83 130.39 128.23 125.20 122.01	167.26	163.02	97.21	62.97	25.72	26.83	196.04	140.34 135.00 127.13 121.94

* = -OCH₃ and -CH₃ group carbon are observed at δ 55.48 ppm and 27.56 ppm, respectively.

spectra of the compounds, (2a–2e), which have been assigned to phenyl group carbons attached to antimony in these compounds.

On the basis of above spectral evidences, it is clear that the benzothiazoline ligands behave as bifunctional tridentate moiety in the complexes. In view of the tridentate nature of ligand moiety, presence of two OPr^i groups between antimony and potassium or antimony and nonelectrolytic nature of compounds (1a–1e), the following structures may be proposed for the complexes (1a–1e) and (2a–2e), respectively.

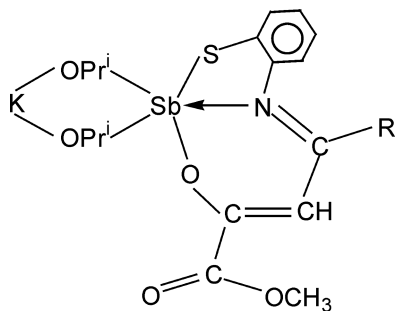


FIGURE 1 Proposed structure for the complexes (1a–1e).

EXPERIMENTAL

All the chemicals used were of reagent grade. Care was taken to exclude moisture throughout the experimental work. Solvents were dried by standard methods.²¹ The benzothiazoline ligands,²² $\text{KSb}(\text{OPr}^i)_4$,¹⁸ and Ph_2SbCl ²³ were prepared by reported methods. Antimony, nitrogen, and sulphur were estimated by iodometric,²⁴ Kjeldhal's,²⁴ and Messenger's method,²⁴ respectively. The isopropanol and isopropoxy groups were estimated oxidimetrically using 1N $\text{K}_2\text{Cr}_2\text{O}_7$ solution in 25% H_2SO_4 .²⁵ Molecular weight of the complexes have been measured ebullioscopically in benzene solution using Beckman's thermometer.

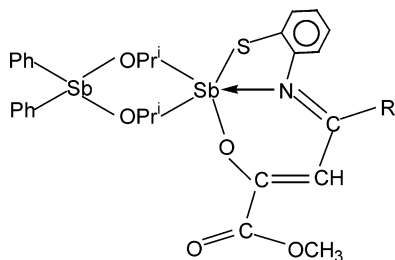


FIGURE 2 Proposed structure for the complexes (2a–2e).

Conductivity of the compounds (1a–1e) has been determined in nitrobenzene solution. Nitrobenzene was purified by standard method.²⁶

The IR spectra were recorded on a FT IR spectrophotometer Model 8400 S Shimadzu as liquid film and nujol mull on KBr optics in the range 4000–400 cm^{-1} . ^1H and ^{13}C NMR spectra were recorded in CDCl_3 or DMSO-d_6 solution on JEOL FT AL 300 MHz spectrometer. TMS was used as an external and internal reference for ^{13}C and ^1H NMR spectra, respectively.

Since similar methods have been used for the synthesis of all the complexes therefore, for the sake of brevity, the synthesis of one representative complex each of both the series is given in detail. The synthetic and analytical data of other analogues complexes have been summarized in Tables V and VI.

Synthesis of $\text{K}(\mu\text{-OPr}^i)_2\text{Sb}[\text{SC}_6\text{H}_4\text{NC}(\text{C}_6\text{H}_5)\text{CHC}(\text{O})\text{COOCH}_3]$

A benzene solution (~ 25 ml) of $\text{KSb}(\text{OPr}^i)_4$ (1.47 g, 3.70 mmol) was mixed with a benzene solution of corresponding ligand, L^1H_2 (1.16g, 3.70 mmol), and the mixture was refluxed on fractionating column for about four h. Isopropanol liberated was fractionated off as an azeotrope with benzene periodically, and it was estimated to check the progress and completion of reaction. After completion of reaction, removal of the excess solvents under reduced pressure, yielded yellow solid compound, which was further purified by dissolving it in benzene; to this, a clear solution n-hexane was mixed until the precipitates began to separate. The precipitate was redissolved by heating. This solution was cooled at low temperature ($0\text{--}5^\circ\text{C}$), when the compound precipitates, the solvent was decanted, and compound was finally dried under vacuum.

Synthesis of

$\text{Ph}_2\text{Sb}(\mu\text{-OPr}^i)_2\text{Sb}[\text{SC}_6\text{H}_4\text{NC}(\text{C}_6\text{H}_5)\text{CHC}(\text{O})\text{COOCH}_3]$

A benzene solution of $\text{KSb}(\text{OPr}^i)_4$ (6.30 g, 15.86 mmol) was mixed with a benzene solution of Ph_2SbCl (4.94g, 15.86 mmol), and the mixture was stirred and gently warmed for about five h. KCl was precipitated during the reaction. The solution was cooled at room temperature and KCl was filtered off. The excess solvent from the filtrate was removed under reduced pressure to yield a yellow-brown liquid compound, $\text{Ph}_2\text{Sb}(\mu\text{-OPr}^i)_2\text{Sb}(\text{OPr}^i)_2$, (**2**). The compound was analyzed to have Sb, 19.30; $-\text{OPr}^i$, 37.08 calculated for $\text{C}_{24}\text{H}_{38}\text{O}_4\text{Sb}_2$, Sb, 19.20; $-\text{OPr}^i$, 37.28%.

A benzene solution (~ 25 ml) of compound (**2**) (1.48 g, 2.33 mmol) was added to the benzene solution (~ 25 ml) of corresponding ligand,

TABLE V Physical and Analytical Data of the Complexes, $K(\mu\text{-OPr}^i)_2\text{Sb}[\text{SC}_6\text{H}_4\text{NC(R)CHC(O)COOCH}_3]$ (1a–1e)

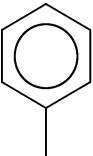
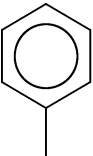
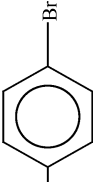
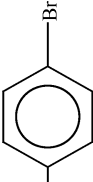
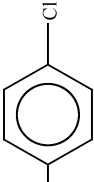
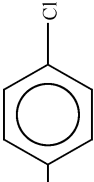
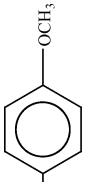
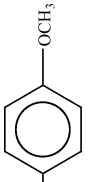
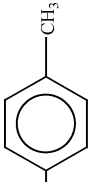
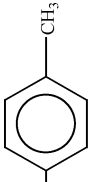
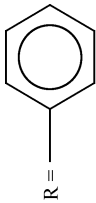
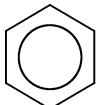
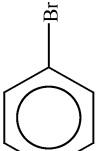
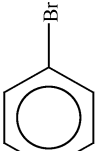
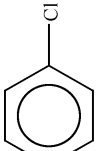
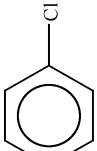
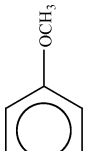
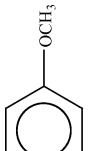
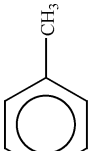
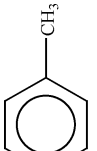
S. no.	Complexes	Molecular formula,			Pr ⁱ OH found (Calc.)	Analysis found (Calc.)				Molecular weight found (Calc.)
		Reagents g (m mol)	colour, physical state, yield (%)	ligands		Sb	N	S	—OPr ⁱ	
		KSb(OPr ⁱ) ₄								
1	 R = 	1.47 (3.70)	C ₂₃ H ₂₇ NO ₅ SKSb Yellow Solid 90%	1.16 (3.70) (L ¹ H ₂)	0.41 (0.44)	20.65 (20.62)	2.35 (2.37)	5.49 (5.43)	20.22 (20.02)	598 (590)
	(1a)									
2	 R = 	1.36 (3.42)	C ₂₃ H ₂₆ NO ₅ SKBrSb Dark yellow Solid 88%	1.34 (3.42) (L ² H ₂)	0.39 (0.41)	18.01 (18.19)	2.07 (2.09)	4.68 (4.79)	17.76 (17.66)	645 (669)
	(1b)									
3	 R = 	1.28 (3.22)	C ₂₃ H ₂₆ NO ₅ SKClSb Yellow Solid 89%	1.12 (3.22) (L ³ H ₂)	0.37 (0.39)	19.60 (19.49)	2.28 (2.24)	5.08 (5.13)	18.82 (18.91)	646 (624)
	(1c)									
4	 R = 	1.32 (3.32)	C ₂₃ H ₂₉ NO ₆ SKSb Dark yellow Solid 85%	1.14 (3.32) (L ⁴ H ₂)	0.39 (0.40)	19.50 (19.62)	2.21 (2.26)	5.11 (5.17)	19.01 (19.05)	601 (620)
	(1d)									
5	 R = 	1.65 (4.15)	C ₂₄ H ₂₈ NO ₅ SKSb Dark yellow Solid 92%	1.36 (4.15) (L ⁵ H ₂)	0.47 (0.50)	20.29 (20.14)	2.30 (2.32)	5.27 (5.31)	19.51 (19.55)	625 (604)
	(1e)									

TABLE VI Physical and Analytical Data of the Complexes, $\text{Ph}_2\text{Sb}(\mu\text{-OPr}^i)_2\text{Sb}[\text{SC}_6\text{H}_4\text{NC(R)CHC(O)COOCH}_3]$ (2a-2e)

S. No.	Complexes	Reagents g (m mol)		Molecular formula, colour, physical state, yield (%)	Pr ⁱ -OH found (Calc.)	Analysis found (Calc.)				Molecular weight found (Calc.)
		Compound (2)	Ligands			Sb	N	S	-OPr ⁱ	
1.	 R =  (2a)	1.48 (2.33)	0.73 (2.33) (L ¹ H ₂)	C ₃₅ H ₃₇ NO ₅ SSb ₂ Light yellow Viscous liquid 90%	0.26 (0.28)	14.82 (14.72)	1.71 (1.69)	3.86 (3.88)	14.38 (14.28)	840 (827)
2	 R =  (2b)	1.39 (2.19)	0.86 (2.19) (L ² H ₂)	C ₃₅ H ₃₆ NO ₅ SBrSb ₂ Yellow Viscous liquid 86%	0.22 (0.26)	13.38 (13.44)	1.53 (1.55)	3.52 (3.54)	13.11 (13.04)	919 (906)
3	 R =  (2c)	1.42 (2.24)	0.78 (2.24) (L ³ H ₂)	C ₃₅ H ₃₆ NO ₅ SClSb ₂ Yellow Viscous liquid 88%	0.26 (0.27)	14.10 (14.13)	1.60 (1.62)	3.69 (3.72)	13.65 (13.71)	847 (861)
4	 R =  (2d)	1.33 (2.10)	0.72 (2.10) (L ⁴ H ₂)	C ₃₆ H ₃₈ NO ₆ SSb ₂ Dark yellow Viscous liquid 82%	0.22 (0.25)	14.24 (14.20)	1.61 (1.63)	3.71 (3.74)	13.71 (13.78)	838 (857)
5	 R =  (2e)	1.55 (2.44)	0.80 (2.44) (L ⁵ H ₂)	C ₃₆ H ₃₈ NO ₅ SSb ₂ Yellow Viscous liquid 89%	0.27 (0.29)	14.41 (14.47)	1.68 (1.66)	3.77 (3.81)	14.10 (14.05)	830 (841)

L¹H₂ (0.73 g, 2.33 mmol) and mixture was refluxed on fractionating column for about four h. The isopropanol liberated during the reaction was fractinated off azeotropically with benzene and estimated to check the progress, as well as completion of reaction. When reaction was complete, the volatile components were removed under vaccum, for the purification of the compound, it was dissolved in benzene and added n- hexane until the oily layer began to separate. The mixture was cooled at low temperature (0–5°C), and then the solvent was decanted off. The compound was finally dried under reduced pressure to yield a yellow-colored viscous liquid.

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